Journal of Food Engineering 149 (2015) 171–180

Contents lists available at ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

Spray-Freeze-Drying approach for soluble coffee processing and its effect on quality characteristics

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ARTICLE INFO

Article history:
Received 12 July 2014
Received in revised form 2 October 2014
Accepted 4 October 2014
Available online 18 October 2014

Keywords:
Soluble coffee
Spray-Freeze-Drying
Volatile retention
Morphology
Bulk density
Tap density

ABSTRACT

The suitability of Spray-Freeze-Drying (SFD) technique for soluble coffee processing was evaluated. The resultant product characteristics were compared against its spray-dried (SD) and freeze-dried (FD) counterparts. SFD and FD coffee powders exhibited a comparable aroma profile as indicated by the electronic nose analysis. SFD resulted in higher volatile retention (93%) than FD (77%) and SD (57%), as inferred from GC–MS analysis. SFD coffee showed instantaneous solubility due to its highly porous nature as observed in morphology studies. SFD coffee depicted monomodal particle size distribution with mean diameter (91.1 μm) ranging between SD (50.41 μm) and FD (636.8 μm) particles. SFD resulted in higher free (ρ_B, 0.612 g/mL) and tapped (ρ_T, 0.679 g/mL) bulk densities of the product against SD (ρ_B: 0.328 g/mL; ρ_T: 0.388 g/mL) and FD (ρ_B: 0.345 g/mL; ρ_T: 0.361 g/mL). SFD coffee exhibited free flowing characteristics as indicated by its Hausner ratio (1.11) and Carr index (10%).

1. Introduction

Coffee is the world's most widely traded tropical agricultural commodity with its process innovations evolving and emerging across years. Soluble coffee refers to the coffee powder obtained from freshly roasted and ground pure coffee beans (FSSAI, 2011). Spray drying (SD) and freeze drying (FD) are the conventional soluble coffee manufacturing techniques. While SD is established as the most economical process for commercial production, FD provides product with superior aroma quality.

Nevertheless, SD and FD are associated with some limitations. Spray drying exhibits possibilities of losing certain characteristic low-boiling aromatic compounds in coffee, due to its high-temperature operation (Taylor, 1983). Freeze drying process is energy intensive and expensive owing to the low-temperature (−40 °C and −50 °C) and low pressure (30–40 Pa) operation (Suwelack and Kunke, 2002) employed to avoid loss of flavours and the development of off-flavours in the dried product. Consequently, freeze drying of aqueous coffee extract requires longer drying time in the order of 8–16 h. Longer drying time results from the lower heat transfer rate to the core of frozen extract in order to counteract the increase in temperature of the dried coffee during sublimation and maintain it below the aforementioned low temperature to avoid flavour loss (Hair and Strang, 1969).

Spray-Freeze-Drying (SFD) technique possesses the main merits of SD and FD and can overcome the challenges aforementioned. SFD involves atomization, freezing and drying of the feed solution. The minute droplets resultant from atomization offer more homogeneous temperature field for heat transfer during the spray freezing step, which leads to uniform nucleation and formation of fine ice crystals (MacLeod et al., 2006). In the freeze drying step, an enhanced sublimation rate is facilitated by reduced product dimension (Pham, 1986). The consequent increase in surface mass transfer coefficient leads to a reduction in total drying time and eventually results in fine and free flowing powder. Meryman (1959) proposed the application of spray freezing in foods, owing to its advantages over conventional freeze drying in terms of water removal at increased mass transfer rates.

Studies on SFD of food products are quite limited when compared to its pharmaceutical applications. In addition, a study that establishes the potential of SFD as a method to prepare soluble coffee powder is not available hitherto. Thus, the aim of this research work is to evaluate the suitability of SFD as a soluble coffee processing technique on parity with SD and FD. The present work comprises of a detailed comparative study between the SFD, FD and SD coffee samples for their aroma profile, volatile retention, solubility, morphology, particle size analysis, flow characteristics and colour.

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http://dx.doi.org/10.1016/j.jfoodeng.2014.10.011
0260-8774/

2. Materials and methods

2.1. Feed solution preparation

Coffee solution was prepared by dissolving commercial instant pure coffee powder (procured from local market) in distilled water at a mass fraction of 40% w/w of the solution.

2.2. Spray Freeze Drying

SFD process was accomplished in a spray freezing rig, designed and fabricated locally. The rig is an arrangement comprising of a twin fluid nozzle, peristaltic pump and polystyrene container (32×10^{-2} m $\times$ 16×10^{-2} m) connected to a liquid nitrogen dewar. A disk blade impeller was used for mixing the contents. The distance between nozzle and liquid nitrogen was optimized and maintained at 10 cm. A twin fluid nozzle atomizer was used with compressed air pressure of 588.39 kPa. Feed flow rate was set at 6 mL/min. The resultant frozen particles were transferred to stainless steel trays and loaded to the freeze dryer where the frozen particles were subjected to primary and secondary freeze drying. Temperature range of primary drying stage was −25 °C to −10 °C under vacuum of 107 Pa and secondary drying was carried out at 10 °C under a vacuum of 40 Pa. On completion of drying, product was collected from the trays, packed in polythene bags, sealed, and wrapped in aluminium foil and stored in a desiccator at the ambient temperature.

