integrity Research Journals

Research Journal of Food Science and Nutrition
Volume 10(2), pages 31-48, April 2025
Article Number: FF46413D1
ISSN: 2536-7080
https://doi.org/10.31248/RJFSN2025.196
https://integrityresjournals.org/journal/RJFSN

Full Length Research

Influence of drying methods on the nutritional composition and functional properties of puree blends produced from watermelon, mango, and orange varieties

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Received 1st February 2025; Accepted 26th February 2025

ABSTRACT: This study aimed to evaluate the effects of spray-drying and freeze-drying on the nutritional composition and functional properties of fruit purees composed of 50% watermelon, 30% orange, and 20% mango. The results demonstrated significant differences in nutrient retention and functionality; freeze-dried fruit powders (FDFP) retained higher levels of key nutrients compared to spray-dried powders (SDFP). Vitamin C content in FDFP was 5.60 mg/100 g, compared to 5.40 mg/100 g in SDFP, while carotenoids reached 16.29 mg/100 g in FDFP against 13.68 mg/100 g in SDFP. Proximate analysis revealed higher protein (0.78%) and fat (2.76%) content in FDFP versus SDFP (protein: 0.31%, fat: 1.11%). Phytochemical analysis indicated elevated total phenols (261.76 mg/100 g) and flavonoids (36.07 mg/100 g) in FDFP. Sensory evaluation ranked FDFP higher in appearance, aroma, taste, and overall acceptability, with a hedonic score of 7.72 on a 9-point scale. Functional properties, including water absorption capacity and bulk density, favored FDFP. These findings endorse freeze-drying as a superior method for producing high-quality, shelf-stable fruit powders, essential for mitigating postharvest losses while providing nutritious options for functional food applications.

Keywords: Freeze-drying, spray-drying, fruit powders, watermelon, orange, mango, nutritional quality, phytochemicals.

INTRODUCTION

Fruits are vital components of human diets, offering a rich source of vitamins, minerals, and bioactive compounds essential for health and well-being. However, significant postharvest losses occur, particularly in tropical and subtropical regions, due to inadequate preservation methods (FAO, 2017). Watermelon (*Citrullus lanatus)*, orange (*Citrus sinensis*), and mango (*Mangifera indica L.*) are among the most consumed fruits globally, valued for their high concentrations of lycopene, vitamin C, and carotenoids, respectively (Meléndez-Martínez *et al*., 2008). Despite their nutritional richness, these fruits are highly perishable, necessitating innovative strategies for preservation and value addition.

The transformation of these fruits into composite powders offers a sustainable approach to address postharvest losses. Such powders provide shelf-stable, nutritionally dense products suitable for diverse food applications. Drying methods like spray-drying and freeze-drying are commonly employed, yet their impacts on the retention of nutrients and functional properties vary significantly. Freeze-drying is known for preserving heat-sensitive compounds like vitamin C and carotenoids, while spray-drying offers a cost-effective alternative, albeit with reduced nutrient retention (Silva-Espinoza *et al*., 2019).

Statement of the problem

Despite the growing interest in mixed fruit powders, there is a dearth of information on the influence of drying

methods on the nutritional, functional, and sensory qualities of composite powders derived from watermelon, orange, and mango. This knowledge gap limits the optimization of drying techniques to achieve high-quality products in terms of shelf life, nutrient retention, and consumer acceptability.

Objectives

The primary objectives of this study were:

1. To evaluate the effects of spray-drying and freeze-drying methods on the nutritional composition of watermelon-orange-mango puree blends.
2. To analyze the functional properties, including water and oil absorption capacities, bulk density, and reconstitution potential, of the resulting powders.
3. To assess the sensory attributes and overall acceptability of the powders.
4. To identify the drying method that provides optimal nutrient retention, functional properties, and consumer preference for mixed fruit powders.

These objectives align with the global food security agenda, emphasizing the development of nutritionally rich, shelf-stable food products by 2030 (UN SDGs).

Research questions

The research questions for this study are as follows:

1. Are spray-drying and freeze-drying methods appropriate for application in the production of fruit powders from a composite of watermelon, orange, and mango purees?
2. What proportional ratios of the fruit purees would provide the best quality combination for powder production?
3. Does spray drying and freeze drying of mixed watermelon, orange, and mango purees produce fruit powders with similar or different functional, chemical, physical, sensory, and moisture transfer properties?

