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## Microwave-convective hot air and vacuum drying of *Syzygium cumini* (L.) Skeels seeds and its effect on total phenolics content, vitamin C and antioxidant activity

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Seeds of (*Syzygium cumini* (L.) Skeels) are rich in medicinal values. In this work, the effect of drying on its total phenolics content (TPC), vitamin C (vit C) and antioxidant activity (AA) was studied. Seeds were dried at 60, 70, 80, and 100 °C using both microwave-convective hot air drying (MCD) at 1, 2, and 3 W/g, keeping air velocity fixed at 0.5 m/s, and vacuum drying (VD) at 60, 160, and 260 mm Hg. Dried seeds were powdered and evaluated. Statistically, MCD at 60 °C, 2 W/g was selected as the best drying method and condition that retained the highest functional properties. Compared to fresh seed powder, this drying condition retained 82% of TPC and  $\approx$  34% vit C and increased AA by about 52%. The drying curve at 60 °C, 2 W/g was fitted to four different mathematical models, viz., Lewis, Page, Henderson and Pabis, and Logarithmic. Logarithmic model was found to be best suited for characterising the drying profile. The final product was non-hygroscopic and free-flowing with a negligible degree of caking.

**Keywords:** Antioxidant activity, jamun seed powder, Microwave-convective hot air drying, *Syzygium cumini* (L.) Skeels, Total phenolics content, Vacuum drying, Vitamin C.

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### Introduction

Several free radicals are constantly generated in the human body leading to various illnesses<sup>1</sup>. Antioxidants are substances that counteract free radicals by multiple mechanisms, and thereby improve the body's immune function, nervous system and quality of sleep. Antioxidants also prevent inflammation, retard ageing, maintain healthy vision, and control obesity. Though some antioxidants are generated in metabolic processes, the body relies on external sources, i.e., diet and/or medicine<sup>1</sup>. Antioxidants are also needed in food processing to extend shelf life by controlling undesirable oxidative reactions. To avoid the use of synthetic antioxidants, researchers are trying to find natural sources such as fruits, vegetables and oil seeds<sup>2</sup>.

*Syzygium cumini* (L.) Skeels, commonly known as jamun in Hindi and Indian blackberry in English, having attractive taste and colour, is a nutritious seasonal fruit found in tropical and subtropical regions of the world but remains underutilized<sup>3</sup>. It consists of a single large dicotyledonous seed sharing

$\approx$  25% of the fruit mass. The fruit, particularly the seed has been documented for a range of therapeutic actions such as antidiabetic, anti-inflammatory, anti-hyperlipidaemic, anticancerous, and antimicrobial<sup>4</sup>. Besides these, jamun seeds, due to the presence of phenolic compounds, vitamin C (vit C) and several other phytochemicals, also exhibits strong antioxidant activities. These antioxidants are responsible for scavenging free radicals and have a protective effect on antioxidant enzymes<sup>4-6</sup>. Benherlal & Arumughan<sup>7</sup> and Vasi & Austin<sup>8</sup> established the free radicals (viz., superoxide, hydroxyl, lipid peroxide, 2,2-diphenyl-1-picrylhydrazyl i.e., DPPH, and nitric oxide) scavenging activity of jamun seeds in the *in vitro* studies. Concerning skin carcinogenesis, Parmar *et al.*<sup>9</sup> estimated the antioxidant activity (AA) of jamun seeds through *in vivo* studies.

Dried jamun seed, in powder form, is popular as an alternative medicine since ancient times. However technical details of drying, for using jamun seed powder as medicine, is not available. It has now been established that the mode of drying has a significant effect on products' phytochemical constituents and functionality<sup>10</sup>. To prepare jamun seed powder fortified food, recently Sood *et al.*<sup>11</sup> dried jamun seed

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using tray drying and then converted it into powder. They judged the quality of the prepared food based on sensory evaluation, but not on improvement in functionality.

