Effect of freeze, microwave-convective hot air, vacuum and dehumidified air drying on total phenolics content, anthocyanin content and antioxidant activity of jamun (*Syzygium cumini* L.) pulp

Indira Dey Paul¹ · Madhusweta Das¹

Revised: 8 November 2017 / Accepted: 9 April 2018 / Published online: 16 April 2018
© Association of Food Scientists & Technologists (India) 2018

Abstract Jamun (*Syzygium cumini* L.) pulp-skin paste was dried using four drying methods viz., microwave-convective hot air drying (MCD), freeze drying (FD), vacuum drying (VD), and dehumidified air drying (DAD) at different conditions. Except for FD, the drying temperatures of MCD (1, 2 and 3 W/g power density), VD (60, 160 and 260 mm Hg pressure) and DAD (1, 1.5 and 2 m/s of air velocity; 20% RH) were varied from 40 to 70 °C. The dried pulp-skin flakes were analysed for total phenolics content [TPC, gallic acid equivalent (GAE)], monomeric anthocyanin content [MAC, malvidin-3-glucoside (M3G)] and antioxidant activity [AA, butylated hydroxyanisole (BHA)]. This study aimed on selection of the best drying method based on the retention of the above functionalities. Compared to the fresh sample, TPC and AA of the dried samples increased for all the methods, whereas MAC showed mixed response. With the retention of 31.52 mg GAE, 11.99 mg M3G and 28.63 mg BHA per gram of dried sample (on dry basis), MCD (70 °C, 1 W/g) was selected as the most suitable method based on statistical analyses

Keywords *Syzygium cumini* · Freeze drying · Microwave-convective hot air drying · Vacuum drying · Dehumidified air drying

Introduction

Berries, e.g., blueberry, cranberry, strawberry, blackberry, mulberry, and raspberry are a diverse group of red, blue or black-purple small fruits. These are consumed widely for their attractive color, delicate texture, unique flavour, and considered as one of the rich sources of micronutrients and phytochemicals counteracting oxidative stress (Mazzoni et al. 2016). As quoted by Prior et al. (2016), berries are excellent source of dietary antioxidants. Giampieri et al. (2014) observed that strawberry extract acts at the cell membrane level to control lipid peroxidation. While reviewing the benefits of strawberry polyphenols, Forbes-Hernandez et al. (2016) further stated that besides the antioxidant capacity these can positively modulate the pathways involved in metabolism, survival, proliferation, and antioxidant defences. Jamun (*Syzygium cumini* L.), also known as Indian blackberry, a tropical fruit in the flowering plant family *Myrtaceae*, is widely popular for its potential bioactive compounds having several health benefits. The jamun pulp together with thin adherent skin ($\approx 75\%$ of the whole fruit) is consumed as food. The presence of anthocyanins and other phytochemicals in the pulp is responsible for its antidiabetic, antioxidant and other therapeutic values (Manganaris et al. 2014; Schwag and Das 2014).

Properly matured jamun fruit is highly perishable and can be stored for maximum 2 days at room temperature (35–40 °C) and 3 weeks at low temperature (Periyathambi 2007). The leading causes of postharvest losses of berries are over-ripening, excessive softening and pathogen attack, mainly by the necrotroph *Botrytis cinerea* (Manganaris et al. 2014). Dehydration of fruits and vegetables is a widely popular preservation technique to extend their shelf-life and make seasonal produce available throughout

✉ Madhusweta Das
madhu@agfe.iitkgp.ernet.in

¹ Department of Agricultural and Food Engineering, Indian Institute of Technology Kharagpur, West Midnapore, West Bengal 721302, India

the year (Orjuela-Palacio and Lanari 2016). Available literature on the jamun pulp drying is limited. Shahnawaz and Sheikh (2008) reported preservation of jamun pulp powder kept in glass containers for 300 days. However, the technical detail of the drying process is not available in their report. Sonawane and Arya (2015) had prepared jamun pulp powder by tray and infra-red drying at 80 and 60 °C to assess the retention of bioactive components in the final dried pulp. Santhalakshmy et al. (2015) prepared jamun juice powder by spray drying. Jebitta and Allwin (2016) estimated total anthocyanin content of jamun pulp dried using vacuum drying. Thus, comprehensive information on functionality of jamun pulp powder dried under different conditions is not available in literature.

Freeze drying (FD) products exhibit excellent quality as most of the deteriorative and microbiological reactions cease owing to the low temperature and absence of liquid water in the drying process. Water sorption isotherms of freeze dried jamun pulp have been studied by de Santana et al. (2015). FD has also been used for drying strawberries and borjón.

The long drying time needed in hot air drying may lead to severe damage to the product's quality attributes. Combining microwave heating with hot air drying may help in reducing drying time thereby retaining the quality attributes of the finished product to a greater extent. Various food materials including garlic cloves, grapes, apples and mushrooms, etc. have been dried using microwave-convective hot air drying (MCD).

Vacuum drying (VD) is suitable for heat-labile products. In this type of drying, removal of moisture takes place under low pressure. Since there is no oxygen, it is suitable for oxygen sensitive food materials. Vacuum drying was attempted to produce mango pulp powder.

Dehumidified air drying (DAD) emerged as a convenient drying method owing to its environment friendly operation, cost-effectiveness, and provision of independent handling of sensible and latent heat loads. DAD has been used for drying of apple and ginger.

Based on the knowledge gap, the present study will focus on the effect of FD, MCD, VD and DAD on the functional properties viz., total phenolics content (TPC), monomeric anthocyanin content (MAC) and antioxidant activity (AA) of jamun pulp, followed by selection of the best drying condition based on the retention of these functionalities.

