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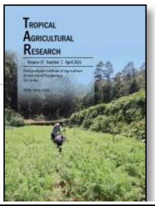

RESEARCH

Effects of Freeze Drying and Spray Drying Techniques on the Encapsulation of Black Tea Aroma Compounds

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ABSTRACT

In this study, freeze drying and spray drying were evaluated as techniques for encapsulating the volatile compounds in the steam distillate of black tea. A mixture of carrier materials was prepared by dissolving 0.1 g of gum arabic and 9.9 g of maltodextrin in warm water, then mixing it with 1 L of steam distillate of black tea. The prepared mixture was divided into two portions: one portion was freeze-dried, and the other was spray-dried to produce encapsulated black tea aroma powders. The sensory properties of the freeze-dried black tea aroma powder (FD-BTA) and spray-dried black tea aroma powder (SD-BTA) were assessed using a pairwise comparison test with thirty semi-trained panellists. Aroma profiles of FD-BTA and SD-BTA were analysed by solid-phase microextraction-gas chromatography-mass spectrometry and gas chromatography-flame ionization detector techniques. The results indicated that FD-BTA had superior sensory properties compared to SD-BTA and contained 17 key volatile compounds, whereas SD-BTA contained only 8 compounds. FD-BTA retained high concentrations of tea aroma available in steam distillate, including linalool (262.64±33.88 mg/L) and methyl salicylate (190.02±28.58 mg/L). The encapsulation efficiencies of key black tea aroma compounds were significantly higher with freeze drying (linalool- 61.89±5.64%, methyl salicylate- 23.59±3.02%, geraniol- 39.45±6.92%, and β-ionone- 29.86±4.50%) than with spray drying (linalool- 4.93±0.25%, methyl salicylate- 0.70±0.10%, geraniol- 13.78±2.41%, and β-ionone- 13.86±1.88%). Overall, freeze drying was found to be more effective than spray drying for encapsulating aroma compounds in black tea steam distillate.

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INTRODUCTION

Tea aroma is one of the key aspects of black tea quality. Instant black tea, as one of the black tea-related products, has a weak aroma profile due to intense heat treatments applied during the manufacturing process (Dalpathadu *et al.,* 2022; Kong *et al.,* 2022). In instant tea manufacture, tea aroma is generated as a co-product during the extraction of soluble solids from black tea using a spinning cone column extraction machine. Although it is possible to reintroduce tea aroma extract into the instant tea manufacturing process before spray drying, the use of high temperatures hinders the retention of tea volatiles (Dalpathadu *et al.,* 2022).

Tea aroma extract (or steam distillate) from black tea has a lower economic value due to the challenges associated with its highly diluted nature. The diluted tea aroma is highly perishable and requires careful handling, transportation, and storage. Therefore, encapsulation can be an efficient technique to preserve tea aroma compounds in steam distillate while enhancing its economic value. Generally, encapsulation is a process of entrapping active material(s) within a single carrier material or a mixture of carriers (Nedovic *et al.,* 2011; Yun *et al.,* 2021). Drying techniques, including spray drying and freeze drying, are commonly used in the food industry for the encapsulation of aroma compounds (Pellicer *et al.,* 2019; Kandasamy and Naveen, 2022; Yaman *et al.,* 2023).

Spray drying is a widely used technique for encapsulating flavours and aroma compounds because it enables greater retention of volatile compounds, is compatible with a variety of carrier materials, and is relatively cost-effective (Pellicer *et al.,* 2019). It involves four steps, including preparation of feed solution with carrier materials, homogenization, atomization, and dehydration (Koca *et al.,* 2015). In this technique, the hot air blown into the chamber removes water from atomized liquid particles, causing the loss of volatile compounds in the food matrices (Ishwarya *et al.,* 2015).

In freeze drying, the water in a food substance is frozen and removed through sublimation at low temperatures and pressure (Vardanega *et al.,* 2019). Freeze drying technique preserves the initial quality characteristics of the dried products, including flavours, nutrition, and texture, as there is no use of heat treatments (Yaman *et al.,* 2023). Preparation of the liquid medium, freezing, sublimation, and evaporation are the four steps included in the freeze drying process (Zheng *et al.,* 2006; Assegehegn *et al.,* 2019). In this study, the spray drying technique was selected due to its wide applicability in the food industry, whereas the freeze drying technique was chosen due to its operation at low temperatures, which can result in high retention of black tea volatiles at the end of the encapsulation process.