Although tray drying is most prevalent, the undesirable product qualities, such as dark colour, low rehydration capacity, and hard texture, due to long drying time and less efficient heat and mass transfer have compelled researchers to look for alternative drying options. Recently, microwave heating is gaining importance in food dehydration. Unlike hot air drying, microwave can penetrate beneath the dry surface and it allows instant heating in all high-moisture regions leading to rapid mass and heat transfer rate. As the product temperature rises very speedily owing to the high heating rate of microwaves, the development of high vapour pressure occurs inside the product. This results in rapid vapour transfer from inside to the product surface, which in turn helps in the development of a more porous structure of the dried product and leads to lower shrinkage, increased crispiness, and lower energy consumption. However, as the microwave is less efficient for drying in a low moisture content region, the use of microwave alone may cause overheating and charring due to such non-uniform heating<sup>12</sup>. Infusing hot air drying with the microwave can overcome these limitations. Hot air will facilitate vaporization and removal of free water from the product surface whereas microwave energy will remove moisture from the product interior, thereby enhancing drying rate and reducing energy consumption<sup>13</sup>. Microwave-convective hot air drying (MCD) has been attempted for various fruits and vegetables, such as oyster mushrooms<sup>14</sup>, okra<sup>15</sup>, and Jamun pulp<sup>10</sup>. Vacuum drying (VD), where removal of moisture takes place under low pressure, low temperature and oxygen-deficient environment, is suitable for heat-labile products, e.g., fruits, vegetables. The vacuum applied expands air and water vapour present in food, thereby developing a frothy or puffed structure that facilitates good mass transfer resulting in enhanced drying rate to preserve colour, shape, aroma/flavour and nutritive values of the product<sup>16</sup>. VD has been successfully applied for ripe mangoes<sup>17</sup>, red currants<sup>18</sup>, sour cherries<sup>19</sup>, and aronia<sup>20</sup>. Though many researchers have shown the total phenolics content (TPC), vitamin C (vit C) and AA in fresh jamun seed, no report is available on the effect of drying on retention of its functionality<sup>7,21</sup>.

The present study focuses on the effect of MCD and VD on retention of TPC, vit C, and AA of jamun seeds, followed by selection and investigation of the kinetics of the method that better conserves these functionalities. Further, since the hygroscopicity of food powder is a critical factor affecting caking and flowability, these properties of the developed product have also been discussed.

## Materials and Methods

### Materials

Mature jamun fruits were procured during the month of June (2015) from Technology Market, IIT Kharagpur, West Bengal, India. The fruits were properly washed with potable water and dried under a fan. The seeds (with seed coat) were manually separated from the pulp, cleaned from the adhered pulp, and packed in low-density polyethylene (LDPE) pouches. These were stored in a deep freezer (Model: IIC-401-V; Instrumentation India, Kolkata, India) at -30°C for subsequent studies.

All the chemicals and reagents used in the experiment were of analar/extrapure grade and procured from different sources. 2,2-diphenyl-1-picrylhydrazyl (DPPH) and 2(3)-tert-butyl-4-methoxyphenol (butylated hydroxyl anisole, BHA, 96%) were obtained from Merck Specialities Pvt. Ltd., Germany. 2,6-dichlorophenol-indophenol and meta-phosphoric acid (HPO<sub>3</sub>) were obtained from Sigma-Aldrich, Bengaluru, India, while glacial acetic acid was from Sisco Research Laboratories Pvt. Ltd., Mumbai, India. Ethanol, methanol, Folin-Ciocalteu phenol reagent (1N), gallic acid, hydrochloric acid, and L-ascorbic acid were procured from Merck Specialities Pvt. Ltd., Mumbai, India. The water used in all analyses was glass distilled water (dw).

### Drying of jamun seeds

Two methods, viz., MCD and VD, under different conditions (Table 1), were used. The frozen seeds (with coat) were thawed in an air bath before loading into the respective dryer.

Table 1—Different drying methods and conditions used for drying Jamun seeds

Drying methods	Drying conditions
Microwave-convective hot air drying	Temperature (4 levels): 60, 70, 80, 100 °C Power density (3 levels): 1, 2, 3 W/g
Vacuum drying	Temperature (4 levels): 60, 70, 80, 100 °C Pressure (3 levels): 60, 160, 260 mm Hg

#### **Microwave-convective hot air drying**

A miniature industrial microwave-convective hot air dryer (Enerzi Microwave Systems Pvt. Ltd., Bangalore, Karnataka, India) comprising of two microwave sources, each having capacity of 1.5 kW, was used. The microwave power ranged from 250 to 3000 W with 2450 MHz working frequency. An in-built heater of 6 kW brought about the hot air circulation, the range of air temperature from 25 to 200 °C. The drying chamber, with the dimension of 2 m x 300 mm x 200 mm, was made up of 2 mm thick stainless steel-304. The outer body of the dryer was made up of mild steel having an anti-corrosive epoxy powder coating. The drying capacity of the equipment was 20-100 kg/h. One hour before sample loading, the dryer heater (temperature set as per the experimental design) and hot air blower (fixed at 0.5 m/s) were switched on, so that the drying chamber reached steady-state temperature. The thawed jamun seeds were spread in a single layer on a teflon plate and loaded inside the drying chamber. After closing the lid properly, the microwave controller was switched on (input power set as per the required power density in watt (W)/g of sample). At every 30 minutes, the sample was taken out and weighed till the attainment of constant weight.