Materials and methods

Materials and chemicals

Fully matured jamun fruits, free from any visible injury, were procured from Technology market, Kharagpur, West Bengal, in the month of May–June. The fruits were thoroughly washed with tap water, surface water removed, manually separated the pulp from the seed, packed the pulp in zip-lock LDPE pouches (about 250 g/pouch; pouch film thickness ~ 0.05 mm) and stored at – 30 °C. Before drying, the stored frozen mass was thawed by putting in a water bath at room temperature. The thawed mass was made in to paste, called jamun pulp paste, in a mixer-grinder (Sumeet Research and Holdings Limited, Chennai, India).

All the chemicals in estimation of TPC, MAC and AA, unless otherwise mentioned, were of analytical/extra pure grade. Glass distilled water (dw) was used in all the analyses.

Drying methods

The jamun pulp paste was dried using FD, MCD, VD and DAD, under case specific different conditions as shown in Table 1. Except FD, in each case the temperature was varied from 40 to 70 °C, at four levels with a gap of 10 °C.

Freeze drying

A batch scale laboratory freeze dryer (Instrumentation India, Kolkata, India) having vacuum drying chamber, digital temperature and vacuum controllers and a thermal circulating bath was used. The jamun pulp paste (~ 12 g) was spread evenly on a glass petridish and frozen at – 30 °C. The vacuum drying chamber was set at – 35 °C. After the frozen sample was loaded inside the chamber, the thermal circulating bath and vacuum switches were adjusted at 35 °C and 150 mbar, respectively. The sample weight was recorded every 3 h till the attainment of constant weight.

Microwave-convective hot air drying

The jamun pulp paste spread evenly over a Teflon plate was dried in fully controlled miniature industrial microwave-convective hot air dryer (Enerzi Microwave Systems Pvt. Ltd., Bangalore, Karnataka, India) having 20–100 kg/h drying capacity, 2450 MHz frequency and 250–3000 W power. An in-built heater of 6 kW provided hot air (25–200 °C) circulation. The drying chamber, 2 m × 300 mm × 200 mm, was made of 2 mm thick

Table 1 Different drying methods and conditions used for jamun pulp

Drying methods	Drying conditions
Microwave-convective hot air drying (MCD)	Temperature (4 levels): 40, 50, 60, 70 °C Power density (3 levels): 1, 2, 3 W/g
Vacuum drying (VD)	Temperature (3 levels): 50, 60, 70 °C Pressure (3 levels): 260, 160, 60 mm Hg
Freeze drying (FD)	Thermal circulating bath temperature: 35 °C Drying chamber temperature: − 35 °C Vacuum: 150 mbar
Dehumidified air drying (DAD)	RH level: 20% (approx.) Temperature (°C): 40, 50, 60, 70 Air velocity (m/s): 1, 1.5, 2

stainless steel. Drying was done for different combination of temperature and power density (W/g of sample loaded). Sample weight was recorded every 30 min till the attainment of constant weight.

Vacuum drying

A laboratory scale vacuum dryer (Instrumentation India, Kolkata, India) was used. It comprised of a stainless steel drying chamber (290 mm × 290 mm × 430 mm), a temperature controller, a pressure gauge (0–760 mm Hg vacuum), and a vacuum pump. About 40 g of jamun pulp, evenly spread on a Teflon plate, was fed to the dryer. Drying was done for different level of temperature and vacuum. The sample weight was recorded every 1 h till the attainment of constant weight.

Dehumidified air drying

A laboratory scale dehumidified air dryer comprised of a dehumidifier unit (Model: BA-1.5A; Make: BRY-AIR), air blower, heating section, plenum chamber, drying chamber, power supply, thermocouple unit, and control panel was used. The relative humidity (RH) of the air before entering the drying chamber was maintained at $18 \pm 2\%$ using a dehumidifier unit. The temperature and velocity of the air were adjusted throughout the process using a digital temperature controller (Model: DTC 113/113p; Make: Dolphin Automation) and Humidity/Anemometer (Model: AM-4205A; Lutron), respectively. About 20 g of jamun pulp paste was subjected to dehumidified air drying and weight loss in samples were recorded after every 1 h until constant weight was noted.

The mass obtained after each drying method was in flake form. It was ground to powder (hereunder called pure jamun pulp powder, PJPP) using the grinder, packed in LDPE pouches, and stored in deep freezer (− 30 °C) for further analysis.

Determination of TPC and AA

Extract preparation

About one gram of PJPP was extracted with 20 ml of 50% aqueous ethanol (99.9% purity, Merck Chemicals) for 3 h in shaking incubator (Sambros & Co India Pvt. Ltd. Kolkata, India) at 27 ± 1 °C, followed by centrifugation (R 24 Remi Instruments Ltd., Mumbai, India) at 5800 g for 20 min. The supernatant volume was measured and stored in dark at 4 ± 1 °C until further analysis. Jamun pulp paste (amount equivalent to PJPP) was also extracted in exactly similar way. The extracts were estimated for TPC and AA as described below.

Estimation of TPC

Folin–Ciocalteu colorimetric method was used for TPC determination. Briefly, 0.025 ml of the extract was mixed with 0.5 ml of ten times diluted Folin–Ciocalteu reagent (1 N; Merck Specialities Pvt. Ltd., India). After 3 min, 2 ml of 20% (w/v) sodium carbonate solution was added. The mixture was diluted up to 10 ml with dw and kept in dark for 45 min before absorbance at 725 nm was measured using spectrophotometer (ELICO Double Beam SL 210 UV VIS Spectrophotometer, Hyderabad, India). The standard curve was prepared using gallic acid of different concentrations. TPC was expressed as gallic acid equivalents (GAE) in mg/g of sample (powder/paste, db).

