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Comparative evaluation of apple juice concentrate and spray dried apple powder for nutritional, antioxidant and rheological behaviour

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RESEARCH ARTICLE

Abstract

A higher proportion of apples produced in Jammu and Kashmir (J&K) is wasted due to pre-harvest drop which can be utilised for the development of various value-added products. Although apples are mostly processed into single-strength juice and concentrate in J&K, there is a need to introduce new apple-based products. Thus, a study was conducted to develop apple powder from apple juice concentrate (AJC) using spray drying technique. AJC was evaluated for physico-chemical composition, phytochemical constituents, flow behaviour, thermophysical aspect, flavour compounds and viscosity, and was compared with spray dried apple powder (SDAP) developed from the same concentrate. Results inferred from the comparison of AJC with SDAP revealed that SDAP recorded lower moisture content (2.91%) and a_w (0.217) but significantly ($p < 0.05$%) higher fibre (0.07%), carbohydrate content (95.28%) and total soluble solids (TSS) (95.78°Brix). An increase in phenolic, anthocyanin and antioxidant potential of SDAP was also recorded. Viscosity analysis demonstrated that AJC possessed content viscous nature with a viscosity of 552.8 mPa-s which was well supported by rheogram depicting Newtonian behaviour of the concentrate. Further, Fourier transform infrared spectroscopy (FTIR) analysis indicated the presence of major ester, aromatic acids, and primary and secondary alcohol groups which were further revealed by GC-MS that detected the presence of major flavour compounds (2-methyl butyl acetate) in AJC. Flow behaviour of SDAP (after reconstitution) depicted non-Newtonian (shear thinning) behaviour with low viscosity. FTIR and GC showed the presence of all chemical constituents already present in AJC thereby confirming efficiency of spray drying process. The results from the comparison study inferred that AJC can prove as an excellent raw material for spray drying after dilution.

Keywords: apple concentrate; thermal conductivity; flow behaviour; FTIR; GC-MS; shear thinning

Introduction

A pragmatic shift has been observed in the preference of consumers from highly processed ready to eat foods to natural or minimal processed foods due to impact of nutrition on overall health of humans. In recent times, the role of phytochemicals, especially the plant-based phytochemicals, in preclusion and elimination of various dreaded human diseases have been proven by various researchers and scientists (Ginwala *et al.*, 2019; Rupasinghe, 2020). Among the different classes of phytochemicals, flavonoids are considered more potent in

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amelioration of various diseases. Thus, an increased interest has been observed in their biochemistry and mechanism of action. These hydroxylated phenolic substances (Kumar and Pandey, 2013) exert their protective action by supressing oxygen and acting as antioxidative compounds similar to ascorbic acid and tocopherols. "An apple a day keeps doctor away," a famous quote of Hippocrates, is believed to have originated from the medicinal properties of its biologically active polyphenolic compounds. Apple phenolics are known for their antioxidative, chemo-preventive, anti-neurodegenerative and anti-obesity properties due to the presence of ample amounts of phytochemicals such as epicatechin, quercetin galactoside, quercetin glucoside, chlorogenic acid, quercetin xyloside, quercetin rhamnoside, phloretin glucoside, quercetin arabinoside, phloretin xylogalactoside, flavan-3-ols and flavanols, procyanidin B2 and procyanidins (Jan *et al.,* 2016). Thus, due to rich phytochemical composition of apples, the consumption of apples and apple-based product has increased manifold.

Apple, a temperate fruit of intermittent Glycemic Index (Foster-Powell *et al.,* 2002), is undoubtedly the most relished fruit in the world because of its exuberant flavour and exotic taste (Sharma *et al.*, 2014). A fruit of pomaceous origin, it belongs to the *Malus domestica* species in the Rosacea family. It is one of the most widely cultivated tree fruits and is the fundamental fruit crop among all the horticulture crops of Jammu and Kashmir (Darzi, 2016). There are hundreds of apple varieties known to exist in the world. The main varieties cultivated in India are Red Delicious, Golden Delicious, Red Cort and Ida Red. Some lesser-known indigenous varieties of Jammu and Kashmir are Ambri, Kesari, Hazratbali, Razakwari, Chemora and Maharaji (Singh *et al.,* 2017). These varieties differ in colour, size, shape, flavour and taste. Jammu and Kashmir, the leading producer of apples in India, has a production capacity of 10.2 tons/Ha, which is significantly higher than Himachal Pradesh (7 tons/Ha) and other minor apple-producing states of the country (Anonymous, 2014). However, it is far below the international production level of 30 tons/Ha.

The state of Jammu and Kashmir is one of the richest apple-producing states of India contributing about 65% of the total domestic production (Bhat *et al*., 2021). However, only 1% of apples produced in Jammu and Kashmir are processed into products. Nearly 30% of the total produce of apple crop is wasted due to pre-harvest drop which accounts for loss of three lakh MT of apples worth Rs. 600 crore (Khan *et al*., 2020). Apples are mostly processed into single-strength juice and concentrate. An insignificant proportion is used for the production of jams, jellies, candies, etc. (Darzi, 2016). Moreover, these manufactured products are substandard and does not fulfil the international standards. Literature survey has shown that apple concentrate from Kashmir valley haven't been subjected to spray drying in order to develop spray dried apple powder (SDAP).

Spray drying is a rapid, continuous, cost-effective, reproducible and scalable process for making dry powders from a fluid material by atomization through an atomizer into a hot drying gas medium, usually air. The drying method has several advantages over its counterparts in terms of yield, cost, energy, throughput and reduction of exposure time of products to high temperatures (Sosnik and Seremeta, 2015). Thus, the present research work was undertaken to evaluate the suitability of available apple concentrate for spray drying operation and later compare the same with the developed SDAP in order to confirm its feasibility to undergo spray drying.