Carrier materials play an important role in entrapping and retaining aroma compounds in the encapsulation process. Cyclodextrin, gum arabic, maltodextrins, modified starches, and different proteins are used as common carrier materials in the food industry (Wani *et al.,* 2016; Azhar *et al.,* 2021). Carrier materials should be certified as ‘generally recognized as safe’ (GRAS) for use in food encapsulation. In this study, gum arabic and maltodextrin were used as the carrier materials. Both carrier materials are soluble in water and have a low viscosity and a neutral flavour profile (Xiao *et al.,* 2022; Zabot *et al.,* 2022).

To the best of our knowledge, there is a limited history of comparing freeze drying and spray drying techniques for encapsulating black tea volatiles. Therefore, the objective of this study was to investigate the effects of freeze drying and spray drying on the retention of black tea aroma compounds during each drying process and on the physicochemical characteristics of encapsulated black tea aroma powder.

METHODOLOGY

Sample collection

Tea aroma extract (steam distillate of black tea), generated from the spinning cone column (SCC) extraction machine during instant black tea manufacture, was collected from Martin Bauer Hayleys (Pvt) Ltd, Hatton, Sri Lanka.

Chemicals

Trans-2-hexenal (98%), linalool (97%), geraniol (97%), 2-phenyl ethanol (99%), methyl salicylate (99%), β-ionone (97%), and 2-ethyl butyric acid (99%) were purchased from Sigma Aldrich (USA). 2-ethyl butyric acid was the internal standard. Maltodextrin and gum arabic (Research-Lab Fine Chem Industries, Mumbai, India) were used as the carrier materials for the encapsulation of black tea aroma compounds.

Sample preparation

A mixture of carrier materials was prepared by dissolving 0.1 g of gum arabic and 9.9 g of maltodextrin in 20 mL of warm distilled water, then stored overnight at 4 °C to obtain complete hydration. The prepared mixture of carrier materials was then mixed with 1 L of tea aroma extract.

Freeze drying

Half of the mixture of carrier materials and tea aroma extract was frozen at -20 °C and freeze-dried using a Benchtop freeze dry system (Model-77500, Labcocnco corporation, Missouri, USA) at 0.133 mbar for approximately 48 – 72 h.

Spray drying

The remaining half of the mixture of carrier materials and tea aroma extract was spray-dried in a laboratory-scale spray dryer (SOLTEQ, Model: FD20) with a co-current drying configuration. The mixture was delivered to the atomizer using a peristaltic pump. The inlet temperature was maintained at 150±2 °C. The dried aroma powder was collected from the cyclone separator.

Packing the samples

Weights of encapsulated aroma powders prepared by freeze drying and spray drying techniques were recorded, and they were labelled as FD-BTA and SD-BTA, respectively. These samples were packed in triple-laminated polythene bags, sealed, and stored in a desiccator at room temperature for further analysis.

Determination of the yield

The yield of the FD-BTA and SD-BTA was calculated as a percentage ratio of the weight of the resultant product to the weight of the carrier materials used for drying (Kalkan *et al.,* 2017).

Sensory analysis

A pairwise comparison test (ISO 5495:2005) was performed using thirty semi-trained panellists to compare organoleptic properties (aroma, colour, and taste) of encapsulated aroma powder obtained from freeze drying (FD-BTA) and spray drying (SD-BTA) techniques. After explaining the study, consent was obtained from the panellists for their participation in the sensory evaluation.
Each 1 g of encapsulated aroma powder sample (FD-BTA and SD-BTA) was reconstituted separately in 100 mL of distilled water. The reconstituted samples were then served to each panellist separately in pairs, using white cups labelled with random three-digit codes. The panellists were instructed to indicate the sample with better aroma, colour, and taste characteristics in the provided scorecard. A bilateral table was used to evaluate the sensory data at a significance level of 0.05.

Aroma profile analysis

Aroma profiles of the tea aroma extract and the reconstituted FD-BTA and SD-BTA samples were identified using headspace solid-phase microextraction coupled with gas chromatography-mass spectrometry (HS-SPME-GC-MS) technique and quantified by headspace solid-phase microextraction coupled with gas chromatography-flame ionization detector (HS-SPME-GC-FID) technique as follows.