Estimation of AA

To determine the AA of PJPP and jamun pulp paste, DPPH (2,2-diphenyl-1-picrylhydrazyl) free radical method was used. An aliquot (1 ml) of the extract was added to 3 ml of DPPH (Sigma-Aldrich, Merck Specialities Pvt. Ltd., Germany) solution (1 g/l in 99.99% methanol). The mixture was shaken vigorously and incubated at 25 °C in the dark for 30 min. Absorbance of the resulting solution was

measured using spectrophotometer at 517 nm against absolute methanol as blank. A control sample containing 1 ml of 99.99% methanol, instead of the extract, was also estimated. The DPPH radical scavenging activity (RSA) of the extract was calculated using Eq. 1.

$$\% \text{Radical Scavenging Activity (\%RSA)} = 1 - \frac{\text{Absorbance of sample at 517 nm}}{\text{Absorbance of control at 517 nm}} \quad (1)$$

To express the antioxidant activity of the extracts in equivalence of Butylated hydroxyanisole (BHA), a standard curve was plotted between %RSA and concentration of BHA. Comparing the %RSA of the extracts with that of BHA, AA of the PJPP/jamun pulp paste was calculated as mg BHA/g of sample (db).

Determination of MAC

The pH differential method was followed for estimating total MAC. About 0.5 g of sample was extracted thrice with 30 ml of acidified methanol (0.1 ml of conc. HCl in 100 ml of 99.99% methanol) in a shaking incubator at 27 ± 1 °C for 3 h (1st extraction). Following centrifugation (5800 g for 20 min), the residue was re-extracted consecutively for 2nd and 3rd time similarly but for 1 h shaking in each case. The supernatants of three extractions were pooled. One millilitre of the extract was diluted five times separately by 0.025 M potassium chloride-HCl buffer (pH 1.0) and 0.4 M acetate buffer (pH 4.5), and allowed to stand in dark for 15 min. The absorbance of both the mixtures was measured at 520 and 700 nm. The estimation was also carried out for jamun pulp paste. The total MAC was measured using Eqs. 2 and 3, and was expressed as mg malvidin-3-glucoside (M3G)/g sample, db.

$$\text{OD} = (\text{OD}_{520 \text{ nm}} - \text{OD}_{700 \text{ nm}})_{\text{pH}=1} - (\text{OD}_{520 \text{ nm}} - \text{OD}_{700 \text{ nm}})_{\text{pH}=4.5} \quad (2)$$

$$\text{Anthocyanins (MAC)} = \frac{(\text{OD} \times \text{MW} \times \text{DF} \times 1000)}{(\epsilon \times l)} \quad (3)$$

where OD, MW, DF, ϵ and l were absorbance, molecular weight of M3G (493.2 g/mol), dilution factor (5), molar absorbance for M3G (28,000 l/(mol.cm)); and pathlength (1 cm), respectively.

Statistical analysis

All the experiments were performed in triplicate and expressed as mean $\pm$ standard deviation (SD). Two- and one-way ANOVA ($p < 0.05$), least significant difference (LSD) and t test (Microsoft Excel, 2013 version) were used for data analyses.

Results and discussion

Time required for drying of the jamun pulp paste till the attainment of constant weight was specific to drying technique as well as process parameters, varying from 3 to 6, 5 to 10 and 4 to 6 h for MCD, VD and DAD, respectively. For FD, the sample reached constant weight by 6 h. It may be mentioned that parametric variation was not possible with the existing FD facility. It was observed that irrespective of technique, time required in drying was inversely proportional to the temperature. The mean values $\pm$ SD (on dry basis, db) of TPC, MAC, and AA of jamun pulp paste, before and after drying (PJPP), is presented in Table 2, where the values before drying were 14.45 mg GAE/g, 8.12 mg M3G/g, and 12.7 mg BHA/g, respectively. Two-way ANOVA indicated significant effect ($p < 0.05$) of the operating parameters for each mode of drying, e.g., temperature and power density in case of MCD, temperature and pressure in case of VD, and temperature and air velocity in case of DAD. LSD₀₅ among the treatment means for variation of temperature obtained at each power density (in case of MCD), each pressure (in case of VD) and each air velocity (in case of DAD) is also included in Table 2.

Effect on TPC

Phenolics remain in free and bound forms in plant materials. Different processing steps have been reported to liberate the phenolic compound from bound state in the food matrix. Here also, drying, in general, was found to increase the phenolics of jamun pulp paste (Table 2). As explained by İzli (2017), cleavage of esterified and glycosylated bonds effected by heat, enzymatic and non-enzymatic reactions might be responsible for the liberation. Additionally, Maillard reaction products formed during thermal treatments may act as precursors of new phenolic compounds (Que et al. 2008). Hoxha and Kongoli (2016) reported increased TPC in hot air dried fig compared to the fresh fruit. Thus, increase in phenolic compounds in jamun pulp could be due to one or more of these reasons. However, decrease as well as insignificant change in TPC has also been reported in drying of persimmon (Akyıldız et al. 2004).

As shown in Table 2, freeze dried PJPP contained TPC of 26.53 mg GAE/g, (db) indicating 83% increase compared to jamun pulp paste. Increase in TPC in freeze drying of date fruit and rabbiteye blueberry has been reported by İzli (2017) and Vuthijumnok et al. (2013), respectively.