These research questions highlight the knowledge gap that this present study seeks to fill and, hence, the justification for the research.

LITERATURE REVIEW

Watermelon

Watermelon (*Citrullus lanatus*) is a member of the Cucurbitaceae family. The fruit is popularly consumed, especially during hot weather and the summer season. The fruit contains high amounts of vitamins, minerals and phytochemicals, especially lycopene, which is a type of carotenoid that is responsible for the red flesh color (Liu *et al.*, 2020). In addition to lycopene, watermelon is the cucurbit crop that contains the highest concentration of L-citrulline (citrulline). Citrulline is a non-proteinaceous, non-essential, physiologically active amino acid with relevance in mammalian metabolism (Summar, 2024). It is an intermediate metabolite that has sparked much human health research in the past forty years. This spike in citrulline-human health interest, in concert with a consumer desire for functional foods, has pointed scientists to watermelons as a natural source of citrulline (Summar, 2024). According to Summar (2024), regular intake of citrulline for at least 7 days improved aerobic performance by increasing the body's production of nitric oxide, which helps expand blood vessels so that the heart doesn't need to work as hard to pump blood through the body. Both citrulline and arginine play important roles in the synthesis of nitric oxide, which helps lower blood pressure by dilating and relaxing your blood vessels. Nitric oxide is a gas molecule that causes the tiny muscles around blood vessels to relax and dilate, resulting in a reduction in blood pressure. Arginine is also important for many organs such as the lungs, kidneys, liver, and immune and reproductive systems and has been shown to facilitate wound healing.

Orange

Orange is a citrus fruit consumed in high quantities all over the world in natural and peeled forms and as a juice, puree, or other forms. Orange is a low-cost fruit that contains many nutrients, including vitamins A and B, minerals (calcium, phosphorus, potassium), dietary fiber, and many phytochemicals, including flavonoids, amino acids, triterpenes, phenolic acids, and carotenoids (Melèndez-Martínez *et al*., 2022; Roussos, 2011). The vitamin C content in oranges helps the body absorb iron. Iron enables the body to use oxygen better, and a lack of iron can cause fatigue. Getting enough iron is especially important for premenopausal people who lose iron through their period (Munro *et al*., 2023). The flavonoids in citrus help prevent cancer cells from growing and spreading. For example, flavonoids help regulate or program cell death (apoptosis). Apoptosis is a process the body uses to kill off abnormal cells before they multiply and grow out of control (Mustafa *et al*., 2024). As an excellent source of the antioxidant vitamin C, oranges may help combat the formation of free radicals that cause cancer (Huang *et al.*, 2020).

Maintaining a low sodium intake is essential to lowering blood pressure. However, increasing potassium intake may be just as important for reducing a person's risk of high blood pressure, as it can help support the relaxation and opening of blood vessels. According to the Office of

Dietary Supplements (ODS, 2 024), increasing potassium intake can reduce the risk Trusted Source of high blood pressure and stroke. Oranges are a good source of fiber and potassium, both of which can support heart health. A medium orange weighing 131 grams (g) contributes 3.14 g of fiber, which is nearly 10% of an adult's daily fiber requirement. Several studies have found that fiber can improve some factors that contribute to the development and progression. According to Sass (2024), weight control is also important for reducing the risk of diabetes, as obesity and being overweight can contribute to the development of type 2 diabetes. The body processes fiber more slowly than other nutrients, so it can help a person feel fuller for longer and reduce their urge to eat snacks throughout the day.

Oranges also contain choline and zeaxanthin (Sass *et al.*, 2024). As reported by this author, choline is an important nutrient in oranges that helps with sleep, muscle movement, learning, and memory. Choline also aids the transmission of nerve impulses, assists in the absorption of fat, and reduces chronic inflammation. Zeaxanthin is a type of carotenoid antioxidant that can reduce inflammation. According to Sass *et al.* (2024), it can positively benefit heart, liver, skin, and eye health.

Mangoes

Mango (*Mangifera indica L*.) is one of the choicest fruits in the world (Joshi *et al.*, 2013). social and economic impact are most relevant in developing and emerging countries, where mango is a high-valued component in diet, rich in vitamins and minerals (Rocha Ribeiro *et al.*, 2007).