#### **Vacuum drying**

A batch scale vacuum dryer (Instrumentation India, Kolkata, India) comprised of a rectangular drying chamber (290 mm x 290 mm x 430 mm), temperature controller with a display board, a pressure gauge (0-760 mm Hg vacuum), and a vacuum pump was used. The inner wall of the drying chamber was made up of stainless steel. The vacuum dryer was switched on and the required temperature was set 1 h before loading the sample, to achieve steady-state temperature during each experimental run. About 100 g of thawed seeds, spread evenly on a teflon plate, was fed into the drying chamber and the required vacuum was set. The vacuum was released at 1 h interval for taking the weight of the sample. The drying was continued till the sample reached a constant weight.

#### **Grinding of dried seeds**

The dried jamun seeds from both processes were ground to a fine powder using a grinder (Sumeet Research and Holdings Limited Chennai, India). An aliquot of the jamun seed powder (JSP) was used for the determination of moisture content, if any, by further drying at 60 °C in a hot air oven (Electronics and Electrical Co., Kolkata, India) following the

method of Ranganna<sup>22</sup>. This was needed to express the results on a dry basis (db). The rest of the powder was packed in double packaging in LDPE pouches, and stored in a deep freezer (-30°C) to analyse for TPC, vit C and AA. Analyses were completed within two days after making the powder.

#### **Analysis of the dried samples for selection of the best drying method**

##### *Preparation of JSP extract for TPC and AA determination*

About 1 g of JSP was taken and 20 mL of 50% aqueous ethanol (99.9% purity) was added to it, followed by shaking for 3 h at 27 °C in an incubator shaker (Sambros & Co India Pvt. Ltd. Kolkata, India). The mixture was centrifuged (R 24 Remi Instruments Ltd., Mumbai, India) at 7500 rpm for 20 minutes. The volume of the supernatant was measured and collected for TPC and AA determination.

##### *Preparation of JSP extract for vit C determination*

About 1 g of JSP was added to 20 mL of 2% HPO<sub>3</sub><sup>22</sup>. The mixture was vortexed for few seconds and centrifuged at 7500 rpm for 20 min. The volume of supernatant was measured, and used for vit C determination.

#### **Estimation of TPC**

The TPC was determined by Folin-Ciocalteu colourimetric method, as was followed by Dey Paul & Das<sup>10</sup>. Briefly, 0.025 mL of the extract was mixed with 0.5 mL of ten times diluted Folin-Ciocalteu reagent. After 3 minutes, 2 mL of 20% (w/v) sodium carbonate solution was added to the sample mixture and diluted by dw to make the volume up to 15 mL. The tubes were then kept in dark for 45 minutes and absorbance at 725 nm was measured using a spectrophotometer (ELICO Double Beam SL 210 UV VIS Spectrophotometer, Hyderabad, India). A standard curve was prepared using gallic acid of different concentrations ranging from 0.01 to 0.08 mg in 0.025 mL of 50% aqueous ethanol. TPC was expressed as gallic acid equivalents (GAE) in mg/g (db).

#### **Estimation of AA**

Antioxidant activity (AA) was determined based on scavenging DPPH free radical<sup>10</sup>. An aliquot (1 mL) of the extract was added to 3 mL of DPPH (1 g/L) in methanol and shaken vigorously. A control sample containing 1 mL of methanol and 3 mL of DPPH was also prepared similarly. The absorbance of the resulting solution was measured using the spectrophotometer at 517 nm against absolute methanol as blank, after keeping the solution in dark at 25 °C for 30 minutes.

The scavenging effect on the DPPH radical was calculated using the equation given below.

$$\% \text{ Radical Scavenging Activity (\% RSA)} = \left(1 - \frac{\text{absorbance of sample at 517 nm}}{\text{absorbance of control at 517 nm}}\right) \times 100$$

The AA of JSP was finally expressed as BHA equivalent. Different concentration of BHA was prepared and the corresponding %RSA was determined using the above methodology. To calculate the AA of the extract(s) as mg BHA/g of sample (db), the standard curve was plotted between % RSA and concentration of BHA.

#### Estimation of vit C

Vitamin C in JSP extract was determined by the direct colourimetric method, as described in Ranganna<sup>22</sup>. This method is based on the decolourization of 2,6-dichlorophenol-indophenol by ascorbic acid. Briefly, 0.5 mL of the extract was mixed with 0.5 mL of 2% HPO<sub>3</sub> to make 1 mL. Then 9 mL of diluted dye solution (12.5 mL of 2,6-dichlorophenol-indophenol solution diluted to 250 mL using dw) was added to it. The absorbance of the solution was measured at 518 nm within 15-20 seconds using the spectrophotometer. A mixture of 1 mL of 2% HPO<sub>3</sub> and 9 mL of dw was used as blank. A standard curve was prepared following a similar methodology with different concentrations of L-ascorbic acid and the result was expressed as mg ascorbic acid/g, db.