In case of MCD (Fig. 1a), TPC showed an increasing trend till 50 °C at 1 W/g power density to produce a value of 35.29 mg GAE/g, db; it did not show any significant

Table 2 Mean values $\pm$ standard deviation of TPC, MAC and AA of jamun pulp paste, before and after drying (PJPP)*

Jamun samples		TPC (mg GAE/g, db)			MAC (M3G, mg/g, db)			AA (mg BHA/g, db)		
Jamun pulp paste		14.45 $\pm$ 0.50			8.12 $\pm$ 1.54			12.7 $\pm$ 0.98		
PJPP (FD)		26.53 $\pm$ 0.58			11.90 $\pm$ 0.71			16.73 $\pm$ 0.54		
PJPP (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	
40 °C	23.38 ^a $\pm$ 0.41	33.94 ^a $\pm$ 0.17	34.78 ^a $\pm$ 0.96	4.28 ^a $\pm$ 0.53	10.5 ^a $\pm$ 0.52	7.03 ^a $\pm$ 0.79	12.12 ^a $\pm$ 0.42	18.52 ^a $\pm$ 0.64	16.3 ^a $\pm$ 0.28	
50 °C	35.29 ^b $\pm$ 0.82	31.5 ^b $\pm$ 0.47	39.95 ^{bc} $\pm$ 0.48	6.64 ^b $\pm$ 0.45	8.34 ^b $\pm$ 0.49	9.13 ^b $\pm$ 0.15	20.55 ^b $\pm$ 0.61	18.22 ^a $\pm$ 0.55	18.07 ^{bc} $\pm$ 0.47	
60 °C	34.93 ^b $\pm$ 0.31	33.74 ^a $\pm$ 0.92	39.18 ^b $\pm$ 1.47	7.67 ^c $\pm$ 0.50	9.16 ^b $\pm$ 0.60	7.53 ^a $\pm$ 0.45	16.23 ^c $\pm$ 0.58	18.08 ^a $\pm$ 0.58	18.74 ^b $\pm$ 0.32	
70 °C	31.52 ^c $\pm$ 0.48	37.16 ^c $\pm$ 0.65	41.12 ^c $\pm$ 0.46	11.99 ^d $\pm$ 0.25	8.04 ^b $\pm$ 0.08	7.79 ^a $\pm$ 0.19	28.63 ^d $\pm$ 0.75	23.09 ^b $\pm$ 0.55	17.92 ^c $\pm$ 0.47	
LSD _{.05}	1.02	1.16	1.77	0.84	0.88	0.88	1.13	1.09	0.75	
PJPP (VD)	60 mmHg	160 mmHg	260 mmHg	60 mmHg	160 mmHg	260 mmHg	60 mmHg	160 mmHg	260 mmHg	
50 °C	27.24 ^a $\pm$ 0.65	24.34 ^a $\pm$ 0.49	20.89 ^a $\pm$ 0.49	5.27 ^a $\pm$ 0.41	6.33 ^a $\pm$ 0.49	4.82 ^a $\pm$ 0.63	19.04 ^a $\pm$ 0.37	16.21 ^a $\pm$ 0.46	16.06 ^a $\pm$ 0.36	
60 °C	31.7 ^b $\pm$ 0.82	27.5 ^b $\pm$ 0.51	25.68 ^b $\pm$ 0.55	6.91 ^b $\pm$ 0.63	7.77 ^b $\pm$ 0.70	7.18 ^b $\pm$ 0.53	24.05 ^b $\pm$ 0.36	17.47 ^b $\pm$ 0.41	16.21 ^a $\pm$ 0.35	
70 °C	30.44 ^b $\pm$ 0.45	29.01 ^c $\pm$ 0.46	30.2 ^c $\pm$ 0.55	8.7 ^c $\pm$ 0.35	8.21 ^b $\pm$ 0.60	7.6 ^b $\pm$ 0.67	19.83 ^c $\pm$ 0.27	18.84 ^c $\pm$ 0.39	18.92 ^b $\pm$ 0.53	
LSD _{.05}	1.31	0.97	1.06	0.95	1.21	1.22	0.68	0.84	0.84	
PJPP (DAD)	1 m/s	1.5 m/s	2 m/s	1 m/s	1.5 m/s	2 m/s	1 m/s	1.5 m/s	2 m/s	
40 °C	23.31 ^a $\pm$ 0.35	24.04 ^a $\pm$ 0.43	25.59 ^a $\pm$ 0.46	8.82 ^a $\pm$ 0.31	9.42 ^a $\pm$ 0.51	8.13 ^a $\pm$ 0.32	17.26 ^a $\pm$ 0.63	3.88 ^a $\pm$ 0.26	17.32 ^a $\pm$ 0.34	
50 °C	22.41 ^b $\pm$ 0.37	23.88 ^a $\pm$ 0.17	25.38 ^a $\pm$ 0.39	8.52 ^a $\pm$ 0.39	9.51 ^a $\pm$ 0.39	10.03 ^b $\pm$ 0.12	16.38 ^a $\pm$ 0.39	3.56 ^a $\pm$ 0.41	18.73 ^b $\pm$ 0.51	
60 °C	26.87 ^c $\pm$ 0.25	25.79 ^b $\pm$ 0.37	27.35 ^b $\pm$ 0.45	6.04 ^b $\pm$ 0.21	7.62 ^b $\pm$ 0.50	8.18 ^a $\pm$ 0.57	19.94 ^b $\pm$ 0.26	4.56 ^a $\pm$ 0.43	19.08 ^b $\pm$ 0.39	
70 °C	29.17 ^d $\pm$ 0.17	29.02 ^c $\pm$ 0.34	28.21 ^c $\pm$ 0.35	4.92 ^c $\pm$ 0.76	8.05 ^b $\pm$ 0.43	10.86 ^b $\pm$ 0.75	20.75 ^b $\pm$ 0.60	6.09 ^b $\pm$ 0.47	18.16 ^b $\pm$ 0.22	
LSD _{.05}	0.55	0.64	0.78	0.88	0.87	0.94	0.93	0.75	0.71	

* Values marked with same letter are not significantly different (LSD, $p > .05$)

change from 50 to 60 °C. However beyond 60 °C, TPC decreased reaching a value of 31.52 mg GAE/g (db) at 70 °C, exhibiting 118% increase from that of jamun pulp paste. In case of microwave assisted hot air drying of highbush blueberry leaves, Routray et al. (2014) observed that the TPC extraction increased with increase of drying temperature from 45 to 60 °C; however, the TPC decreased with further increase of drying temperature to 75 °C. At 2 W/g, in the present study, within 40–60 °C TPC maintained an average value of 33.06 mg GAE/g (db), followed by increasing to 37.16 mg GAE/g (db) at 70 °C. For samples dried at 3 W/g, TPC increased from 34.78 mg GAE /g at 40 °C to an average value of 40.35 mg GAE/g (db) within 50–70 °C. If compared on discrete data basis, the condition of 3 W/g at 50–70 °C could be used for highest possible TPC. Positive influence of microwave power density on TPC was also observed by Aghilinategh et al. (2015).