Material and Methods

Selection of material

A commercially available apple juice concentrate (AJC) of local brand was procured from Kashmir valley. Analytical grade chemicals were used for this study.

Physico-chemical analysis of material

Proximate composition

The procedures laid down by AOAC (2005) were pursued for the estimation of moisture, crude protein, crude fibre, volatile ash and crude lipid content. Carbohydrate content was determined by the subtraction method.

Mineral content, total sugar, reducing and non-reducing sugar

Standard AOAC (2005) procedures were followed for the determination of mineral content in the sample using atomic absorption spectrometer. Total sugar content and reducing sugar were determined by following Fehling's titration method (ICUMSA, 1994). Non-reducing sugar content was obtained by calculating the difference between total sugar and reducing sugar contents.

Total soluble solids, a_w and ascorbic acid content

The method provided by AOAC (2005) was used for determining total soluble solids (TSS) of the sample. TSS was obtained directly by digital refractometer (Atago RX-1000). Vitamin C content was estimated by 2,6-diclorophenolindophenol dye indicator method as per AOAC (2005) procedure.

Water activity analyser (Pre-Aqua Lab, Water Activity Analyzer) was employed for the estimation of sample water activity.

Titrable acidity and pH

Titrable acidity was estimated by titration method laid down by AOAC (2005). Sample titration was carried out against 0.1M NaOH with phenolphthalein as indicator.

pH of the sample was determined with the help of an electronic pH meter (model Labindia).

Phytochemical content and antioxidative potential of apple concentrate

Sample extraction

For sample preparation, a mixture of 80% acetone was used with a ratio of 1:10 (sample: solvent). Afterwards, the sample was mixed with acetone solution and placed in a shaking incubator (Certomat 1S, Sartorius) for 90 min at 20°C. Once the sample was completely dissolved, the crude extract was transferred to centrifugal tubes for centrifugation at 3000 rpm (Hettich-Zentrifugen, Germany) for 15 min. The clear liquid was syphoned out and was stored at –20°C for later use.

Total phenolic content

The sample extract was analysed through the Folin-Ciocalteau assay using the AOAC (2005) method for total phenolic content (TPC). The spectrophotometric method was employed and sample absorption was recorded on a UV-Vis spectrophotometer at 765 nm. (Manti Lab MT-137A Double Beam UV-VIS Spectrophotometer). The results were reflected in mg GAE/ 100 g.

Total flavonoid content

The total flavonoid content (TFC) of sample was estimated by treating it against aluminium trichloride and then recording absorbance at 415 nm in a UV-VIS spectrophotometer (Manti Lab MT-137A Double Beam UV-VIS Spectrophotometer) against deionised water as blank (AOAC, 2005). Results were denoted in mg quercetin equivalent (mgQE/100g) per 100g of sample weight.

Total anthocyanin content

The total anthocyanin content (TAC) measurement was conducted by a pH differential method (Giusti and Jing, 2007). The total anthocyanin material was measured using the formula below and was written as mg cyanidin 3-glucoside equivalent (Cy3-GE)/g fw.

$$TAC = \frac{A \times MW \times DF \times V \times 100}{€ \times L \times W} \quad (1)$$

$$A = (A_{510nm} - A_{700nm})_{pH=1} - (A_{510nm} - A_{700nm})_{pH=4.5}$$

Where A = absorbance
MW = molecular weights of cyanidin 3-glucoside (449.2)
molar absorptivity (€) = 26,900
DF = Dilution factor
V = Volume
W = Sample weight and
L = Path length

Spray drying

Spray drying of apple fruit juice slurry was carried out as per the method described by Sarabandi *et al.* (2018) with some modifications. Experiments were performed under various combinations of operating parameters using co-current air flow type pilot scale spray dryer fitted with two-fluid nozzle atomizer having orifice diameter of 0.5 mm (SM Scientech, Kolkata). The drying chamber was made of food-grade stainless steel and measured 90 cm in diameter and 35 cm in height. It had a water evaporation capacity of 3 L/h and was equipped with a dehumidified air drying system via a stretchy air duct. Dehumidified air was supplied to the drying chamber through spray drying air inlet. Compressed air was also dehumidified before entering through the nozzle. The feed slurry was fed to the dryer by means of peristaltic pump. Operating parameters viz inlet air temperature, feed flow rate, maltodextrin, gum arabic concentration and feed TSS were taken as 160°C, 350 rpm, 4%, 6% and 15° Brix, respectively (after optimisation in previous study) while the outlet temperature, atomizer disc speed, air flow rate and air pressure were kept constant at 80°C, 18,000 rpm, 25 m^3/h and 0.80 mPa, respectively. After the completion of each experimental run, the powder was collected from the cyclone and the cylindrical parts of the dryer chamber by lightly sweeping the chamber wall. The powders were then packed in polyethylene bags and stored in desiccator for further analysis.