FD-BTA and SD-BTA samples were reconstituted by dissolving 1 g of each sample in 100 mL of distilled water. The internal standard stock solution was prepared by mixing 1 mL of 2-ethyl butyric acid (0.92 g/mL) in 9 mL of dichloromethane (Analytical reagent grade, Fishel Scientific UK, Loughborough).

HS-SPME procedure

One millilitre of the sample and 2 µL of the internal standard stock solution were transferred to a headspace vial (20 ml) and sealed with a crimp cap and PTFE septa. One-third of the vial's height was immersed in a water bath and equilibrated at 60 °C for 10 min. Volatiles from the headspace of the vial were extracted into an SPME fibre (80 µm divinyl benzene/ Carbon wide range/polydimethylsiloxane:DVB/CWR/PDMS) for 20 min at 60 °C using a manual device equipped with a holder (Agilent Technologies, Switzerland). The fibre was preconditioned by inserting it into the GC injector port at 220 °C for 3 min before each analysis.

GC-MS analysis

GC-MS analysis was carried out using a gas chromatograph system (Agilent, 7890B) fitted with a TR-WAX column (30 m in length, 0.25 mm inner diameter, 0.25 µm film thickness) and a mass spectrometer (Agilent, 5977A). The analyses were performed under the following conditions: desorption at 220 °C for 3 min, helium as the carrier gas with a constant flow rate of 1 mL/min, interface port temperature at 250 °C, ion source temperature at 230 °C, and quadrupole temperature at 150 °C. The oven temperature was programmed to stay at 60 °C for 3 min, then increase to 190 °C at a rate of 5 °C/ min and hold for 1 min. After that, it was increased to 240 °C at a rate of 20 °C/ min and held for 1 min. The mass spectrometer was operated in an electron-impact mode of 70 eV.

The GC-MS data were identified by comparing the MS spectra with the National Institute of Standards and Technology (NIST) library (8 L) database. The relative content of each volatile compound was determined by Equation 1.

GC-FID analysis

GC-FID analysis was carried out on a gas chromatograph system (Agilent, 7890A) equipped with a flame ionization detector (FID) (Agilent Technologies, CA, USA) and a DB-WAX column (30 m × 200 µm × 0.2 µm). Immediately after the extraction, the fibre was removed from the headspace of the vial and inserted into the injector port of the GC and desorbed for 3 min at 220 °C. The oven temperature was initially held at 60 °C for 2 min and then increased to 225 °C at a rate of 4 °C/ min, and held at 225 °C for 2 min. The injector and detector temperatures were maintained at 220 and 300 °C, respectively. Helium was used as a carrier gas (1.0 mL/min).

Quantification of key volatile compounds

Concentrations of the selected key aroma compounds (trans-2-hexenal, linalool, geraniol, 2-phenyl ethanol, methyl salicylate, and β-ionone) present in the steam distillate of black tea and reconstituted FD-BTA and SD-BTA samples were calculated using Equation 2 together with previously established relative response factors of individual volatile compounds (Hettiarachchi *et al.,* 2026).

Encapsulation efficiency (%)

Encapsulation efficiency was calculated using Equation 3, as the percentage ratio of the concentrations of the active materials in the encapsulated product to their concentrations in the initial sample (Piacentini, 2016).

$$Relative\ Content\ \% = \frac{Peak\ area\ of\ a\ single\ constituent}{Total\ peak\ area} \times 100$$ ………… Equation 1

$$Con(X = \frac{PA(X)}{RRF(X)} \times \frac{Con(IS)}{PA(IS)}$$ ……………………………………………… Equation 2

(Where PA(X) and PA(IS) are the peak areas of volatile compound "X" and the internal standard, respectively. Con(IS) is the concentration of the internal standard in mg/L, while RRF(X) is the relative response factor of volatile compound "X")

$$Encapsulation\ efficiency(\%) = \frac{CETAP}{CATA} \times 100$$ ………………………….. Equation 3

(Where CETAP is the concentration of aroma compounds in the reconstituted encapsulated tea aroma powder (in mg/L), and CATA is the concentration of aroma compounds in the tea aroma extract (in mg/L).

Scanning electron microscopy (SEM)

The morphological characteristics of FD-BTA and SD-BTA were evaluated with a scanning electron microscope (Carl Zeiss EVO-18), operated at 8 kV.

Statistical analysis

Data from the analysis of physicochemical properties were subjected to analysis of variance (ANOVA) and mean separation using Tukey's post-hoc test using Minitab (Version 17) software at a 0.05 level of significance. All the experiments were conducted in triplicate.