In case of VD at 40 °C, irrespective of pressure, samples took long time to attain constant weight, developing fermented smell at the end of drying and hence, were not evaluated for quality. Onwards 50 °C, at each pressure (Table 2 and Fig. 1b), TPC usually increased with increase of temperature. At 260 mmHg, TPC increased linearly

from 20.89 mg GAE/g (db) at 50 °C to 30.2 mg GAE/g (db) at 70 °C, i.e., 44% increase. In case of 160 mmHg, starting from 24.34 mg GAE/g (db) at 50 °C, TPC reached to 29.01 mg GAE/g (db) at 70 °C. For 60 mmHg, TPC increased till 60 °C, followed by insignificant change thereafter. It was also observed that at 50 and 60 °C, as the pressure increased, the TPC of PJPP decreased; the value at 70 °C were comparable for all the pressure levels. The average value of 31.07 mg GAE/g, db obtained at 60–70 °C under 60 mmHg appeared to be the maximum, indicating 115% increase from that of pulp (Table 2).

Considering DAD as shown in Fig. 1c, TPC of samples indicated increasing trend with increase in temperature for all three air velocities viz., 1, 1.5 and 2 m/s. At 1 m/s, there was a slight decrease in TPC when temperature was increased from 40 to 50 °C; at 1.5 and 2 m/s there was no significant change. Onwards 50 °C, TPC increased with increase of temperature. At 40 and 50 °C, TPC is found to be directly proportional to drying air velocities. At higher temperatures of 60 and 70 °C, the difference in TPC reduces, ranging only within 28.21–29.17 mg GAE/g, db at 70 °C.

It is clear from the above discussion that, excepting a few cases (Table 2 and Fig. 1), samples dried at higher

Fig. 1 Relationship between TPC and **a** microwave-convective hot air drying temperature at different power densities, **b** vacuum drying temperature at different pressures, **c** dehumidified air drying temperature at different air velocities

temperatures contained higher TPC. Besides the factors influencing liberation of phenolics, an opposite phenomenon of destruction of phenolics by polyphenoloxidase is also simultaneously operating. High temperature helps to inactivate the enzyme facilitating retention of phenol compounds. Arslan and Özcan (2010) observed that onion slices dried at 70 °C had higher TPC than those dried at 50 °C. Moreover at any W/g, MCD produced higher TPC than VD and DAD for same temperature level. Microwave creates a high vapor pressure and heat from inside the mass, which, possibly, destroys food matrix to a higher degree releasing more phenolics, as compared to VD and DAD.

Effect on MAC

Stability of anthocyanins depend on their structure including the composition of the matrix in which they reside, and various other processing factors, e.g. temperature, time, light and oxygen exposure, pH, and water activity (Busso Casati et al. 2015; Howard et al. 2016). Orjuela-Palacio and Lanari (2016), while studying on freeze dried black currant-yerba mate instant drink powder, observed that in addition to temperature MAC stability was strongly influenced by RH. Under favourable conditions, anthocyanin also forms stable complex with other

polymeric and phenolic compounds (co-pigments) present in the food (Cortez et al. 2017). Release of membrane bound anthocyanin was discussed by Chauhan et al. (2015), while drying *Ficus carica*. The amount estimated by pH differential method is a resultant of the concomitant mechanisms including degradation, copigmentation and anti-copigmentation (Cortez et al. 2017).

Table 2 shows that MAC in PJPP, depending on the drying technique and condition, either increased or decreased, compared to jamun pulp paste (8.12 mg M3G/g, db). In FD, the value was 11.90 mg M3G/g, db (46% increase). Wojdyło et al. (2014) explained this fact as freeze drying release bound phenolics from the cellular matrix, making them more accessible in extraction.

From Fig. 2a and Table 2, it is seen that for each successive increase in temperature of MCD, MAC increased significantly at 1 W/g, and the maximum value obtained at 70 °C was $\approx$ 47% higher than that of jamun pulp paste. In case of other two power density levels, sensitivity was low. At 2 W/g, MAC at 40 °C was 10.50 mg M3G/g, db; for rest of the temperature 50–70 °C, the level came down and an average value of 8.51 mg M3G/g, db was maintained. At 3 W/g, no definite trend was followed. MAC increased from 7.03 mg M3G/g (40 °C) to 9.13 mg M3G/g (50 °C) and again decreased to 7.53–7.79 mg M3G/g for rest of the temperature levels. It can be assumed that for the full

temperature range at 3 W/g an average of 7.87 mg M3G/g was seen, which is less than the jamun pulp paste. MAC of dried highbush blueberry leaves was also highest at 60 °C in the range corresponding to 45–70 °C (Routray et al. 2014). On the other hand, Sengkhampan et al. (2013), observed that anthocyanin content of hot air dried pitaya peel decreased at 70 °C and again increased at 80 °C, compared to that dried at 60 °C. They assumed that it might be due to low drying time at 80 °C. While studying on anthocyanin degradation kinetics of pulps of thermally treated blackcurrant, blueberry and maqui berry, Busso Casati et al. (2017) observed that maqui berry showed highest values of half-life especially at 90 °C.