Flavour determination

GC-MS experiment was conducted for the identification of flavour compounds in AJC and SDAP by following the procedure laid down by Davies (1990). The gas chromatograph (Varian CP 3800) was fitted with a DB-5 capillary column (30 m × 0.25 mm × 0.25 µm) and a Varian Saturn 2000 ion trap mass detector. Supelco SPME devices coated with polydimethylsiloxane (PDMS, 100 µm) were used to extract the sample into headspace. Before commencing the experiment, sample (apple concentrate) was gushed into a 300 mL glass beaker and later on sealed with aluminium foil. To it, PDMS was inserted and allowed an equilibration period of 60 min. Once sample extraction was done, the prepared sample was injected on to the head of GC-MS injector port system. Injector and transfer line temperatures were set at 250°C and 240°C, respectively, while oven temperatures were programmed to increase from 60°C to 240°C at 3°C/min. and the rate of transfer of carrier gas (helium) through column was kept constant (1 mL/min). By comparing their retention times with those of pure standards of detected compounds, the identification of the sample constituents was carried out. In addition, their linear retention indices (LRI) were matched by machine matching software (NIST 98 and Adams) and homemade library mass spectra, and MS literature results, relative to a series of n-hydrocarbons.

Rheology analysis of apple concentrate

Viscosity analysis

Viscosity measurements were carried out using digital viscometer model NDJ-5S (Shanghai Drawell Scientific Instruments Co., Ltd). The viscometer was equipped with a spindle type 2 that rotated in the sample contained in a 50 mL glass. The speed of the spindle was kept constant at 6 rpm which generated a torque of 55.3%. The sample temperature was kept constant at 20°C. All viscosity measurements were performed in triplicate and average measurements were taken and registered in mPa/s (Sharma *et al.,* 2014).

Flow behaviour

The Brookfield Digital Rheometer, Model HA DVIII Ultra (Brookfield Engineering Laboratories Inc.) was used for the determination of flow properties (shear stress, shear rate) of apple concentrate and apple powder. The samples were kept in small sample adaptor which was placed in a thermostatic water bath, to maintain the pre-requisite temperature and also provided with a viscometer. For the measurement, the SC4-21 spindle was chosen. Rheological parameters of sample were analysed at constant conditions of sample temperature (20°C), spindle shear rate (9.3–93 s^{-1}) and sample concentration (70° Brix) (Brookfield manual, 1998).

Thermophysical behaviour

Density

The density of apple concentrate was measured by pycnometry method. Sucrose solution of known density was used to calibrate the pycnometer (Bayindirli, 1992). Bulk density (BD) of SDAP was determined using the formula

$$BD = \left(\frac{kg}{m3}\right) = \frac{Weight}{Volume} \tag{2}$$

Specific heat and thermal conductivity

Differential scanning calorimeter (DSC model Q100, TA instrument), equipped with data logger and software for the treatment of data (Specialized Design Office of Instrument Making, Pushchino, Russia), was used for determination of specific heat and thermal conductivity. Before the commencement of experiment, helium gas was passed through containment cells to prevent moisture condensation. The instrument was calibrated before measurement using synthetic sapphire α-Al_2O_3 as a specific heat standard reference material (Sabbah *et al.,* 1999). The measurements were carried out as per protocols laid down by DSC (Mykhailyk *et al.,* 2002). Sample, pre-heated to 40–70°C, was placed in an empty container in comparison cell and standard reference material was placed in operating cell. The specific heat and thermal conductivity were estimated from the characteristic DSC curves.

Fourier Transform Infrared Spectroscopy

The quality and authenticity of AJC and SDAP was assessed through Fourier transform spectroscopy using an IRPrestige-21 (Shimadzu) spectrophotometer equipped with Fourier transform infrared spectroscopy (FTIR) Silver Gate module and IR solution software. The program includes data collection and processing modules, quantitative analysis, generation of own spectrum libraries, identification of compounds by means of own and normal spectrum libraries, conversion of spectrum file formats, processing of microscopic images and IR spectroscopy bibliography. Sample spectra were collected in the range of 500–4500 cm^{-1}, slot width 4 cm^{-1}, magnification 1 and number of scans 40 (Golubtsova, 2017).

Results and Discussion

The proximate composition, phytochemical components, thermophysical behaviour and other physico-chemical constituents of AJC and SDAP are presented in Tables 1–4.

Nutritional composition of AJC and SDAP

Proximate composition and mineral analysis of SDAP

Rich nutrient composition of natural fruit powder is its major advantage over synthetic counterpart. Proximate composition and mineral profile of AJC and SDAP are depicted in Table 1. Moisture content of SDAP and AJC differed significantly ($p < 0.05$) with each other. SDAP

Table 1. Proximate composition and mineral analysis of SDAP and AJC.

Parameters (%)	SDAP	AJC
Moisture	2.91 ± 0.04[a]	28.8 ± 0.02[b]
Crude Protein	0.37 ± 0.03[a]	0.39 ± 0.01[a]
Crude Fat	0.06 ± 0.02[a]	0.1 ± 0.04[b]
Crude fibre	0.07 ± 0.02[a]	0.01 ± 0.01[b]
Ash	1.31 ± 0.12[a]	1.2 ± 0.2[b]
Carbohydrate	95.28 ± 0.34[a]	69.5 ± 0.22[b]
Mineral analysis (mg/100 g)		
Potassium	146 ± 0.07[a]	121 ± 0.11[b]
Phosphorus	1.15 ± 0.01[a]	0.11 ± 0.08[b]
Magnesium	94.22 ± 0.25[a]	80.17 ± 0.14[b]
Iron	26.74 ± 0.07[a]	14.44 ± 0.21[b]
Calcium	207.38 ± 0.22[a]	164.5 ± 0.33[b]
Sodium	34.33 ± 0.12[a]	28.4 ± 0.06[b]
Zinc	1.23 ± 0.02[a]	0.7 ± 0.01[b]

Values in the table are presented as mean ± SD. Values with different superscripts are statistically different ($p < 0.05$).
Abbreviations: AJC, apple juice concentrate; SDAP, spray dried apple powder.

Table 2. Physico-chemical composition of SDAP and AJC.