Fresh mango contains vitamin C and folate in significant amounts of the Daily Value as 44% and 11%, respectively. Mango is an excellent source of bioactive compounds such as carotenoids (provitamin A), vitamin C, phenolics, and dietary fibre (Lemmens *et al*., 2013; Rincon and Kerr, 2010; Lebaka *et al*.,2021), essential to human nutrition and health. Moreover, mango is known to contain other vitamins, carbohydrates, and minerals such as calcium, iron, and potassium, and to be low in calories and fat.

Drying methods

A variety of drying techniques are available for industrial use. According to Chopda and Barrett (2001), the most successful methods for producing fruit juice powder are freeze drying, foam mat drying, and spray drying.

Spray drying

Spray drying is an excellent common method for preserving heat and oxygen-sensitive fruits (Shi *et al.*, 2018). The method entails atomizing the solution in hot air to produce a powder product in a short amount of time. It provides a large surface area in the form of fine liquid droplets through atomization in the drying chamber, which leads to the production of regularly and spherically shaped powder particles (Fazaeli *et al.*, 2012, Turchiuli *et al.*, 2011). It is the most economical technique for maintaining quality by rapid dehydration. Aside from preserving colour and aroma, dried powders offer significant cost savings over liquids, such as in lowering the weight, minimizing packaging, and ensuring longer shelf life (Tuyen *et al.,* 2010).

Freeze drying

Freeze drying is another approach well used in industry. It is about a dehydration process that removes water through ice sublimation from the frozen products. It is regarded as the most efficient in nutrient preservation in powdered products, but its industrial-scale application is hampered by the high expenditures of the instrumentation and high energy consumption, as well as by a low throughput (Hsu *et al.,* 2003, Ratti, 2001). According to Dincer (2000), the main advantages of freeze-drying over other traditional methods of food preservation (such as conventional drying and spray drying) are the preservation of morphological, biochemical, and immunological properties; high levels of viability and activity; lower temperatures and shear conditions than other drying methods; high recovery of volatiles; preservation of structure, surface area, and stoichiometric ratios; high yield; and long shelf life. Equipment for freeze-drying is very expensive and might not be economical for some goods. However, according to Jude *et al*. (2023), the equipment requires a lot of space and operates for a long period to freeze-dry foods with the attendant high energy costs. Again, not all foods can be freeze-dried, and some may require additives. The process preserves the natural fresh fruit colour, texture, flavor, nutritional content, taste, physical characteristics, chemical compound, and biological activity with just minor alteration (Nawirska *et al.,* 2009).

Drying aids

Different drying aids such as maltodextrins, gum Arabic, modified starches, and proteins are used in spray drying to minimize the stickiness problem (Caliskan and Drim, 2013; Şahin-Nadeem *et al*., 2013; Rascón *et al.,* 2011).

Maltodextrin

To minimize the stickiness problem during spray-drying, high molecular weight drying aids are added to the feed material before atomization so as to increase its glass transition temperature (Cabral *et al.,* 2004; Shrestha *et al.,* 2007; Santhalakshmy *et al.,* 2015). These drying aids not only overcome the stickiness problem and reduce powder hygrosopicity but also protect sensitive components of food material, including phenolics, vitamins, and caroteinoids (Ferrari *et al.,* 2012).

Justification of the study

Watermelon is a good source of antioxidants and nutrients. In addition to supporting hydration, it may also promote many aspects of health, including heart health, and prevent certain health conditions. On the other hand, oranges contain no sodium, which helps keep a person below the daily limit while boosting daily potassium intake by 14 times (NASEM, 2019). Maintaining a low sodium intake is essential to lowering blood pressure, while increasing potassium intake can reduce the risk of high blood pressure, as it can help support the relaxation and opening of blood vessels (Weaver, 2013). The major nutrients in oranges include vitamins A and B, minerals (calcium, phosphorus, potassium), dietary fiber, and many phytochemicals, including flavonoids, amino acids, triterpenes, phenolic acids, and carotenoids (Melèndez-Martínez *et al.,* 2008; Roussos, 2011). Mango contains lutein, zeaxanthin, and vitamin A, all of which support eye health and immunity. Lutein and zeaxanthin may protect the eyes from the sun, while a lack of vitamin A may cause vision problems. The transformation of watermelon, oranges, and mango fruits into mixed fruit powders while helping to reduce postharvest losses will add value to the fruits chain from cultivation through harvesting to processing and marketing to consumption. Along this chain, more jobs will be created, and more women who are the major and key players in the chain will be empowered.