At 50 °C, VD samples showed MAC of 4.82, 6.33 and 5.27 M3G/g, db for 260, 160 and 60 mmHg, respectively. Such low values may be due to lengthy drying time at this temperature. It may be reiterated that sample dried at 40 °C developed fermented odour. Increase in temperature to 60 °C, increased the MAC at all pressure levels, compared to sample dried at 50 °C. For further increase to 70 °C, increase in MAC (from that of 60 °C) was only noticed at 60 mm Hg, with no significant change for other two pressure levels. In general, the VD operation seemed to be detrimental to the pigment (Table 2, Fig. 2b). Only in two cases, at 160 mm Hg and 60 mm Hg both at 70 °C, there were slightly higher values of 8.21 and 8.7 mg M3G/g, db, i.e., 1 and 7% increase respectively, compared to undried jamun pulp paste. It may be mentioned that Jebbitt and Allwin (2016) dried jamun pulp by vacuum drying at 40, 50 and 60 °C; while estimating total anthocyanin, they obtained 5.10, 6.98 and 6.09 mg cyanidin-3-rutinoside/100 g of dried matter in extracted solution. This indicated increase and then decrease in anthocyanin for sequential increase in drying temperature from 40 to 50, and from 50 to 60 °C.

In case of DAD, at 1 m/s air velocity MAC decreased with increase of temperature from 40 to 70 °C, where beyond 50 °C, the MAC reduced even from the respective content of undried pulp. Similar trend was seen for 1.5 m/s; for 40–50 °C there was no significant change, followed by decrease to 7.62 mg M3G/g, db at 60 °C, with no noticeable change for still higher temperature. At this air velocity also, for 60 °C and onwards MAC reduced compared to the pulp in paste form. The sample dried at 2 m/s of air velocity indicated an average value of 9.3 mg M3G/g, db for the whole range of temperature.

Effect on AA

The PJPP showed, except in a very few cases of operating conditions (vide Table 2), more AA than the jamun pulp paste (12.7 mg BHA/g, db). Rababah et al. (2005) opined that strawberry, peach and apple dried 40.4 °C for 24 h

possessed higher antioxidant capacity (TPC and anthocyanin as well) compared to the fresh ones. Similar observation was reported for hot air dried fig and sundried apricot by Hoxha and Kongoli (2016) and Güclü et al. (2006), respectively. In the present context, the maximum AA (db) observed in MCD, VD, and DAD were 28.63 mg BHA/g, 24.05 mg BHA/g, and 20.75 mg BHA/g, respectively; in FD the value was 16.73 mg BHA/g. Hence, it can be said that MCD was most suitable drying method for AA of jamun pulp. As reviewed by Manganaris et al. (2014), microwave heating resulted in lower losses of antioxidants in raspberries as compared to conventional heating. The effects of various drying conditions used during MCD, VD and DAD on AA are discussed below.

It is seen in Fig. 3a that at 1 W/g, AA increased from 12.12 mg BHA/g (db) at 40 °C to 20.55 mg BHA/g (db) at 50 °C. However, at 60 °C the AA reduced by 21.02% (16.23 mg BHA/g, db) w.r.t 50 °C. At 70 °C, AA again increased to reach 28.63 mg BHA/g, db. At 2 W/g, the drying temperatures from 40 to 60 °C did not affect AA significantly as evaluated by LSD (Table 2), the value being around 18 mg BHA/g (db) in all cases. At 70 °C, the AA increased by almost 28% (23.09 mg BHA/g, db) w.r.t to the lower drying temperatures. At 3 W/g, the AA value increased from 16.3 mg BHA/g (db) at 40 °C to around 18 mg BHA/g (db) at 50 and 60 °C and then again reduced marginally at 70 °C (17.92 mg BHA/g, db). Thus at 70 °C, the AA of PJPP was found to be inversely proportional to power density.

Under 260 mm Hg absolute pressure in case of VD, AA remained around (Table 2; Fig. 3b) 16 mg BHA/g, db for the temperature of 50–60 °C, and increased to 18.92 mg BHA/g, db at 70 °C. At 160 mmHg, AA increased linearly and significantly with each successive increase in temperature from 50 to 70 °C. Madrau et al. (2009) found that antioxidant capacity increased significantly in Cafona apricot as a consequence of increasing drying temperature. However, in case of 60 mmHg (Fig. 3b), AA value increased from 19.04 mg BHA/g (db) at 50 °C to the maximum value of 24.05 mg BHA/g (db) at 60 °C and then again reduced to 19.83 mg BHA/g (db) at 70 °C. While heating pre-dried (at 105 °C) mango seed kernel to still higher temperature from 140 to 200 °C, Soong and Barlow (2004) reported that antioxidant capacity of the extracts made therefrom increased for heating temperature up to 160 °C. However for further heating onwards 160–200 °C, the antioxidant capacity decreased.

The relationship between AA and DAD temperatures at different air velocities are presented in Fig. 3c. At 1 m/s drying air velocity, the AA value remained at average value of 16.8 mg BHA/g (db) within 40–50 °C, followed by increasing to an average value of $\approx$ 20 mg BHA/g (db) for in-between 60 and 70 °C. At 2 m/s, the AA value

Fig. 2 Relationship between MAC and **a** microwave-convective hot air drying temperature at different power densities, **b** vacuum drying temperature at different pressures, **c** dehumidified air drying temperature at different air velocities

obtained at 40 °C was 17.32 mg BHA/ g (db), whereas at 50, 60 and 70 °C, the values were not significantly different, and an average of 18.6 mg BHA/g (db) was maintained. At 1.5 m/s, surprisingly, the values reduced drastically ranging from 3.88 to 6.09 mg BHA/g (db); an average of 4 mg BHA/g (db) was maintained for 40–60 °C while showing 6.09 in the same unit at 70 °C.