Parameters	SDAP	AJC
Water activity	0.217 ± 0.04[a]	0.77 ± 0.06[b]
TSS (°Brix)*	95.78 ± 0.33[a]	70 ± 0.4[b]
Titrable acidity (% malic acid)	1.70 ± 0.05[a]	1.31 ± 0.04[b]
pH	3.56 ± 0.01[a]	3.82 ± 0.03[b]
Total sugar content (%)	67.51 ± 0.2[a]	64.76 ± 0.4[b]
Reducing sugar content (%)	61.79 ± 0.5[a]	59.35 ± 0.6[b]
Non-reducing sugar content (%)	5.72 ± 0.1[a]	5.41 ± 0.2[a]
Ascorbic acid content (mg/100 g)	9.37 ± 0.07[a]	12.8 ± 0.04[b]

Values in the table are presented as mean ± SD. Values with different superscripts are statistically different ($p < 0.05$).
Abbreviations: AJC, apple juice concentrate; SDAP, spray dried apple powder; TSS, total soluble solids.
*Determined in SDAP after reconstitution

Table 3. Phytochemical analysis of SDAP and AJC.

Parameters	SDAP	AJC
Anthocyanin content (mg/100 g)	2.58 ± 0.03[a]	2.49 ± 0.07[a]
Total phenolic content (mg GAE/100 g)	3.82 ± 0.3[a]	3.65 ± 0.1[a]
Total flavonoid content (mg QE/100 g)	1.77 ± 0.05[a]	1.95 ± 0.1[b]
Antioxidant activity (%DPPH inhibition)	14.88 ± 0.06[a]	13.26 ± 0.05[b]

Values in the table are presented as mean ± SD. Values with different superscripts are statistically different ($p < 0.05$).
Abbreviations: AJC, apple juice concentrate; SDAP, spray dried apple powder.

Table 4. Thermo-physical behaviour of SDAP and AJC.

Parameters	SDAP	AJC
Viscosity (mPa/s)	5.15[a] ± 0.34	552.8 ± 2.11
Density (kg/m^3)	314.1 ± 0.14[a]	1.4 ± 0.06
Specific heat (KJ/kg°C)	2.24 ± 0.0^2 a	2.09 ± 0.0^{3}a
Thermal conductivity (W/m°C)	0.610 ± 0.05[a]	0.37 ± 0.04[b]

Values in the table are presented as mean ± SD. Values with different superscripts are statistically different ($p < 0.05$).
Abbreviations: AJC, apple juice concentrate; SDAP, spray dried apple powder.
*Determined in SDAP after reconstitution

recorded lower moisture content (2.91%) as compared to AJC (28.8%). During spray drying, high rate of heat transfer between particles and the drying medium is achieved which acts as greater driving force for evaporation consequently reducing moisture content of final powder (Goula and Adamopoulos, 2004). The values for moisture content were more or less similar to the values reported by Sarabandi *et al.* (2018) for apple powder. Crude protein content (0.37%) of SDAP was low but comparable to AJC (0.39%) due to incorporation of gum arabic as carrier agent. Gum arabic contains 2% protein in the form of protein polysaccharide complex, thereby enhancing final protein content (Mouecoucou *et al.,* 2003). Furthermore, although thermal treatments are known to cause reduction in protein content of samples due to denaturation process (Grabowski *et al.,* 2008), the shorter contact time between the heating medium and feed material during spray drying leads to insufficient denaturation, thus enhancing overall protein content (Chen *et al.*, 2021). Crude fibre and ash content of SDAP were recorded as 0.07% and 1.31% respectively. Increase in fibre and ash content of SDAP as compared to AJC might be due to the removal of moisture that in turn may have enhanced the concentration of these constituents (Morris *et al.*, 2004). Furthermore, addition of gum arabic might have led to significant ($p < 0.05$) increase in fibre content of SDAP (Subtil *et al.*, 2014). A significant ($p < 0.05$) reduction in fat content of SDAP was recorded. Spray drying leads to particle atomization, thereby increasing surface area and consequently enhancing exposure of feed particles to oxidation and degradation reactions hence, reducing lipid content (Grabowski *et al.,* 2008). Carbohydrate content of SDAP was recorded significantly ($p < 0.05$) higher than AJC probably due to addition of maltodextrin and gum arabic (Subtil *et al.*, 2014). In addition, reduction in moisture content may have subsequently increased the carbohydrate content of SDAP. The result for proximate composition is in concomitance with those reported by Muzaffar *et al.* (2016) for spray dried tamarind powder.

Mineral analysis

A significant ($p < 0.05$) increase in potassium (146 mg/100 g), phosphorus (1.15 mg/100 g), magnesium (94.22 mg/100 g), iron (26.74 mg/100 g), calcium (207.38 mg/100 g), sodium (34.33 mg/100 g) and zinc (1.23 mg/100 g) content of SDAP was observed as compared to AJC due to their heat stable nature and concentration effect due to moisture removal (Karimian-Khosroshahi *et al.*, 2016). Furthermore, the presence of minerals in trace amounts in the carrier agents (maltodextrin and gum arabic) might have significantly ($p < 0.05$) increased minerals content of SDAP (Musa *et al.*, 2018). Grabowski *et al.* (2008) also reported comparable mineral content of spray dried sweet potato powder and its puree.

Physico-chemical analysis

Table 2 illustrates the physico-chemical constituents of SDAP and AJC.