2.3. Spray drying

SD was carried out in a single stage, short type, pilot scale dryer (Bowen Engineering Inc., Somerville, NJ, USA) of co-current drying configuration. Peristaltic pump was used to deliver the feed liquid to the atomizer. A twin fluid nozzle atomizer was used at a compressed air pressure of 392.27 kPa. Ambient air was directly heated in a burner using LPG gas, allowing control of the inlet air temperature at 150° ± 2 °C. While, the outlet air temperature was maintained at 100° ± 2 °C by the adjusted feed flow rate. Product was collected from the outlet chamber, packed in polythene bags, sealed, and wrapped in aluminium foil and stored in a desiccator at the ambient temperature.

2.4. Freeze drying

The coffee solution was freeze-dried using the pilot scale freeze dryer (Model: Lyodryer-LT-5S; Lyophilization systems Inc., USA) at a shelf temperature starting from −40 °C, progressing towards 10 °C. The freeze dried coffee powder was collected from the trays, packed in polythene bags, sealed, and wrapped in aluminium foil and stored in a desiccator at the ambient temperature.

2.5. Moisture content determination

The moisture content (% wet basis) was analyzed based on the gravimetric determination of the mass loss on drying (IS 2791, 1992). 1 g of the coffee sample was placed on an aluminium dish (flat bottomed; 90 × 12 mm) and heated at 95° ± 2 °C for 2 h in the hot air oven. The analysis was performed in duplicates, and the mean and standard deviation were calculated.

2.6. Headspace analysis using electronic nose

A known amount of coffee sample (1 g) was weighed and taken in 15 mL screw-capped vial. The aroma analysis was conducted using an electronic nose (Alpha Fox 4000, Alpha MOS, Toulouse, France with 18 metal oxide semiconducting sensors) as a function of volatile molecules over time under standardized conditions. Data analysis was done based on the values of maximum change in resistance of the sensors by subjecting to principal component analysis (PCA) using the in-built software supplied by the manufacturer.

2.7. Volatiles identification by HS–SPME gas chromatography/mass spectrometry

Volatile analysis was done using headspace solid phase microextraction (HS–SPME) followed by gas chromatography – mass spectrometry (GC–MS). 1 g of sample was placed in 15 mL screw-capped vial with PTFE septa and equilibrated in a thermostatic oven at 70° ± 1 °C for 1 h. Volatile extraction was done with a 60 μm polydimethylsiloxane/divinylbenzene (PDMS/DVB) Stableflex™ fibre for 10 min by insertion into the vial headspace. The compounds were thermally desorbed in the GC–MS injector port at 250 °C for 3 min.

A Perkin Elmer Turbomass Gold GC–MS system was used to analyze the headspace volatiles. An Elite-Wax polyethylene glycol (PEG) polar capillary GC column (30 m length, 0.32 mm ID with 0.25 μm film thickness) was used. Helium was used as the carrier gas at a flow rate of 1 mL/min. The injection temperature was 250 °C and oven temperature was set at 40 °C for a period of 1 min before heating at a rate of 3 °C/min to 150 °C; then the temperature was raised from 150 °C to 230 °C at 5 °C/min (held for 5 min). The mass spectrometer was operated with a source temperature of 180 °C and electron energy of 70 eV. Mass spectrum was obtained in the mass range from 40 to 400, using a scan time of 0.2 s and an inter-scan time of 0.1 s. The analysis was performed in duplicates, and the average volatile retention was calculated on an area basis.

2.8. Solubility

2.5 g of coffee powder was weighed and taken in a 500 mL beaker. Then, 150 ml of freshly boiled water was poured into the beaker containing coffee powder and examined for solubility and presence of lumps on the surface of the solution if any (IS 2791, 1992). The time taken for complete dissolution without any lumps on the surface was recorded. The analysis was performed in duplicates, and the average time for solubility was calculated.

2.9. Morphology

Scanning electron microscope (Leo 435 VP, Leo Electronic Systems, Cambridge, UK) was used to study the morphology of the SFD, FD and SD coffee powder samples. The samples were mounted on the specimen holder and sputter-coated with gold (2 min, 2 mbar) and observed at 15 kV and vacuum of 9.75×10^{-5} Torr.

2.10. Particle size analysis

Particle size and shape of dried coffee powder samples (SFD, FD and SD) was measured using laser diffraction-based particle size and shape analyzer (Microtrac S3500, USA). Small amount of sample was suspended in absolute ethanol (EMSURE®, ACS, ISO Reagent, Merck, Germany), and the particle size distribution and shape parameters were recorded during each measurement. The analysis was performed in triplicates, and the mean values were calculated. The particle mean diameter was expressed as the volume mean diameter, and the uniformity of particle size distribution was determined by the relative span factor (RSF) from the equation below.