RESULTS AND DISCUSSION

Yield of encapsulated black tea aroma powder

Freeze drying showed a significantly higher yield (84.30±0.50%) compared to the spray drying technique (7.47±0.20%). Although both drying methods used identical feed compositions, the extremely low solid content (10 g of carrier materials in 1 L of feed solution) likely contributed to the poor recovery in spray drying. Feeds with low solid concentrations produce very fine particles, which are difficult to efficiently collect in cyclones of laboratory-scale spray dryers (Bhandari, 2008). Further, this may result in substantial powder loss through the exhaust air (Goula and Adamopoulos, 2004; Neves *et al.,* 2019; Filková and Mujumdar, 2020). In addition, the prolonged drying time (approximately 7 h) in spray drying may have increased wall deposition in the drying chamber, further reducing product recovery.

In contrast, freeze drying does not involve particle transport or cyclone separation. Since sublimation occurs within the same container in which the sample is frozen, most of the solid material remains in place after water removal (Roy and Gupta, 2004). This fundamental difference in recovery mechanism explains the large disparity in yield between freeze drying and spray drying.

Sensory properties of encapsulated black tea aroma powder

In this study, twenty-one out of the thirty panellists preferred the freeze-dried product (FD-BTA) for both aroma and taste characteristics compared to the spray-dried product ($P<0.05$). There were no significant differences in the preference for the colour between the reconstituted FD-BTA and SD-BTA samples at a 0.05 level of significance.

Further, 77% of the panellists preferred FD-BTA overall. Therefore, it is evident that FD-BTA is superior to SD-BTA in both aroma and taste.

Complete aroma profile

The relative contents (%) of volatile compounds in the steam distillate and the reconstituted FD-BTA and SD-BTA samples are summarized in Table 1, and the corresponding total ion chromatograms (TICs) of these samples are presented in Figure 1.
In tea aroma extract, alcohols were the most prominent aroma group with linalool (18.39±0.84%) and geraniol (7.09±0.63%) as the key components. However, the primary aroma compound found in it was an ester, methyl salicylate (19.54±1.33%), known for its sweet, spicy, and minty aroma (Wang *et al.,* 2008). Additionally, it contained benzeneacetaldehyde (5.57±0.49%), benzaldehyde (3.90±0.34%), β-ionone (4.07±1.04%), and β-damascenone (1.62±0.10%) in high relative contents.

FD-BTA showed 17 volatile compounds, including 6 aldehydes, 3 alkenes, 6 alcohols, an ester, and a ketone. Methyl salicylate (18.08±0.44%) was the main aroma compound in FD-BTA, followed by linalool (17.51±1.89%), β-ionone (10.56±0.23%), benzeneacetaldehyde (7.32±0.74%), and benzaldehyde (2.80±0.40%). Nevertheless, only eight aroma compounds were detected in SD-BTA, with benzeneacetaldehyde accounting for 12.22±0.99%.

Concentration of key black tea volatiles

Table 2 shows the concentrations of black tea aroma compounds in steam distillate and reconstituted FD-BTA and SD-BTA samples. Steam distillate of black tea with high concentrations of key black tea volatiles (trans-2-hexenal, linalool, geraniol, 2-phenyl ethanol, methyl salicylate, and β-ionone) served as the raw material for encapsulation by freeze drying and spray drying techniques. A significant reduction of these key volatiles was observed during both drying techniques. However, the decline rate was considerably higher in spray drying than in the freeze drying process. This significant difference can be explained by the various drying mechanisms and the physicochemical characteristics of individual aroma compounds.

Spray drying consists of intense heat and mass transfer under high temperature gradients and rapid moisture evaporation. The inlet temperature (150 °C) in the spray drier increases the vapor pressure of volatiles, enhancing their diffusion from the droplet surface into the surrounding drying air. Due to the rapid droplet shrinkage, the diffusion path length is reduced, releasing volatiles before stable encapsulation (Jauhari *et al.,* 2025). Furthermore, the high-water content of the feed (approximately 99%) likely intensified volatile losses during spray drying. In contrast, freeze drying removes water via sublimation under low temperature and reduced pressure conditions. This minimizes thermal degradation and limits volatility-driven losses. As a result, heat-sensitive and highly volatile compounds were better retained in FD-BTA.