Water activity and TSS

SDAP recorded significantly ($p < 0.05$) high TSS and low water activity. Water activity and TSS of SDAP were recorded as 0.217 and 95.78%. Spray drying causes reduction in moisture content of powder (Goula and Adamopoulos, 2004) which subsequently reduced water activity of the resultant powder. Furthermore, significant ($p < 0.05$) increase in TSS of powder might be due to incorporation of carrier agent (Mishra *et al.*, 2017). The values for water activity and TSS are in concomitance to those reported by Sarabandi et al. (2018).

Total sugar, reducing sugar and non-reducing sugar content

A significant ($p < 0.05$) but comparable increase in total sugar and reducing sugar content of SDAP was observed as compared to AJC. This might probably be due to the addition of carrier agents and concentration effect (Subtil *et al.*, 2014). However, some of the sugars are lost through caramelisation and Maillard reactions (Songchai *et al.,* 2019).

Titrable acidity, pH and ascorbic acid

SDAP recorded significantly high acidity (1.70% malic acid) and high pH (3.96) but low ascorbic acid content (9.37 mg/100 g) in comparison to AJC probably due to thermal degradation of ascorbic acid and other organic acids. Both ascorbic acid and its oxidised form, dehydroascorbic acid, contribute to vitamin C activity. However, further hydrolysis of dehydroascorbic acid to 2,3- diketogulonic acid leads to loss of vitamin C activity. Rate of dehydroascorbic acid hydrolysis is temperature dependent and increases with increase in temperature (Gregory, 1996). Grabowski *et al.* (2008) also reported loss of ascorbic acid in spray dried sweet potato powder.

Phytochemical behaviour of AJC and SDAP

Table 3 shows phytochemical behaviour of AJC and SDAP.

TPC and TFC

TFC of SDAP (1.77 mgQE/100 g) was significantly ($p < 0.05$) lower than AJC (1.95 mgQE/100 g) due to the heat liable nature of these flavonoids. However, the degradation was not pronounced which implies that carrier material have efficiently encapsulated them during spray drying and thus provided sufficient protection to these compounds against thermal degradation on application of heat. Muzzaffar *et al.* (2016) also reported lower TFC values for spray dried tamarind powder in comparison to fresh tamarind puree. Anthocyanin content of SDAP (2.58 mg/100 g) was relatively higher than AJC (2.49 mg/100 g) possibly due to their heat stable nature (Saikia *et al.,* 2014). Moreover, removal of moisture led to the concentration of the bioactive compounds in some cases when compared with that of the raw samples (Saikia *et al.,* 2014).

A non-significant increase in TPC of SDAP (3.82 mgGAE/100 g) was observed as compared to AJC (3.65 mgGAE/100 g). In some cases, application of heat results in formation of phenolic aglycons by cleaving of phenolic-sugar glycosidic bonds. Phenolic aglycons have better reactivity to Folin-Ciocalteu Reagent than their sugar-bonded precursors, thereby increasing TPC (Singleton *et al.*, 1999).

Antioxidant potential

Phenols and flavonoids contribute to scavenging activity of apple powder by electron transfer and hydrogen donating mechanisms (Muzaffar *et al.*, 2016). Thus, phenolic and flavonoid content of apple powder are a symbol of its nutraceutical properties due to their antioxidant potential (Khalid *et al.*, 2017). SDAP (14.88%DPPH inhibition) recorded significantly ($p < 0.05$) higher antioxidant activity as compared to AJC (13.26%DPPH inhibition) contrary to the results of Saika *et al.* (2014) that reported a decrease in antioxidant potential of spray dried water melon powder. Few times, oxidation reactions succeed drying which results in the formation of oxidised phenolic compounds with higher antioxidant activity than non-oxidised polyphenols (Mrkic *et al*., 2006). In addition, formation of Maillard products at high temperature also contribute to increased antioxidant activity as they are known to possess scavenging activity alone or in combination with other natural phenolic compounds (Nicoli *et al*., 1997). Therefore, in some cases, although loss of TPC was observed, DPPH values increase.

Thermophysical behaviour of AJC and SDAP

Thermophysical behaviour of AJC and SDAP is illustrated in Table 4. Thermal properties of food are studied to facilitate the development of efficient heating, drying and refrigeration system and to reduce energy consumption and process times for refrigerating, freezing, heating or drying of food materials (Becker and Fricke, 2003).

Viscosity measurement

Viscosity is an important factor that governs the concentration of juices, especially in the production of high-density concentrates as the efficiency of operation degrades when the product becomes highly viscous (Magerramov *et al*., 2007). Moreover, viscosity of the feed material is important for its efficient atomization and subsequent drying during spray drying operation (Tonon *et al*., 2008). High viscosity leads to larger particles and reduced drying rate as higher viscosity solution will require greater energy to attain required flow rates from the atomizer and higher viscosity limits the turbulent flow available for breakup of the ejected stream into individual particles (Davanlou *et al*., 2015). Furthermore, infeed viscosity affects the retention of flavour during drying by influencing circulation currents within the drying droplet and time to the formation of discrete droplets. Low viscosity facilitates internal mixing of particles during drying and thus, delays formation of a semipermeable surface. This delay permits greater flavour losses during early drying, thus enhanced infeed viscosity should favour volatile retention. However, excessive viscosity retards formation of discrete particles during atomization which promotes volatile losses. Thus, there is an optimum infeed viscosity for the retention of volatiles (Reineccius *et al*., 2004). Hence, AJC can be subjected to spray drying operation after dilution only in order to obtain desirable particle size and maximum flavour retention.