Research gap

The World Health Organization (WHO) millennium goal of food security by the year 2030 emphasizes the formulation of high nutrient-dense, convenient, and shelf-stable food products. The production of watermelon, orange, and mango mixed fruit powder is in line with this goal. A multi-fruits powder comprising watermelon, orange, and mango juices may provide these nutritional benefits in a single pack. However, information on the nutritional, physico-chemical, and shelf-stability of the mixed fruits powders, especially as influenced by spray and freeze drying methods are very essential but lacking in literature. Currently, there is a growing trend toward the production of mixed fruit powders for the development of new sensations and functional properties. The development of watermelon-orange-mango mixed fruit powders will contribute to addressing these research gaps.

MATERIALS AND METHODS

Sources of raw materials and preliminary handling

The mango, orange varieties (20kg each) and ten (10) fruits each were procured from the Gboko local market in Gboko Benue State Nigeria, while five (5) fruits of the 'Sugar Baby' variety of watermelon were sourced from the Makurdi Railway market also in Benue State, Nigeria. All fruit varieties were transported in polyethylene bags to the Joseph Tarkaa Federal University of Agriculture, Makurdi, Nigeria for proper identification. They were then refrigerated in preparation for further processing and analysis.

Sample preparation

The fruits were washed and their average weights taken and recorded. They were peeled and the weights of the peels measured and also recorded. The remaining processes to produce the puree prior to drying were according to the following flow charts (Figures 1 to 3).

Each puree type, depending on its stickiness and viscosity were mixed with 15%, 20%, 25% and 30% (w/w) commercial maltodextrin for water melon, orange and mango puree (the ratio of puree solids to carrier being 1:1.38; 1:1.95; 1:2.60; 1:3.35) respectively with Dextrose Equivalent (DE) 20 – 30. The purees were formulated into smoothies. With selected addition of maltodextrin, the most acceptable smoothie was subjected to the spray and freeze drying techniques.

Fruit puree production process

Watermelon fruit puree production

The flow chart for the production of watermelon puree is shown in Figure 1 using the method described by Mamadou *et al.* (2018). After washing and sorting, the fruits were peeled manually using stainless steel knives followed by slicing, removal of the seeds followed by blending of pulps in a household electric blender (Kenwood Electricals, UK) at speed number 3 for 15 seconds into smooth pastes which were pasteurized at 70°C for 15 seconds in 250 ml glass beakers with aluminum foil coverings. After cooling, the watermelon purees were kept in a refrigerator prior to use for composite purees formulation.

Orange fruit puree production

Orange fruit puree was produced with slight modification as described for orange juice production by Obasi *et al*. (2017). Essentially, as shown in Figure 2, the fruits were sorted, washed, peeled, and sliced using stainless steel knives. After the removal of the seeds, the slices were blended into a smooth paste using the household electric blender. The orange puree was then pasteurized at 70°C for 15 seconds in 250 ml glass beakers with aluminum foil covers. The pasteurized orange puree was rapidly cooled in an ice bath and promptly stored in a refrigerator prior to use for mixed purees formulation.

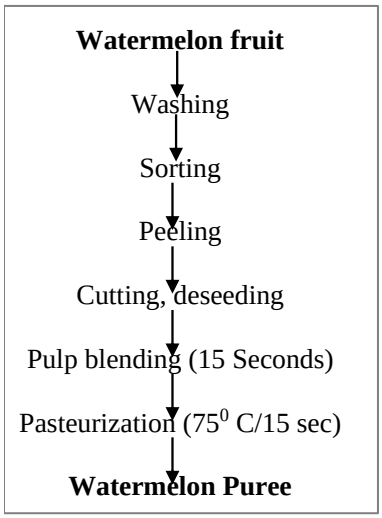

Figure 1. Flow chart for watermelon fruits puree production (Source: Mamadou *et al.*, 2018).