The main aroma compound in the steam distillate was methyl salicylate (807.61±48.21 mg/L), followed by linalool (426.08±57.60 mg/L). However, it was the opposite for FD-BTA, which had higher concentrations of linalool (262.64±33.88 mg/L) compared to methyl salicylate (190.02±28.58 mg/L). Steam distillate of black tea was used as the core material for the preparation of encapsulated black tea aroma powders. The observed differences in the concentrations of volatile compounds between the steam distillate and the resulting encapsulated powders are primarily attributed to variations in the retention of these compounds during the encapsulation process. This retention is largely governed by the physicochemical properties of the individual volatiles, including their boiling point, volatility, and interactions with the wall materials.

Table 1: Complete aroma profiles of steam distillate, FD-BTA and SD-BTA

Functional group	Compound name	RT	Relative Content %			Odour description	References
			Steam distillate	FD-BTA	SD-BTA		
Aldehydes	(E)-2-Hexenal	7.3447	3.67±0.48[a]	0.78±0.06[b]	0.05±0.01[c]	Green	(Wang *et al.*, 2008)
	(E, E)-2,4-hexadienal	12.0973	0.39±0.01	n.d.	n.d.	citrus	(Alluhayb and Logue, 2017)
	(E)-2-Octenal	12.7527	1.47±0.40	n.d.	n.d.	Fatty, green	(Feng *et al.*, 2022)
	(E, E)-2,4-Heptadienal	14.3933	1.00±0.04[a]	0.35±0.05[b]	n.d.	Fatty	(Qin *et al.*, 2013)
	Benzaldehyde	15.0981	3.90±0.34[a]	2.80±0.40[b]	n.d.	Almond and sweet	(Wang et al., 2008)
	(E, E)-2,6-Nonadienal	16.6892	0.55±0.04	n.d.	n.d.	Green	(Luo *et al.*, 2022)
	Benzeneacetaldehyde	18.6141	5.57±0.49[b]	7.32±0.74[b]	12.22±0.99[a]	Floral and green	(Xu *et al.*, 2022)
	β-Citral	18.8862	0.33±0.01	n.d.	n.d.	Fruity	(Wang *et al.*, 2022)
	2,4-Decadienal	21.7757	0.57±0.03[a]	0.16±0.04[b]	n.d.	Fatty, fried	(Flaig *et al.*, 2020)
	5-Methyl-2-phenyl-2-hexenal	25.0655	n.d.	7.46±0.63	n.d.		
Alkenes	β-myrcene	6.1081	0.61±0.07	n.d.	n.d.		
	Limonene	6.8542	0.54±0.14	n.d.	n.d.	Citrus	(Qin *et al.*, 2013)
	β-cis-Ocimene	7.8146	0.37±0.09[a]	0.46±0.03[a]	n.d.	Fruity, floral	(Chen *et al.*, 2021)
	1S-alpha-Pinene	19.3644	3.23±0.49	n.d.	n.d.		
	(E, Z)-alpha-Farnesene	26.6772	n.d.	0.39±0.06	n. d.		

Ketone	2,2,6-trimethylcyclohexanone	9.6406	0.39±0.02	n.d.	n.d.		
	(E)-5-Octadien-2-one	16.3347	0.21±0.10	n.d.	n.d.		
	β- Damascenone	21.9571	1.62±0.10	n.d.	n.d.	Sweet Hay-like	(Zheng *et al.,* 2016)
	β-Ionone	22.5547	4.07±1.04[b]	10.56±0.23[a]	2.78±0.54[b]	Sweet, Hay-like	(Zheng *et al.,* 2016)
Alcohols	cis-3-Hexenol	12.4436	0.93±0.06[a]	0.58±0.05[b]	n.d.	Woody, violet	(Kinoshita *et al.,* 2010)
	Linalool	15.9926	18.39±0.84[a]	17.51±1.89[a]	0.48±0.09[b]	Floral	(Qin *et al.,* 2013)
	Nerol	22.4311	0.77±0.06[b]	n.d.	1.80±0.17[a]	Rosy and floral	(Lv *et al.,* 2012)
	Geraniol	23.4163	7.09±0.63[a]	0.97±0.06[b]	n.d.	Rosy and floral	(Zheng *et al.,* 2016)
	Benzyl Alcohol	23.8202	0.49±0.03	n.d.	n.d.	Burning taste	(Wang *et al.,* 2011)
	Phenylethyl Alcohol	24.7765	0.85±0.03[a]	1.05±0.04[b]	0.82±0.05[b]	Honey-like	(Wang *et al.,* 2011)
	Eugenol	29.0304	0.12±0.01[a]	0.09±0.00[b]	n.d.		
	cis-Linaloloxide	13.136	2.25±0.08	n.d.	n.d.		
	cis-Linalool oxide	13.911	6.96±1.01[a]	1.57±0.14[b]	n.d.	Honey	(Chen *et al.,* 2022)
	Linalool oxide (pyranoid)	21.3925	0.85±0.07	n.d.	n.d.	Honey	(Chen *et al.,* 2022)
Esters	Methyl salicylate	20.9554	19.54±1.33[a]	18.08±0.44[a]	0.55±0.02[b]	Mint	(Qin *et al.,* 2013)
Other	Pyridine, 3-ethyl-, 1-oxide	25.5268	0.53±0.01	n.d.	n.d.		