Viscosity of apple concentrate was recorded to be 552.8 mPa·s (Table 4). A viscosity of 2.3 pas was reported by Entsar (2016) for apple puree at 10°C. Viscosity of SDAP juice prepared after reconstitution (25% in 100 mL of water) was recorded as 5.15 mPa which was significantly ($p < 0.05$) low as compared to AJC. The viscosity of pome fruit (apple) gets highly affected by the solid content (sugars and organic acid). The more the solids in the concentrate, the greater is the viscosity. As the concentration of sugar increases, more viscous solution is obtained due to the formation of hydrogen bonds with hydroxyl groups (Bayindirli, 1992). Viscosity of juices is dependent on intermolecular forces between molecules and water-solute interactions, which result from the strength of hydrogen bonds and intermolecular spacing (Manjunatha *et al*., 2012).

Density

Density of AJC was found to be 1.4 kg/m^3. Ramos and Ibarz (1998) reported density of apple puree around 1.50 kg/m^3. BD of SDAP (314.1 kg/m^3) was found significantly ($p < 0.05$) higher than the density of AJC. Spray drying leads to formation of dry, hard particles with reduced particle size and increased density (Santos *et al.,* 2017).

Thermal conductivity (K)

Thermal conductivity of AJC was found to be 0.37 W/m°C. A thermal conductivity value of 0.56 W/m°C and 0.40 W/m°C were reported by Fellows (2009) and Frandas and Bicanic (1999) for apple puree, respectively.

SDAP recorded significantly ($p < 0.05$) higher thermal conductivity values, that is, 0.610 W/m°C as compared to AJC. Formation of small particles during atomization yielded enhanced BD and consequently led to significant increase in the thermal conductivity of SDAP (Santos *et al*., 2017).

Specific heat (Q)

Specific heat of apple concentrate was recorded as 2.09 kJ/kg°C which was statistically similar to that of SDAP (2.24 kJ/kg°C). Fellows (2009) reported a specific heat value of 3.59 kJ/kg°C for fresh apple. The decrease in specific heat value for apple concentrate can be explained by the presence of large proportions of water-soluble fraction and absence of insoluble fraction due to depectinization process (Ziegler and Rizvi, 1985).

Thus, low thermal conductivity and high specific heat values will facilitate drying operation and ultimately will result in less consumption of heat energy.

Fourier transform infrared spectroscopy

FTIR analysis was done to study the micromolecular composition of AJC and SDAP. FTIR spectra of AJC are shown in Figure 1A. The predominant absorption peak was observed at a frequency range of 3280 cm^{-1}. Firdaus *et al.* (2017) also reported a maximum absorbance band at 3410 cm^{-1} for water extract of apple. Absorption bands from 3000 to 3500 cm^{-1} is the characteristic of vibrations

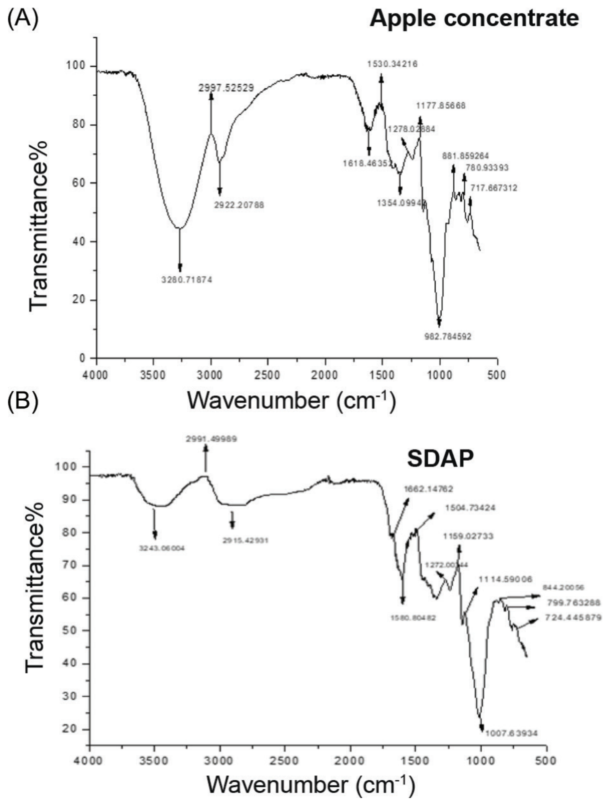

Figure 1. FTIR spectra of (A) AJC and (B) SDAP.

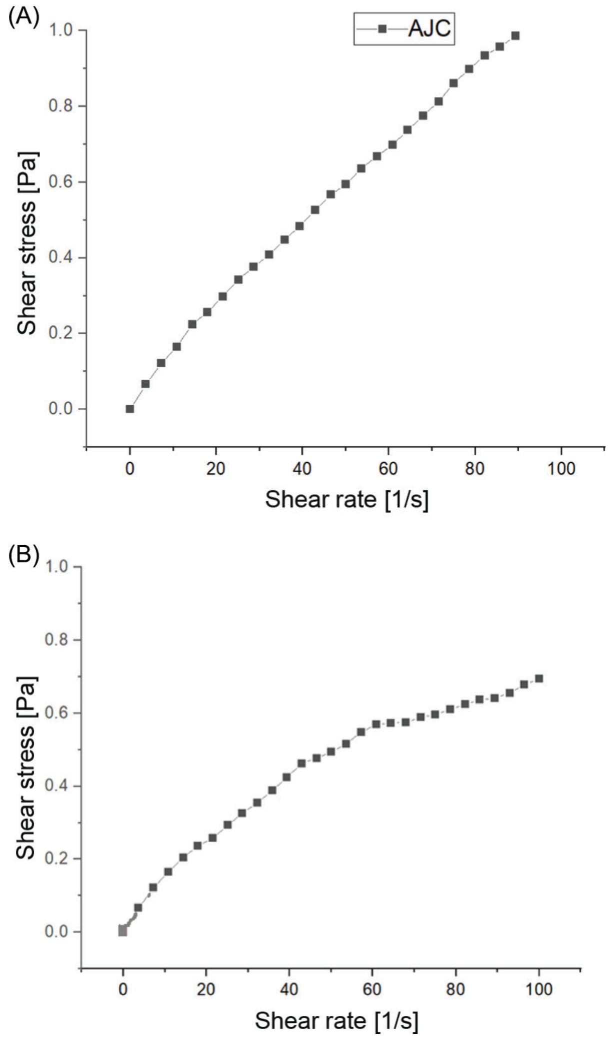

Figure 2. Rheogram depicting flow behaviour of (A) AJC and (B) SDAP.