#### Selection of best drying method

The method and condition that retained the maximum value of TPC, vit C, and AA were selected as the best method for drying jamun seed.

#### Kinetics of drying

The drying rate was plotted (not shown) against the corresponding moisture content of the sample. The whole drying was found to occur in the falling rate period (discussed later). Next, the moisture ratio (MR) was calculated using the equation given below at different time during drying by the best-selected method.

$$\text{Moisture ratio} = \frac{M - M_e}{M_o - M_e}$$

where M is the moisture content at any time, M<sub>e</sub> is the equilibrium moisture content, and M<sub>o</sub> is the initial moisture content<sup>23</sup>. To express mathematically, different models<sup>23</sup>, as shown in Table 2, were attempted using Origin Pro 8.5 software (UK).

#### Hygroscopicity, degree of caking, and flowability of JSP

The fresh seed (containing ≈107% moisture on db) in ground condition, and the dried powder, i.e., JSP obtained under the best-selected method were estimated for hygroscopicity (HG, %), degree of caking (DC, %), and flowability (s); the methodologies followed were similar to that of Jaya *et al.*<sup>24</sup> for vacuum dried mango powder. Since the drying operation greatly affects a product's microstructure, knowledge about these parameters are required to understand the shelf-stability and handling properties<sup>25</sup>.

HG (%) was expressed as the final moisture content achieved after exposure of the powder sample at a high relative humidity (79.2% at 20 °C)<sup>26</sup>. The HG (%) was calculated using the equation given below.

$$\text{HG (\%)} = \frac{(\text{MI\%} + \text{IM\%}) \times 100}{100 + \text{MI\%}}$$

where

$$\text{Moisture increase in sample (MI\%)} = \frac{(\text{Final weight of the sample} - \text{Initial weight of the sample}) \times 100}{\text{Initial weight of the sample}}$$

and

IM% = Initial moisture content in the sample (% wet basis).

To measure DC (%), the wet sample obtained after HG determination was dried at 102±2 °C for 1 hour, cooled, and sieved (500 μm mesh). The DC (%) was calculated using the equation below:

$$\text{DC (\%)} = \frac{b}{a} \times 100$$

where a (g) is the total amount of powder taken for sieving and b (g) is the weight of powder left on the sieve after sieving.

The flowability was expressed as the time (s) required for a given volume of powder to leave a rotary drum through a slit of a particular size<sup>24</sup>.

#### Statistical analysis

All measurements were performed in triplicate and the mean±standard deviation (SD) was calculated.

Table 2 — Mathematical models applied to drying curves of the seeds dried under the selected drying condition

Model name	Model equation
Newton/Lewis	MR = exp(-kt)
Page	MR = exp(-kt <sup>n</sup> )
Henderson and Pabis	MR = aexp(-kt)
Logarithmic	MR = aexp(-kt) + c

t: drying time in min; k, n, a, c: model constants

Two-way analysis of variance (ANOVA) was carried out to check the effect of operating parameters, viz., temperature and power density for MCD, while temperature and pressure for VD, on TPC, vit C and AA. At each power density (in case of MCD) and each pressure (in case of VD), treatment means were compared for temperature variation (60-100 °C) based on the least significant difference at 5% level of significance (LSD: 0.05) (in case of positive F-test through one-way ANOVA). The paired t-test ( $p < 0.05$ ) was applied for comparison between any two means.

Selection of the best fit mathematical model for drying was done in terms of adjusted coefficient of determination (adjusted  $R^2$ ), root mean square error (RMSE) (equation below) and residual plot, i.e., a plot of 'actual value minus predicted value' versus 'actual value'.

$$RMSE = \left[ \frac{1}{n} \times \sum_{i=1}^n (M_i - M_{pi})^2 \right]^{1/2}$$

where  $M_i$  and  $M_{pi}$  are the actual and predicted values of MR and  $n$  is the number of observations.

## Results and Discussion

The time needed to attain constant weight was specific to the drying method and associated condition, varying from 4 to 10 hours and 5 to 17 hours for MCD and VD, respectively. The mean value $\pm$ SD (db) of TPC, vit C, and AA of fresh *Jamun* seed (in ground condition) and dried powder samples (JSP) are presented in Table 3. Unless otherwise mentioned, the operating parameters, viz., temperature and power density for MCD, and

temperature and pressure for VD, individually and their interactions, indicated a significant effect ( $P < 0.05$ ) on these functionalities. Values of  $LSD_{0.05}$  for variation in temperature (60-100 °C) at each power density (MCD) and each pressure (VD) are included in the same table. It is worth mentioning that in addition to the defined operating variables, the time needed for drying was also an uncontrolled parameter for retention of functionalities<sup>10</sup>.