Possibly at high temperatures, the increase in AA implies inactivation of oxidative enzymes and/or development of new phenolic compounds, from their precursors, having high antioxidant activity (Que et al. 2008). Regarding the Maillard reaction produces, Carunchia et al. (2015) explained that intermediate compounds in browning reaction increase antioxidant activity by absorbing free radicals. It is also worth mentioning that Prior et al. (2016) studied processing effects on the antioxidant capacity of blueberries using five radicals. According to them, although the total antioxidant capacity did not change, there was a shift in the amount of protection from peroxy radical and hydroxyl radical into protection against singlet oxygen, indicating radical-condition dependent sensitivity of antioxidant. Thus, the exhibited increase in PJPP may be due to one/or more of these reasons.

Selection of the best drying method

The highest value (Table 2) of TPC, MAC and AA obtained in MCD, VD, and DAD, irrespective of drying condition, and the values from FD are shown in Fig. 4. In the figure, MAC, except between MCD and VD, and VD and FD, did not differ significantly for all possible pairs of drying (t test, $p > .05$); TPC and AA, however, were significantly different between any two methods. It is seen that TPC and AA were maximum for MCD, whereas MAC was not very sensitive of drying methods, irrespective of selecting their best conditions. However, in MCD at 70 °C (Table 2), TPC of 41.12 mg GAE/g, db was obtained at 3 W/g, while the AA of 28.63 mg BHA/g, db corresponded to 1 W/g. In spite of the fact that at 70 °C, the TPC obtained at 1 W/g (31.52 mg GAE/g, db) was significantly less compared to that of at 3 W/g (41.12 mg BHA/g, db), MCD at 70 °C and 1 W/g was chosen to achieve the maximum available AA in PJPP, antioxidant being the ultimate benefit derivable from phenolics. At the outset, the selection may appear to be with a sacrifice of some TPC. In this regard, it may be mentioned that in MCD, the Pearson's correlation between AA and MAC (0.7289) was appreciable, while between AA and TPC it was weak (0.2306) (Beldjazia and Alatou, 2016), indicating that

Fig. 3 Relationship between AA and **a** microwave-convective hot air drying temperature at different power densities, **b** vacuum drying temperature at different pressures, **c** dehumidified air drying temperature at different air velocities

Fig. 4 Effect of MCD, VD, FD and DAD on the functional properties of jamun pulp

MAC is the predictor of AA, not the TPC. In case of freeze dried Andes berry extract powder also, Estupiñan et al. (2011) reported good correlation between AA and MAC, while it was poor between AA and TPC. Amakura et al. (2000) stated that DPPH activity in berries may be contributed due to the selected phenolics among the total phenolics

Conclusion

It can be concluded that TPC, AA and MAC of jamun pulp are significantly affected by drying. Among MCD, FD, VD and DAD methods, MCD proved to be most favourable for the retention of the above stated functionalities. Based on the statistical analyses, the MCD condition of 70 °C and 1 W/g was the most suitable for drying of jamun pulp. The TPC, AA and MAC values (db) of the sample dried at the selected condition were 31.52 mg GAE/g, 28.63 mg BHA/g and 11.99 mg M3G/g, respectively. The developed method of MCD can be expected to be used for large scale production of jamun pulp powder.

References

- Aghilinategh N, Rafiee S, Hosseinpour S, Omid M, Mohtasebi SS (2015) Optimization of intermittent microwave-convective drying using response surface methodology. *Food Sci Nutr* 3(4):331–341
- Akyıldız A, Aksay S, Benli H, Kiroğlu F, Fenercioğlu H (2004) Determination of changes in some characteristics of persimmon