due to hydrogen bonds and stretching of O–H bond between carbohydrates, carboxylic acids and residual water. The frequency range between 2850 and 3000 cm^{-1} corresponds to the C–H stretching linkages of sugars. Golubtsova (2017) also reported absorption bands at frequency of 2930 cm^{-1} for carbohydrate developed due to the valence resonance of CH_2 groups. Bi-atomic linear chained molecules held in single plane vibrate at higher energy level thus give absorption peaks at higher frequency ranges while tri-atomic molecules having double or triple bonds or even single bonds and held out of the plane oscillate at lower energy level thus give absorption bands at lower frequency range (Khan *et al*., 2018). Peaks between frequency ranges of 1600–1695 cm^{-1} and 1500–1585 cm^{-1} correlate with C=O stretching of amides and α-CH_2 bending and C=O stretching of carboxylic acids and esters, respectively. Anthocyanins, major colouring pigment in AJC, give absorption bands between 1250 and 1380 cm^{-1}. Peak shown in the frequency range of 1060–1170 cm^{-1} is due to stretching fluctuations of C–O bonds of sugars and organic acids. Phenols, a group of bioactive compounds that define the therapeutic value of plants, are known to absorb frequencies in the range of 1270–1140 cm^{-1}. The frequency range of 1100–1000 cm^{-1} is mainly due to vibrations of C–O–H group present in primary and secondary alcohols. Alcohols, found in copious amount in fruits, are the main flavouring elements in AJC. Absorption peak obtained in the frequency range of 880–1010 cm^{-1} is the characteristic of =CH and =CH_2 bending while peak between 780 and 846 cm^{-1} is assigned to CH bending and ring puckering.

Figure 1B depicts the FTIR spectra of SDAP. As evident from graph, no new peak is detected in SDAP which embellish that wall material has efficiently encapsulated the juice particles and have not entered into any compound formation. However, the absorption peaks have varied intensity possibly due to dilution effect of carrier material and concentration effect on constituents due to drying (Muzaffar *et al*., 2016). The peak at frequency range of 3243cm^{-1} is ascribed to stretching vibrations of hydroxyl group of water molecules (Muzaffar *et al*., 2016). However, the intensity of peak is considerably low as compared to peak obtained in AJC (Figure 1A). Low moisture content of SDAP (Table 1) and consequently low hydroxyl groups due to removal of moisture

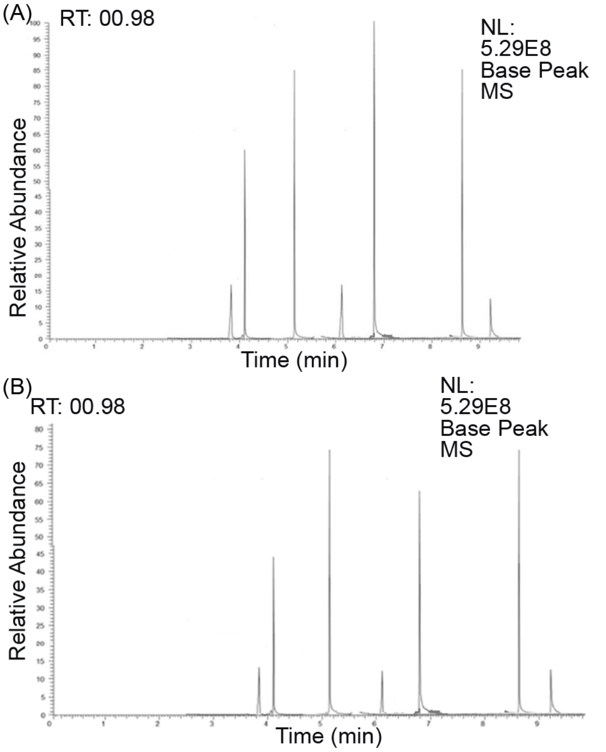

Figure 3. GC chromatogram revealing major flavouring compounds in (A) AJC and (B) SDAP.

during spray drying results in its reduced peak intensity (Muzaffar *et al.*, 2016). Most of the peaks are depicted in absorption band range of 700-1700cm^{-1} which is characteristic of sugars and Vitamin C present in SDAP (Suhag *et al.*, 2021). A pronounced peak around 2991cm^{-1} is obtained in SDAP due to N–H stretching of proteins from gum arabic as gum arabic contains 2% proteins in the form of glycoproteins (Mouecoucou *et al.*, 2003). Suhag *et al.* (2021) also ascribed peak between 2938 cm^{-1} and 2919 cm^{-1} to N–H stretching of proteins from gum arabic in spray dried honey powder.