RT: retention time in minutes, FD-BTA- Encapsulated black tea aroma powder prepared by freeze drying, SD-BTA- Encapsulated black tea aroma powder prepared by spray drying, n.d-not detected. Different superscript letters in the same row indicate statistically significant differences ($p \leq 0.05$). Data are expressed as mean ± standard deviation (n=3).

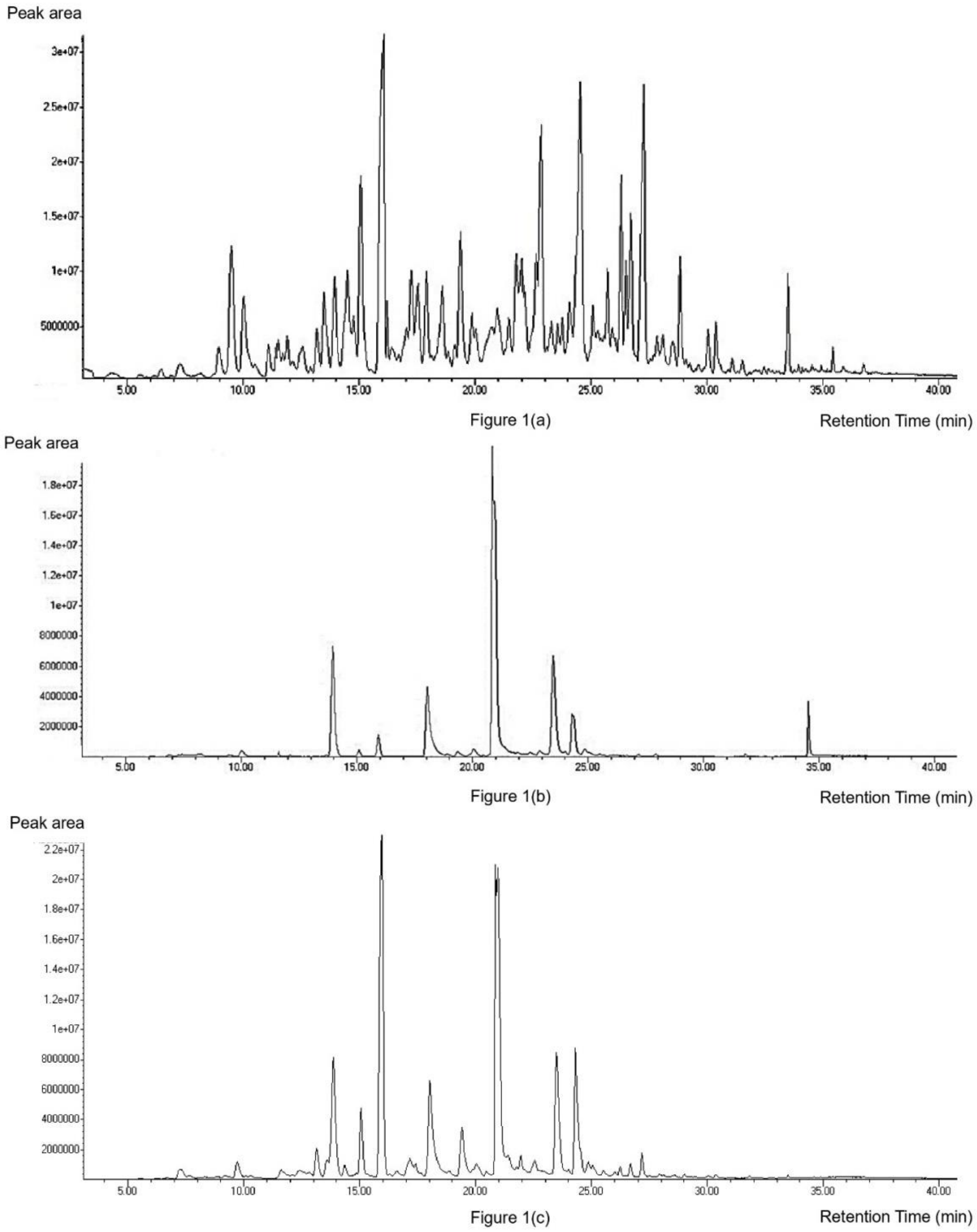

Figure 1: Total Ion Chromatograms of tea aroma extract (a), Total Ion Chromatograms of SD-BTA (b), and Total Ion Chromatograms of FD-BTA (c)

Table 2. Concentrations of black tea aroma compounds

Black tea aroma compounds	Retention time (min)	RRF	Concentration of aroma compounds (mg/L)		
			Steam distillate	FD-BTA	SD-BTA
Trans-2-hexenal	7.02	0.24±0.04	274.58±22.64[a]	55.42±7.98[b]	n.d.
Linalool	17.85	0.56±0.06	426.08±57.60[a]	262.64±33.88[b]	21.07±3.61[c]
Methyl salicylate	24.22	0.22±0.04	807.61±48.21[a]	190.02±28.58[b]	5.75±1.41[c]
Geraniol	26.30	0.72±0.11	232.47±28.85[a]	90.29±11.48[b]	31.46±3.41[c]
2-Phenyl ethanol	28.05	0.27±0.03	6.81±0.81[a]	1.11±0.08[c]	3.69±0.24[b]
β-ionone	28.52	0.10±0.02	191.70±26.79[a]	56.38±5.53[b]	26.51±5.06[c]

RRF- relative response factor, FD-BTA- Encapsulated black tea aroma powder prepared by freeze drying, SD-BTA- Encapsulated black tea aroma powder prepared by spray drying, n.d-not detected. Different superscripts within the same row indicate statistically significant differences ($P \leq 0.05$, n=3).

Further, spray-dried powder did not contain trans-2-hexenal, which is considered an off-flavour when present in excessive amounts in black tea (Wang *et al.,* 2008; Chen *et al.,* 2022). These concentration variances in SD-BTA may be attributed to the co-evaporation behaviour of volatiles with water vapour under an intense moisture removal mechanism. This can be further supported by the differences in matrix interaction and diffusion behaviour within the drying droplets. Although volatile compounds are immobilized within the frozen matrix in freeze drying, weakly bound