- during dehydration at different temperatures. *J Food Eng* 65(1):95–99
- Amakura Y, Umino Y, Tsuji S, Tonogai Y (2000) Influence of jam processing on the radical scavenging activity and phenolic content in berries. *J Agric Food Chem* 48(12):6292–6297
- Arslan D, Özcan MM (2010) Study the effect of sun, oven and microwave drying on quality of onion slices. *LWT Food Sci Technol* 43(7):1121–1127
- Beldjazia A, Alatou D (2016) Precipitation variability on the massif Forest of Mahouna (North Eastern-Algeria) from 1986 to 2010. *IJMSBR* 5(3):21–28
- Busso Casati C, Baeza R, Sanchez V, Catalano A, Lopez P, Zamora MC (2015) Thermal degradation kinetics of monomeric anthocyanins, colour changes and storage effect in elderberry juices. *J Berry Res* 5(1):29–39
- Busso Casati C, Baeza R, Sanchez V (2017) Comparison of the kinetics of monomeric anthocyanins loss and colour changes in thermally treated blackcurrant, maqui berry and blueberry pulps from Argentina. *J Berry Res* 7(2):85–96
- Carunchia M, Wang L, Han JH (2015) The use of antioxidants in the preservation of snack foods. In: Shahidi F (ed) *Handbook of antioxidants for food preservation*, Woodhead Publishing Series in Food Science, Technology and Nutrition, No.276, Elsevier, United Kingdom, pp 447–474
- Chauhan A, Tanwar B, Intelli (2015) Influence of processing on physico-chemical, nutritional and phytochemical composition of *Ficus carica* (fig) fruit. *Res J Pharm Biol Chem Sci* 6(6):1474–1489
- Cortez R, Luna-Vital DA, Margulis D, de Mejia EG (2017) Natural pigments: stabilization methods of anthocyanins for food applications. *Compr Rev Food Sci F* 16(1):180–198
- de Santana RF, Neto ERO, Santos AV, Soares CMF, Lima AS, Cardoso JC (2015) Water sorption isotherm and glass transition temperature of freeze-dried *Syzygium cumini* fruit (jambolan). *J Therm Anal Calorim* 120(1):519–524
- Estupiñán DC, Schwartz SJ, Garzón GA (2011) Antioxidant activity, total phenolics content, anthocyanin, and color stability of isotonic model beverages colored with Andes berry (*Rubus glaucus* Benth) anthocyanin powder. *J Food Sci* 76(1):S26–S34
- Forbes-Hernandez TY, Gasparrini M, Afrin S, Bompadre S, Mezzetti B, Quiles JL, Giampieri F, Battino M (2016) The healthy effects of strawberry polyphenols: which strategy behind antioxidant capacity? *Crit Rev Food Sci Nutr* 56:S46–S59
- Giampieri F, Alvarez-Suarez JM, Mazzoni L, Forbes-Hernandez TY, Gasparrini M, Gonzalez-Paramas AM, Santos-Buelga C, Quiles JL, Bompadre S, Mezzetti B, Battino M (2014) An anthocyanin-rich strawberry extract protects against oxidative stress damage and improves mitochondrial functionality in human dermal fibroblasts exposed to an oxidizing agent. *Food Funct* 5(8):1939–1948
- Güclü K, Altun M, Özyürek M, Karademir SE, Apak R (2006) Antioxidant capacity of fresh, sun-and sulphited-dried Malatya apricot (*Prunus armeniaca*) assayed by CUPRAC, ABTS/TEAC and folin methods. *Int J Food Sci Technol* 41(1):76–85
- Howard LR, Brownmiller C, Mauromoustakos A, Prior RL (2016) Improved stability of blueberry juice anthocyanins by acidification and refrigeration. *J Berry Res* 6(2):189–201
- Hoxha L, Kongoli R (2016) Influence of drying process on phenolic content and antioxidant activity of two different autochthonous Albanian fig varieties. *Sci Pap Ser A Agron* 59:509–514
- İzli G (2017) Total phenolics, antioxidant activity, colour and drying characteristics of date fruit dried with different methods. *Food Sci Technol (Campinas)* 37(1):139–147
- Jebbita SR, Allwin SJ (2016) Antioxidant activity, total phenol, flavonoid and anthocyanin contents of jamun (*Syzygium cumini*) pulp powder. *Asian J Pharm Clin Res* 9(2):361–363
- Madrau MA, Piscopo A, Sanguinetti AM, Del Caro A, Poiana M, Romeo FV, Piga A (2009) Effect of drying temperature on polyphenolic content and antioxidant activity of apricots. *Eur Food Res Technol* 228(3):441–448
- Manganaris GA, Goulas V, Vicente AR, Terry LA (2014) Berry antioxidants: small fruits providing large benefits. *J Sci Food Agric* 94(5):825–833
- Mazzoni L, Perez-Lopez P, Giampieri F, Alvarez-Suarez JM, Gasparrini M, Forbes-Hernandez TY, Quiles JL, Mezzetti B, Battino M (2016) The genetic aspects of berries: from field to health. *J Sci Food Agric* 96(2):365–371
- Orjuela-Palacio JM, Lanari MC (2016) The impact of moisture sorption properties on the color and bioactives concentrations of black currant-herb mate instant drinks. *J Berry Res* 6(3):303–319
- Periyathambi R (2007) Jamun-the potential untapped. *Horticulture* 1:30–32
- Prior RL, Sirtara M, Chang T (2016) Multi-radical (ORAC_{MR5}) antioxidant capacity of selected berries and effects of food processing. *J Berry Res* 6(2):159–173
- Que F, Mao L, Fang X, Wu T (2008) Comparison of hot air-drying and freeze-drying on the physicochemical properties and antioxidant activities of pumpkin (*Cucurbita moschata* Duch.) flours. *Int J Food Sci Tech* 43(7):1195–1201
- Rababah TM, Ereifej KI, Howard L (2005) Effect of ascorbic acid and dehydration on concentrations of total phenolics, antioxidant capacity, anthocyanins, and color in fruits. *J Agric Food Chem* 53(11):4444–4447
- Routray W, Orsat V, Garipey Y (2014) Effect of different drying methods on the microwave extraction of phenolic components and antioxidant activity of highbush blueberry leaves. *Dry Technol* 32(16):1888–1904
- Santhakshmy S, Bosco SJD, Francis S, Sabeena M (2015) Effect of inlet temperature on physicochemical properties of spray-dried jamun fruit juice powder. *Powder Technol* 274:37–43
- Sehwag S, Das M (2014) Nutritive, therapeutic and processing aspects of Jamun, *Syzygium cumini* (L.) Skeels—an overview. *Indian J Natl Prod Res* 5(4):295–307
- Sengkhamparn N, Chanshotikul N, Assawajitpukdee C, Khamjae T (2013) Effects of blanching and drying on fiber rich powder from pitaya (*Hylocereus undatus*) peel. *Int Food Res J* 20(4):1595–1600
- Shahnawaz M, Sheikh SA (2008) Analytical description on stickiness, shelf life and colour of Jamun (*Eugenia jambolana* L.) fruit powder. *Pak J Agric Agric Eng Vet Sci* 24(1):44–47
- Sonawane SK, Arya SS (2015) Effect of drying and storage on bioactive components of jambhul and wood apple. *J Food Sci Technol* 52(5):2833–2841
- Soong YY, Barlow PJ (2004) Antioxidant activity and phenolic content of selected fruit seeds. *Food Chem* 88(3):411–417
- Vuthijumnok J, Molan AL, Heyes JA (2013) Effect of freeze-drying and extraction solvents on the total phenolic contents, total flavonoids and antioxidant activity of different Rabbiteye blueberry genotypes grown in New Zealand. *IOSR-JPBS* 8:42–48
- Wojdyło A, Figiel A, Lech K, Nowicka P, Oszmiański J (2014) Effect of convective and vacuum-microwave drying on the bioactive compounds, color, and antioxidant capacity of sour cherries. *Food Bioprocess Technol* 7(3):829–841