Rheological studies

Flow behaviour

Rheological studies are important in understanding the changes that occur in molecular structure of fruit product during processing and thus are necessary for process optimisation of fruit product and equipment development (Makroo *et al.*, 2019). A rheogram presented in Figure 2A was used to study the flow behaviour of AJC by developing a relation between shear stress and rate. The resultant rheogram showed Newtonian flow of apple concentrate, that is, change in viscosity is independent of applied stress. Depectinized fruit juices are known to show Newtonian behaviour (Manjunatha *et al.*, 2012). Since AJC was devoid of any fibrous material (Table 1), thus, it recorded Newtonian behaviour. In depectinized juices, soluble solids (acids and sugars) content plays a vital role in the magnitude of viscosity as they are arranged in ordered crystalline form (Manjunatha *et al.*, 2012). Continuous application of stress at increasing rates does not impact the viscosity of AJC possibly due to presence of sugars and acids in ordered form which are capable to withstand applied stress. Hence, a change in shear stress does not lead to significant change in viscosity of concentrate. Flow curve validated the need for dilution of AJC before subjecting it to spray drying.

A graph between shear stress and shear rate (Figure 2B) for SDAP juice (reconstituted at 25% in 100 mL of water) depicted its non-Newtonian shear thinning nature (pseudoplastic behaviour). Muzaffar *et al.* (2016) also reported non-Newtonian pseudo plastic behaviour for spray dried tamarind powder reconstituted at different concentrations. High molecular weight substances are known to depict shear thinning behaviour (Keshani *et al.*, 2012). Incorporation of gum arabic during spray drying of AJC results in formation of network due to its highly ramified structure (Lee *et al.*, 2018). At low shear rates, the mess structure of SDAP juice absorbs the applied stress and will experience the build-up of shear stress. However, with the increase in shear rate, a distortion of the organised structure of SDAP juice occurs and re-ordering of molecules and bonds take place in the fluid. This induction of higher degree of order in the fluid reduces shear stress and leads to non-proportionality between shear rate and shear stress consequently resulting in shear thinning pseudoplastic behaviour.

Flavour determination

Chromatogram of apple concentrate is shown in Figure 3A. The different aroma compounds responsible for flavour of apple are represented in chromatogram. The peak at 3.8 min was identified as ethyl propionate while peak at 4.3 min was ascribed to methyl butanoate. The fruity attribute of concentrate could be explained by volatile esters that have fruity and apple-like odours. The peaks corresponding to residence time of 5.3 and 6.1 min were associated with ethyl-2-methyl butanoate and butyl acetate respectively. These methyl butyrate esters are important for the characteristic fruity and sweet aroma of apple (Plotto *et al.*, 1999). Alcohols are the second most prominent group of organic compounds that contribute flavour to apple. They are enzymatically produced from esters present in the cortex of fruit. Short-chained alcohols give off sweet aroma and thus their presence is important for development of characteristic apple flavour (Mehinagic *et al.*, 2006). Peak obtained at 6.9 min was identified as 2-methyl butyl acetate while Butyl-2-methyl butanoate was ascribed to peak obtained at residence time of 8.7

min. The characteristic flavour of apples is mainly due to presence of numerous volatile organic small molecules in their peel and flesh. Ethyl propionate, butyl acetate and 2-methylbutyl acetate have been identified as primarily flavour compounds by Sharma *et al*. (2014) responsible for aroma in several apple cultivars.

Flavour constituents of SDAP are depicts as gas chromatogram in Figure 3B. All of the major flavour compounds have been well encapsulated in SDAP due to synergistic effect of wall material (maltodextrin and gum arabic). However, a slight decrease in concentration of volatile constituents is recorded due to their low boiling point. Furthermore, 2-methyl butyl acetate, which is a major flavouring compound in AJC (Figure 3A) recorded predominant decrease in its concentration as compared to other volatile flouring compounds probably due to its small molecular weight as low molecular weight compounds have very low vapour pressure and thus, gets vaporises easily in the beginning of the drying process (Reineccius, 2004). Volatile loss takes place at three stages during spray drying viz at atomization step, at constant rate period when temperature of water is below boiling point and finally at falling rate period when temperature of water has received its boiling point. When droplet comes in contact with hot air, instantly surface moisture evaporates forming hard crust around juice droplets and thus protecting volatiles against thermal degradation. However, small molecular weight volatiles exhibit low vapour pressure and thus get lost even before complete atomization of juice particles take place. This loss is inevitable. Nevertheless, incorporation of gum arabic along with maltodextrin as wall material have improved the recovery of major flavouring compounds in SDAP due to high emulsion stability of gum arabic as a result of its lipophilic character (Reineccius, 2004).

Conclusions

The experiment conducted to study the detailed chemical composition and physical behaviour of apple concentrate revealed that the concentrate was low in moisture, protein and water activity and high in sugars, TSS, acidity and pH. The study also concluded that apple concentrate contained flavonoids, phenolics, ascorbic acid and anthocyanins in relatively lower amount which leads to a low antioxidant activity of concentrate. IR spectra confirmed the presence of major chemical constituents of apple such as phenols, primary and secondary alcohols and aromatic acids. Flavour analysis carried through GC-MS also indicated the presence of few major flavouring compounds in apple concentrate. Rheological study showed that apple concentrate behaved as Newtonian and concluded that highly viscous solution results in reduced drying rates. Thermophysical investigation inferred that the concentrate could be used for drying operation with minimal use of energy. Thus, the study revealed that the locally available apple concentrate had acceptable nutritional composition, superior rheological as well as thermophysical behaviour and hence can be put to development of various marketably acceptable high-quality fruit juice powder both at national and international market. Based on the physico-chemical, thermal and rheological properties, it was concluded that AJC can serve as an excellent base material for development of spray dried powder. However, diluting the concentrate is recommended to facilitate easily pumping of feed across spray dryer and increased flavour retention.

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