aroma compounds may gradually diffuse through the matrix as sublimation proceeds (Niranjan *et al.,* 2002). Some moderately volatile compounds, such as 2-phenyl ethanol, exhibited comparatively higher retention in SD-BTA, possibly due to rapid crust formation during spray drying that enhanced matrix entrapment.

Encapsulation efficiency (%)

Table 3 presents the encapsulation efficiency of key black tea volatile compounds by freeze drying and spray drying techniques. The results indicate that the encapsulation efficiencies of the key black tea volatiles differ significantly between the freeze drying and spray drying techniques at the 0.05 significance level.

The freeze drying technique consistently outperformed spray drying in terms of encapsulation efficiency for all the black tea aroma compounds, similar to the findings of Kraujalytė *et al.* (2016). Freeze drying was effective in encapsulating linalool (61.89±5.64%), followed by geraniol (39.45±6.92%) and β-ionone (29.86±4.50%). Linalool is a volatile monoterpene alcohol with a relatively low boiling point, making it highly susceptible to evaporation when exposed to elevated temperatures experienced in spray drying. Therefore, freeze drying is more effective in retention of low-boiling volatiles such as linalool during encapsulation. However, spray drying retained 54.51±4.18% of 2-phenyl ethanol from tea aroma extract. 2-phenyl ethanol has a considerably higher boiling point (~219 °C) compared to many monoterpenes and other aroma compounds (Guckenbiehl *et al.,* 2024). This high boiling point reflects lower vapor pressure and reduced volatility at the temperatures typically experienced during spray drying. In addition, 2-phenyl ethanol contains a hydroxyl (–OH) functional group that increases its polarity and enables hydrogen bonding interactions with hydrophilic wall materials. These intermolecular interactions enhance its affinity for the encapsulating matrix, thereby improving its retention during spray drying (Mamusa *et al.,* 2020).