### Effect on TPC

As in Table 3, the JSP obtained through MCD and VD suffered an overall decline (15-39%) in TPC with respect to (w.r.t.) that of fresh seed powder (81.81 mg GAE/g), the extent was dependent on the particular method-condition combination. Reduction in TPC in hot air drying was observed for murta berries (*Ugnimolinae* T.) by Rodríguez *et al.*<sup>27</sup>, and quinoa seeds (*Chenopodium quinoa* Willd.) by Miranda *et al.*<sup>28</sup>. Structural and/or chemical changes of phenolics or their entangling with other coexistent phyto-components, under influence of temperature, to form inextricable compound could be the possible reason(s) of such lowering, as explained by Mrad *et al.*<sup>29</sup> and Lutz *et al.*<sup>30</sup>. Contrary to this, an increase in TPC due to enzymatic and non-enzymatic reactions in the food matrix has also been reported by Cheng *et al.*<sup>31</sup> and Izli<sup>32</sup>.

Here, in the case of MCD at 1 W/g (Table 3), TPC value increased from 59.80 mg GAE/g at 60 °C to 65.33 mg GAE/g at 70 °C, and then followed a decreasing trend reaching 50.30 mg GAE/g at 100 °C.

Table 3 — Total phenolics content (TPC), vitamin C (vit C) and antioxidant activity (AA) values of fresh, MCD, and VD jamun seed powder<sup>@1</sup>

Jamun seed powder samples	TPC (mg GAE/g, db)			vit C (mg ascorbic acid/g, db)			AA (mg BHA/g, db)		
	1 W/g	2 W/g	3 W/g	1 W/g	2 W/g	3 W/g	1 W/g	2 W/g	3 W/g
Fresh seed (ground)	81.81 $\pm$ 0.18			3.62 $\pm$ 0.08			42.73 $\pm$ 0.54		
JSP (MCD)	1 W/g	2 W/g	3 W/g	1 W/g	2 W/g	3 W/g	1 W/g	2 W/g	3 W/g
60 °C	59.80 $\pm$ 0.68	<b>67.08<math>\pm</math>0.80</b>	63.18 $\pm$ 0.30*	<b>1.40<math>\pm</math>0.10</b>	1.22 $\pm$ 0.07*	1.02 $\pm$ 0.27	62.08 $\pm$ 0.18*	65.14 $\pm$ 0.29	64.39 $\pm$ 0.50
70 °C	65.33 $\pm$ 0.76	64.09 $\pm$ 0.60*	63.32 $\pm$ 0.73 <sup>#</sup>	1.30 $\pm$ 0.04	1.20 $\pm$ 0.10*	1.05 $\pm$ 0.13	<b>66.12<math>\pm</math>0.50</b>	60.33 $\pm$ 0.82	60.47 $\pm$ 0.49
80 °C	62.92 $\pm$ 1.30	63.04 $\pm$ 0.75*	62.13 $\pm$ 0.96 <sup>#</sup>	1.19 $\pm$ 0.04	1.19 $\pm$ 0.12*	1.00 $\pm$ 0.10	62.48 $\pm$ 0.57*	52.11 $\pm$ 0.89	49.21 $\pm$ 1.23
100 °C	50.30 $\pm$ 0.62	52.16 $\pm$ 0.31	64.53 $\pm$ 0.47 <sup>#</sup>	1.01 $\pm$ 0.49	0.98 $\pm$ 0.04	1.32 $\pm$ 0.11	46.46 $\pm$ 1.62	45.44 $\pm$ 1.69	57.59 $\pm$ 0.52
LSD <sub>0.05</sub>	1.67	1.21	1.25	-	0.16	-	1.69	1.98	1.42
JSP (VD)	60 mmHg	160 mmHg	260 mmHg	60 mmHg	160 mmHg	260 mmHg	60 mmHg	160 mmHg	260 mmHg
60 °C	55.93 $\pm$ 0.40	53.33 $\pm$ 0.26	54.73 $\pm$ 0.25	0.23 $\pm$ 0.01	0.09 $\pm$ 0.02	0.60 $\pm$ 0.07*	48.34 $\pm$ 0.53*	43.86 $\pm$ 1.49	51.87 $\pm$ 1.28
70 °C	52.80 $\pm$ 0.26	<b>68.77<math>\pm</math>0.23</b>	56.72 $\pm$ 0.20	0.48 $\pm$ 0.03	0.02 $\pm$ 0.01	0.62 $\pm$ 0.08*	49.31 $\pm$ 1.04*	38.94 $\pm$ 1.71	59.01 $\pm$ 0.12*
80 °C	64.44 $\pm$ 0.59	55.18 $\pm$ 0.28	68.06 $\pm$ 0.41*	0.57 $\pm$ 0.06	0.23 $\pm$ 0.07	<b>0.84<math>\pm</math>0.05</b> <sup>#</sup>	66.11 $\pm$ 1.62	51.46 $\pm$ 0.47	58.05 $\pm$ 0.72*
100 °C	51.51 $\pm$ 0.43	51.70 $\pm$ 0.61	68.06 $\pm$ 0.41*	0.31 $\pm$ 0.04	0.16 $\pm$ 0.02	0.80 $\pm$ 0.06 <sup>#</sup>	54.23 $\pm$ 0.70	56.49 $\pm$ 1.20	<b>67.08<math>\pm</math>1.22</b>
LSD <sub>0.05</sub>	0.82	0.72	0.63	0.07	0.07	0.12	1.99	2.45	1.80