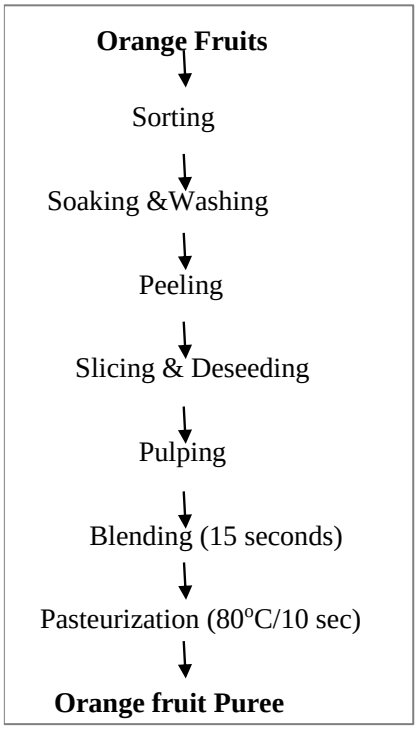

Figure 2. Flow chart for production of orange fruits puree (Source: Obasi *et al*., 2017).

Mango fruit puree production

The production of the mango fruit puree was by the method of Labaky *et al*. (2020), as provided in Figure 3. The mango fruits were sorted, washed, and blanched by immersion in a boiling hot water bath maintained at 98°C for 5 minutes.

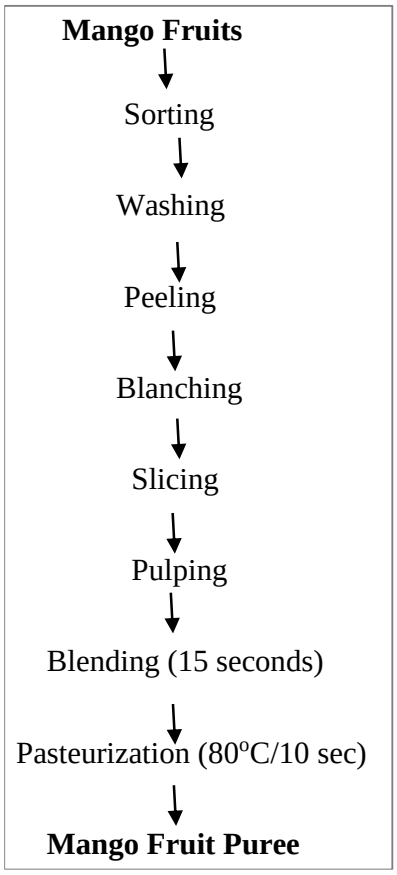

Figure 3. Flow chart for mango puree production (Source: Labaky *et al*., 2020).

The blanched mango fruits were then cooled in running tap water, peeled using stainless steel knives, and the fleshy mesocarp sliced to obtain pieces that were blended in the Kenwood mixer in the presence of 0.2 M citric acid buffer (pH 5.2) into a smooth slurry. The slurry was then stored in the freezer compartment of a household refrigerator prior to use for composite purees formulation.

Composite fruit purees formulation

The composite fruit purees compositions are shown in Table 1. In order to minimize bias, the formulations were each coded using 3-digit random numbers. Each puree type was treated with commercial maltodextrin as a carrier agent respectively to obtain a dextrose equivalent (DE) of 30 for each group. The composite purees together with the malodextrins were each blended into smoothies and subjected to preliminary sensory evaluation which indicated that the composite puree sample comprising 50% watermelon, 30% orange and 20% mango composite puree (code: 618) was the most acceptable smoothie and hence was used for the spray and freeze drying experiments respectively.

Table 1. Composite purees formulation.

Sample code	Puree composition (%)		
	Watermelon	Orange	Mango
573	30	50	20
618	50	30	20
335	20	50	30
804	50	20	30
732	20	30	50
408	30	20	50

Sensory evaluation

Sample labelling

The Sensory Attributes for the fruit purees are shown in Table 2. The samples were labelled with random 3-digit codes to ensure blind testing and avoid bias during the sensory evaluation process. The reconstituted fruit samples were served at room temperature (~20°C), as temperature fluctuations can influence the perception of taste and aroma (Lawless and Heymann, 2010). The samples were presented in 200ml disposable identical cups that did not influence taste perception (neutral color and odor-free). Consistent portions of 50 mL per sample were used (Lawless and Heymann, 2010). The sensory evaluation of the fresh composite purees was carried out using a trained sensory panel consisting of staff and students of the University of Mkar. The panel consisted of 50 members, including male and female members of the University of Mkar, Mkar. All evaluation sessions were held in the Food Chemistry Laboratory of the Food Science and Technology.