Table 3. Encapsulation efficiencies of black tea aroma compounds by freeze drying and spray drying techniques

Black tea aroma compound	Encapsulation efficiency (%)	
	Freeze drying	Spray drying
Trans-2-hexenal	20.42±3.76	n.d.
Linalool	61.89±5.64[a]	4.93±0.25[b]
Methyl salicylate	23.59±3.02[a]	0.70±0.10[b]
Geraniol	39.45±6.92[a]	13.78±2.41[b]
2-Phenyl ethanol	16.56±2.68[b]	54.51±4.18[a]
β-ionone	29.86±4.50[a]	13.86±1.88[b]

Different superscripts within the same row indicate statistically significant differences (P ≤ 0.05, n=3, n.d-not detected).

Morphology of encapsulated aroma powders

The external structures of FD-BTA and SD-BTA as observed by SEM are shown in Figures 2(a) and (b). FD-BTA appeared as voluminous flakes with irregular shapes (Jovanović *et al.,* 2021). In contrast, SD-BTA formed spherical particles with relatively smooth but uneven surfaces. The spherical morphology observed in spray-dried particles is typical of atomized droplets undergoing rapid surface crust formation (Zamani and Morris, 2022; Zhang *et al.,* 2024). As moisture evaporates from the surface, internal vapor pressure may cause structural collapse, producing wrinkles and concavities (Jovanović *et al.,* 2021). In contrast, freeze drying generates a highly porous, flake-like structure due to ice crystal sublimation, which preserves macrostructure but increases porosity and surface area.

(a) (b)

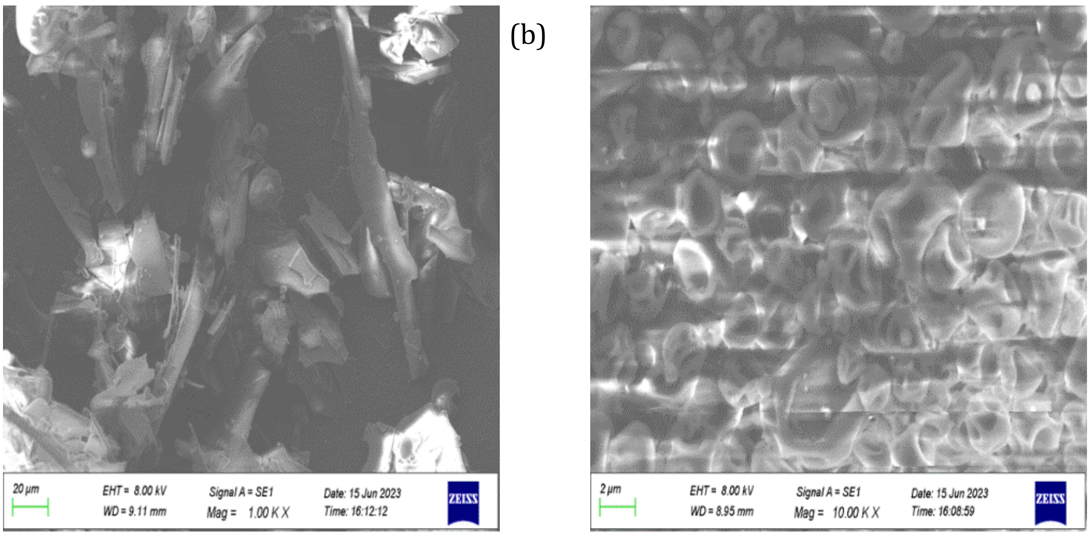

Figure 2: SEM micrographs of encapsulated tea aroma powder produced by (a) freeze drying and (b) spray drying

CONCLUSIONS

Based on the findings of this study, it can be concluded that freeze drying is more effective than spray drying for encapsulating black tea aroma compounds, largely due to its low-temperature that minimizes thermal degradation and volatile loss. In contrast, spray drying, particularly with low-solids and high-moisture feeds, results in significant loss of aroma compounds due to rapid moisture evaporation and high temperature gradients. These findings highlight the potential of freeze-dried tea aroma powders for use in instant tea products and other flavour formulations where retention of delicate aroma is crucial. Future work could explore alternative carrier materials to encapsulate a wide range of black tea aroma compounds using optimised drying parameters.

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COMPETING INTERESTS

The authors declare that there are no competing interests.

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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