<sup>@</sup>, mean $\pm$ SD of 3 replicates; <sup>!</sup>, Highest value from MCD and VD are marked bold

At 2 W/g, the TPC decreased gradually from 67.08 mg GAE/g at 60 °C to 52.16 mg GAE/g at 100 °C (at 70 and 80 °C, values were not significantly different); percentage retention of TPC was noticed to be higher at 2 W/g compared to 1 W/g. At 3 W/g, JSP maintained an average value of 63.29 mg GAE/g. Thus at 60 °C, TPC showed the order for 2 W/g > 3 W/g > 1 W/g, while at 70 and 80 °C, the values showed close similarity. Corresponding to 100 °C, TPC was the maximum at 3 W/g, whereas for 1 and 2 W/g, the values remained  $\approx$  50-52 mg GAE/g. A shorter drying time (about 4 hours) was probably the reason for displaying such a result.

In the case of VD, the TPC did not follow any specific trend under 60 mm Hg absolute pressure (Table 3). With an increase in temperature from 60 to 70 °C, TPC value decreased from 55.93 to 52.80 mg GAE/g; at 80 °C, phenolics content increased to 64.44 mg GAE/g. With further increase in temperature (100 °C), the TPC declined to 51.51 mg GAE/g. Under 160 mm Hg, TPC increased from 53.33 mg GAE/g (60 °C) to 68.77 mg GAE/g (70 °C), and then followed a decreasing trend reaching 51.70 mg GAE/g at 100 °C. The TPC followed a positive trend under 260 mm Hg, where the value increased gradually from 54.73 mg GAE/g at 60 °C to 68.06 mg GAE/g at 80 °C; no further change in TPC was noted when the temperature was increased to 100 °C. Thus, TPC value at 70 °C and 160 mm Hg was observed to be the highest irrespective of the drying method and condition, exhibiting retention of  $\approx$  84%.

#### Effect on vit C

Drying of jamun seeds led to considerable deterioration in vit C content, the minimum loss was no less than about 61% w.r.t. 3.62 mg ascorbic acid/g of fresh seeds (Table 3). Apparently, the effect of VD was more detrimental than MCD.

As shown for MCD in Table 3, the vit C content in JSP at 60 °C and 1 W/g was highest (1.40 mg ascorbic acid/g) indicating about 39% retention w.r.t. that of fresh seeds. Beyond 60 °C, the vit C kept on decreasing with increasing temperature; at 100 °C, the value reduced to 1.01 mg ascorbic acid/g. However, this reduction was found to be statistically insignificant, hence, it can be considered that an average value of 1.23 mg ascorbic acid/g was maintained throughout for 1 W/g. At 2 W/g, vit C content revolved around the average value of 1.2 mg ascorbic acid/g within a temperature range of 60-80 °C, however, about 18% decrease was noticed

when the temperature was increased from 80 to 100 °C (0.98 mg ascorbic acid/g, db). At 3 W/g, the response was insignificantly influenced by the change in drying temperature, corresponding to an average value of 1.10 mg ascorbic acid/g (30% retention). From two-way ANOVA, though individually the drying air temperature and power density had no significant effect on the vit C content of JSP, their interactive effect had a significant influence ( $P < 0.05$ ).

The values of vit C in JSP from VD were much less than that of fresh jamun seed, even compared to that from MCD (Table 3). The long period during VD may be the dominant factor in the loss of thermolabile vit C. Two-way ANOVA further showed that the pressure levels have a stronger influence on this response than the variation in temperature. The interactive effect of these two independent variables was also significant, but lower compared to their individual effects. Under 60 mm Hg, vit C content of JSP was observed to be 0.23 mg ascorbic acid/g at 60 °C. As the temperature was increased beyond 60 °C till 80 °C, the vit C content kept on rising significantly by reaching 0.57 mg ascorbic acid/g; at 100 °C the value again reduced to 0.31 mg ascorbic acid/g. The pressure level of 160 mm Hg at 60 and 70 °C resulted in the maximum loss, retaining only 0.09 and 0.02 mg ascorbic acid/g respectively. Sample dried at 80 °C under the same pressure level showed little improvement (0.23 mg ascorbic acid/g), but the value again reduced to 0.16 mg ascorbic acid/g at 100 °C. Among the three pressure levels, 260 mm Hg was observed to be the least harsh. The vit C content did not vary significantly when the temperature was increased to 70 °C. The drying temperature of 80 °C increased vit C to 0.84 mg ascorbic acid/g; further increase to 100 °C did not lead to any significant change. This favourable effect of drying temperature (80 °C) on vit C content was also observed by Sonawane & Arya<sup>33</sup>.