Evaluation

The sensory evaluation of the fresh samples was carried out four hours after formulation, while the sensory evaluation of the dried products was carried out after one week of production. The samples were stored at 5°C and taken out three hours before serving. Appearance, aroma, taste, texture, consistency and overall acceptability were evaluated following a nine-point hedonic scale (9 = like extremely, 8 = like very much, 7 = like moderately, 6 = like slightly, 5 = neither like nor dislike, 4 = dislike slightly, 3 = dislike moderately, 2 = dislike very much, 1 = dislike extremely). The panelists were thoroughly briefed on how to use the sensory evaluation forms and terminologies of sensory attributes. All samples were presented before the panelists at room temperature under normal lighting conditions in 50 ml cups coded with random, 3-digit numbers to ensure blind testing and avoid bias during the sensory evaluation process. Drinking water was provided for oral rinsing. The average values of the sensory scores (appearance, aroma, taste, texture, consistency, and overall acceptability) were used in the analysis as described by Ihekoronye and Ngoddy (1985).

Statistical (data) analysis

All the experiments were conducted in triplicate samples, and the data were the mean of the three replications. All data obtained were statistically analyzed using the Analysis of Variance (ANOVA) using SPSS Version 20 and the Duncan Multiple range test to separate means with significance level $p<0.05$ (Ihekoronye and Ngoddy)

Fruit powder production

The most organoleptically accepted composite fruit puree containing 50% watermelon, 30% orange, and 20% mango was subjected to freeze drying and spray drying respectively as follows:

Freeze drying operations

Freeze drying of the composite fruits puree was as described by Silva-Espinoza *et al.* (2021) using a pilot scale freeze dryer (model: LabconcoFreeZone Triad, Freeze Dry System, Freeze Dry Ltd., Warwickshire, UK). The freeze-drying machine (with a Capacity of 18 liters of ice condensing capacity) was preheated to 50°C with an initial pressure of 0.030 mbar according to the manufacturer's instructions. The mixed fruit puree was poured into 250 g capacity freezer bags, sealed, and placed on freezer trays prior to loading and in the freeze-drying chamber of the freeze dryer. The initial freezing was done to −45°C while during drying the temperature was increased up to 60°C. A vacuum of 100 mmHg was maintained during freeze drying. The process was regularly monitored to ensure proper drying. The freeze-dried composite fruit puree powder containing 2–3 % moisture was allowed to cool to room temperature. The freeze-dried puffy material was then blended in a household Kenwood dry mixer for 2 minutes, and the powder packaged in air-tight aluminum pouches, which were then stored on dry shelves in glass desiccators containing activated silica.

Table 2. Sensory attributes of the fresh mango-orange-water melon composite puree samples.

Sample codes	Appearance	Aroma	Taste	Texture	Consistency	Overall acceptability
573	7.000±1.080^{ab}	6.7200±1.243^{a}	6.2800±1.021^{c}	6.3200±1.435^{a}	6.5200±1.530^{b}	7.0800±1.115^{a}
618*	7.7200±1.060^{b}	7.2000±1.251^{ab}	7.2000±1.040^{ab}	7.1200±1.301^{a}	7.3200±1.069^{a}	7.7200±1.208^{a}
335	6.8400±0.943^{b}	7.0000±1.154^{ab}	7.0800±1.222^{ab}	6.4000±2.020^{a}	7.0800±1.382^{ab}	7.2000±1.208^{a}
804	6.8800±1.201^{b}	7.5200±0.770^{b}	7.4800±1.084^{a}	6.7600±1.984^{a}	6.8800±1.268^{ab}	7.5200±1.357^{a}
732	7.2400±1.640^{ab}	6.9600±1.206^{ab}	6.9200±1.288^{abc}	7.0400±1.428^{a}	7.1200±0.781^{ab}	7.6400±1.036^{a}
408	7.4400±1.193^{ab}	6.8400±1.374^{ab}	6.6400±1.350^{ab}	7.1200±0.971^{a}	6.9200±1.037^{ab}	7.1200±1.266^{a}

Values are mean ± standard deviation (SD) of triplicate determinations. Samples with different superscripts within the same column were significantly ($p<0.05$) different. **Key:** 573 = 20% mango, 50% orange, 30% watermelon; *618 = 20% mango, 30% orange, 50% watermelon; 335 = 30% mango, 50% orange, 20% watermelon; 804 = 30% mango, 20% orange, 50% watermelon; 732 = 50% mango, 30% orange, 20% watermelon; 408 = 50% mango, 20% orange, 30% watermelon; ***Most Acceptable (Overall Acceptability).**

Table 3. Proximate composition of fresh, freeze-dried & spray-dried puree mixture (composite) (%).