#### Effect on AA

Contrary to TPC and vit C, the AA of JSP samples was usually enhanced by both the drying methods (Table 3). The percentage increase in AA ranged from  $\approx$  2 to 57% (w.r.t the initial AA value of 42.73 mg BHA/g) depending on the drying method and condition employed. This indicates that AA was not only related to phenolics and vit C, but also other compounds<sup>34</sup>. Lutz *et al.*<sup>30</sup> explained that this opposite observation could be due to other compounds with AA, the new compound formed in Maillard reaction,

and the deactivation of oxidative and hydrolytic enzymes. Drying method, extraction solvents, assay methods, and even interactions of several antioxidants may contribute to such discrepancy<sup>35</sup>. Concordance to the present findings, absence of correlation between AA and TPC was also documented for other fruits by Ikram *et al.*<sup>36</sup> and Al-Farsi *et al.*<sup>37</sup>. For MCD samples, Aghilinategh *et al.*<sup>38</sup> opined that high vapour pressure, heat and vibration generated by microwave inside the tissue may lead to rupturing of the cell matrix releasing antioxidant compounds.

At the power density of 1 W/g, the AA of MCD samples increased from 62.08 mg BHA/g at 60 °C to 66.12 mg BHA/g at 70 °C (Table 3). Beyond 70 °C, AA followed a negative slope; at 100 °C the value dropped to 46.46 mg BHA/g. This type of trend was also observed for coriander leaves that showed maximum DPPH radical scavenging activity at 60 °C in the range of 40 to 100 °C<sup>39</sup>. The AA depicted a gradual decline with the rising temperature at 2 W/g power density; the value ranged from 65.14 mg BHA/g at 60 °C to 45.44 mg BHA/g at 100 °C. Similarly, at 3 W/g, AA of JSP decreased from 64.39 mg BHA/g at 60 °C to 49.21 mg BHA/g at 80 °C. However, at 100 °C, the value again increased to 57.59 mg BHA/g; the reduced drying period at 100 °C ( $\approx$  4 h) compared to other MCD conditions (6 h at 100 °C, 1 W/g; 5 h at 100 °C, 2 W/g) might have promoted better retention of AA<sup>40</sup>.

Considering VD (Table 3), under 60 mm Hg, AA was increasing with temperature ranging from 60 °C (48.34 mg BHA/g) to 80 °C (66.11 mg BHA/g). However, the change in AA with temperature change from 60 to 70 °C was not statistically significant ( $P > 0.05$ ). When the temperature was further raised to 100 °C, the AA value stepped down to 54.23 mg BHA/g. The AA under 160 mm Hg encountered loss from 43.86 mg BHA/g at 60 °C to 38.94 mg BHA/g at 70 °C, which is even lesser (by 8.87%) than that of fresh seeds. Drying beyond 70 °C improved AA; the obtained values at 80 °C and 100 °C were 51.46 mg BHA/g and 56.49 mg BHA/g, respectively. The AA value at 60 °C-260 mm Hg was estimated to be 51.87 mg BHA/g (21% increase w.r.t. fresh samples); at still higher temperatures of 70 and 80 °C, the AA increased to an average of 58.53 mg BHA/g. The sample dried at 100 °C, 260 mm Hg recorded the highest among all the samples (including MCD and VD) with a value of 67.08 mg BHA/g (increased by 57%).

#### Selection of best drying method for JSP

As indicated in Table 3, the maximum value (marked bold) of TPC, AA and vit C, irrespective of the operating parameters obtained in both the drying were judged based on paired t-test. In the case of TPC, the maximum attainable value of 67.08 mg GAE/g from MCD was not significantly different from that of VD (68.77 mg GAE/g). No significant difference was observed between the maximum values of AA derived from both the drying, whereas vit C was significantly higher for MCD (1.40 mg ascorbic acid/g) compared to 0.84 mg ascorbic acid/g from VD. Moreover, to attain the respective highest value, the time required in MCD was less than that of VD. Therefore, preliminarily, MCD was preferred over VD for JSP.

The next target was to find out the most favourable MCD condition. In the case of TPC, the values at 60 °C-2 W/g (67.08 mg GAE/g) and 70 °C-1 W/g (65.33 mg GAE/g) were the 1<sup>st</sup> and 2<sup>nd</sup> highest among all other MCD conditions (Table 3). These two values were significantly different ( $P < 0.05$ ). Hence, 60 °C-2 W/g was the best drying condition for TPC. Regarding AA, the two highest treatment means were obtained at 70 °C-1W/g (66.12 mg BHA/g) and 60 °C-2 W/g (65.14 mg BHA/g) (Table 3). The paired t-test showed no significant difference between them ( $P > 0.05$ ). Since the best MCD condition for TPC was found to be suitable for AA too, the MCD condition of 60 °C-2 W/g was selected to be the most favourable. However, the vit C at 60 °C-2 W/g (1.22 mg ascorbic acid/g) was marginally lower than the value corresponding to 60 °C-1 W/g (1.40 mg ascorbic acid/g).

#### Modelling of drying curve

The whole drying process under the selected method-condition combination occurred in the falling rate period and diffusion-controlled mechanism appeared to be prevalent. The values of model constants, adjusted R<sup>2</sup>, RMSE and nature of residual plots are given in Table 4. All the values of R<sup>2</sup> (close to 1) and RMSE (close to zero) were acceptable, whereas the residual plot was random (desirable) only for the Logarithmic model. Hence, the Logarithmic model was found to be the best suitable model for characterising the drying curve of JSP.

The predicted MR versus drying time curve from the Logarithmic model, along with experimental data marked on it, is shown in Fig. 1. Compliance to Logarithmic model was reported by Lee & Kim<sup>41</sup>

Table 4 — Model constants, adjusted R<sup>2</sup>, RMSE and nature of the residual plot of different drying models

Models	Model coefficients				Adjusted R <sup>2</sup>	RMSE	Residual plot
	k	n	a	c			
Newton/Lewis	0.0082	-	-	-	0.98	0.0394	Pattern
Page	0.0041	1.1397	-	-	0.99	0.0328	Pattern
Henderson and Pabis	0.0083	-	1.0145	-	0.98	0.0391	Pattern
Logarithmic	0.0060	-	1.1240	-0.1439	0.99	0.0205	Random

Table 5 — Flow properties of fresh and dried jamun seed powder (JSP) at the selected MCD condition

Properties	Fresh jamun seed powder	JSP (MCD: 60 °C, 2 W/g)
Hygroscopicity (%)	30	7.46
Degree of caking (%)	33.4	0.82
Flowability (s)	-	18

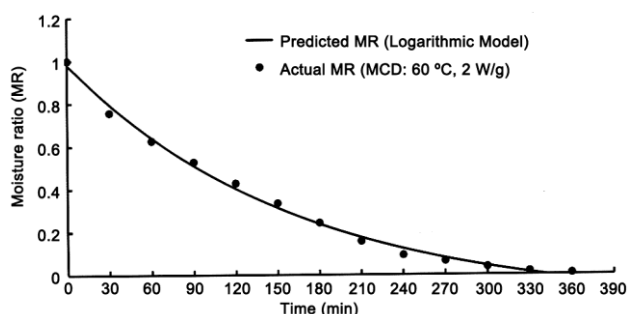


Fig. 1 — Variation of moisture ratio (MR) of jamun seed as a function of drying time.

for Asian white radish and Xanthopoulos *et al.*<sup>42</sup> for peeled and unpeeled whole figs.

#### Flow properties of JSP dried at the selected drying condition

The HG (%), DC (%), and flowability (s) of fresh seed (in ground condition) and JSP (moisture content: 1.17% db) obtained under MCD condition of 60 °C and 2 W/g are presented in Table 5.

According to Jaya *et al.*<sup>24</sup>, a powder with HG less than 10% is considered as ‘non-hygroscopic’. They have also stated that the powder having DC of more than 50% is considered as ‘highly caking’, between 20 and 50% as ‘caking’ and from 10 to 20% as ‘slightly caking’. It is clear from the table that JSP is non-hygroscopic with negligible DC, and has flowability. The flowability measurement of the fresh jamun seed powder was also attempted. However, the high moisture content of the fresh seed powder led to the formation of big lumps which hindered the said operation to be carried out. Hence, the high values of HG and DC of fresh seed powder indicated that MCD improved the moisture sorption characteristics of JSP imparting it with impressive flowability.

## Conclusion

The MCD at 60 °C, 2 W/g and 0.5 m/s air velocity was found to be the best suited for drying the jamun seed to retain the target functionalities. The produced JSP preserved 82% TPC and ≈ 34% vit C of fresh seed, while AA of the product got increased by about 52%. Further, the powder thus produced was non-hygroscopic with good flowability. The logarithmic model was the best suitable model to describe the drying profile. The recommended method can be used for the large scale production of JSP.

## Conflict of interest

The authors declare that there is no conflict of interest.

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