Product	Moisture	Ash	Protein	Crude fibre	Fat	Carbohydrate	Energy (Kcal)
Fresh sample	83.45±1.243^{a}	0.737±1.213^{a}	0.960 ±1.428^{a}	0.743±1.428^{a}	0.503±1.530^{c}	14.00±1.060^{b}	64.37±1.357^{a}
SDFP	2.83±1.060^{b}	3.49±1.053^{b}	0.31±1.206^{b}	1.21±1.021^{c}	1.11± 0.971^{a}	6.15±1.115^{a}	41.35±1.021^{c}
FDFP	3.55±1.021^{c}	4.39±1.063^{b}	0.78 ±1.530^{c}	2.89±1.288^{bc}	2.76±1.037^{ab}	5.51±1.208^{a}	46.62±1.201^{b}

FDFP = freeze-dried fruit powder; SDFP = spray-dried powdered powder.

Spray drying operations

Spray drying of the composite fruits puree containing 50% watermelon, 30% orange, and 20% mango was as described by Jeyanth *et al.* (2020) using a pilot plant spray-dryer (Simon Dryers Ltd, Cheshire, England) with a co-current air flow. The speed of the blower was set at 2400 rpm for all the drying. Distilled water was pumped into the dryer at a set flow rate at 10 rpm (10 rpm ~ 30 ml/min) to achieve inlet and outlet temperatures of 200°C and 120°C, respectively. The dryer was run at this condition for about 10 minutes prior to the introduction of the feed. The feed puree was passed through the spray-dryer chamber (500 mm x 21 mm) with the aid of a centrifugal pump. The speed of rotation of the pump controls the feed flow rate, which passes from the atomizer nozzle with an inner diameter of 0.5 mm. The inner temperature and feed rate were maintained at 160°C and 400 ml/h, respectively. After the spray-drying operation, the powder obtained was collected in a pre-weighed, insulated glass bottle connected at the end of the cyclone collector and packed in aluminum pouches, which were stored at 25°C in a desiccator containing activated silica gel prior to prompt use for analyses. The powders were produced 24 hours prior to the sensory evaluation and stored in air-tight sealed polyethylene containers, at low temperatures (20°C), and away from light and moisture to prevent degradation of flavor, color, and aroma compounds (Feguš *et al*., 2015)

Nutritional composition

Proximate analysis

The composite fruit purees' proximate compositions are shown in Table 3. The moisture, crude protein, crude fiber, fat, ash, and total carbohydrate contents and also the energy values of the working composite fruit puree and the fruit powders were determined according to the AOAC (2012) official methods.

Moisture content

Two grams of the sample (in triplicate) were

weighed into an empty, dry, and clean crucible of a known weight. The crucible containing the sample was placed in an oven at 105°C for 24 hours. After that, the crucible was removed and placed in a desiccator containing dry silica gel and weighed three times at 10 minutes intervals, and the weights were calculated as averages. This was repeated twice, and the moisture content was calculated as a percentage according to the following equation:

$$\% \text{ Moisture } = \frac{W_1 - W_2}{W_0} X\ 100 \quad (1)$$

Where: W_1 is the weight of crucible and sample before drying, W_2 is the weight of crucible and sample after drying, and W_0 is the original weight of the Sample.

Crude protein

For the test composite puree and each of the powders, 0.2 g of sample was placed in a 10 mL Kjeldahl digestion flask; then, 0.4 g of Kjeldahl catalyst tablets and 3.5 mL of concentrated sulfuric acid were added. The flask was heated in an electrical heater for 2 hours. The samples were cooled and diluted with distilled water and placed in the distillation apparatus. Twenty mLs of 50% sodium hydroxide (NaOH) was added, and the distillation took place for 10 minutes. The evolved ammonia received in 10 mL of 2% boric acid contained in a 100ml conical flask was titrated against 0.02M HCl using a universal indicator (bromocresol green and methyl red in alcohol). The protein content was calculated as a percentage according to the following equation:

$$\text{Protein Content } (\%) = \frac{\text{itre (less blank) x Molarity x 0.014 x 100 x 6.25}}{W0} \quad (2)$$

Where: W_0 is the original weight of the sample.

Crude fiber

Five grams of the sample was digested with tri-chloroacetic acid by refluxing for 40 minutes followed by filtration. The residue was washed with boiling distilled water and then with acetone. The washed residue was dry-heated at 150°C in an oven, and the dried residue was scraped into a porcelain crucible, weighed, and placed in a muffle furnace for ashing for 2 hours. After cooling in desiccators, the crucibles and contents were weighed. Crude fiber was calculated as a percentage as follows:

$$\text{Crude fibre}(\%) = \frac{W_1 - W_2}{W_0} X100 \quad (3)$$

Where: W_1 is the weight of crucible + ash, W_2 is the weight of crucible+ residue, and W_O is the initial weight of the sample.

Ash

Two grams of the sample were weighed into a clean ashing dish with a known weight. The ashing dish containing the sample was ignited in a muffle furnace at 550°C for 3 hours. The ashing dish was removed, cooled in a desiccator, and weighed again. The ash content of the sample was calculated as a percentage according to the following equation.

$$\text{Ash content } (\%) = \frac{W_1 - W_2 \times 100}{W_0} \quad (4)$$

Where: W_1 is the weight of empty ashing dish (before ignition), W_2 is the weight of the ashing dish containing the ash (after ignition), and W_0 is the original weight of the sample.

Fat

For each fruit powder and the original test composite puree, an extraction thimble was weighed empty, filled with sample up to half, and weighed again. The mouth of the extraction thimble was plugged with cotton wool to prevent the sample from spilling. The thimble containing the sample was then placed over petroleum ether. The extractor containing the thimble and sample was then fitted into the quick-fit flask and connected to the reflux condenser. The flask was heated on the heating mantle, and the extraction carried out for 16 hours, after which the petroleum spirit was evaporated. The weight of the flask and oil were determined after heating in boiling water to remove all traces of the solvent, followed by drying over calcium chloride. Fat content was determined as a percentage according to the following equation:

$$\text{Crude fat content } (\%) = \frac{D - C}{B - A} x100 \quad (5)$$

Where: A is the weight (g) of thimble, B is weight (g) of thimble + sample, (B - A) is the weight (g) of sample, C is weight (g) of empty quick fit flask, D is weight (g) of quick fit flask + oil and D - C = Weight (g) of oil.

Total carbohydrates

Total carbohydrates were determined by difference as follows:

100 - (% protein + % fat + % crude fiber + % moisture + % ash) (6)

Determination of energy values

The energy values of the samples were estimated by calculation using Atwater's Conversion factor (4 x %

Table 4. Vitamin composition of fresh, freeze-dried & spray-dried puree mixture (composite) (mg/100g).

Product	Vit.C	Vit. A	Vit. B1 (Thiamine)	Vit. B2 (Riboflavine)	Vit. B3 (Niacin)	Vit. B2 (Riboflavine)	Vit. B5 (Pantothenic acid)
Fresh sample	23.42±1.481[a]	16.64 ±1.428[a]	0.637 ±1.115[a]	0.052 ±1.435[a]	5.203 ±1.084[a]	0.052 ±1.301[a]	0.274 ±1.435[a]
SDFP	5.40±0.702[b]	0.01 ±0.770[b]	0.02 ±1.056[b]	0.02 ±0.770[b]	0.00±0.001[c]	0.021±1.201[b]	0.000 ±0.002[b]
FDFP	5.60±1.067[b]	0.04 ±1.060[b]	0.06 ±0.770[b]	0.06 ±1.036[a]	0.21 ±1.201[b]	0.063 ±1.037[ab]	0.127±1.021[ab]

Table 4. Contd.

Product	Vit.B6 (pyridoxine)	Vit.B9(folic acid)	Vit.E (α-tocopherol)	Vit. K	Vit B12 (Cobalamin)	Vit. C
Fresh sample	4.15	0.531±1.357[a]	1.073	1.567±1.037[a]	0.000±0.000[b]	17.851±1.315[a]
SDFP	5.16(µg)	2.430±1.541[b]	0.000	0.000 ±0.010[b]	0.000±0.010[b]	4.563±1.15[b]
FDFP	41.30(µg)	0.873±1.251[a]	4.23 (µg)	6.003 ±1.106[c]	0.000±0.010[b]	5.253±1.11[b]

Average results from at least triplicate determinations. Values are mean ± standard deviation (SD) of triplicate determinations. Samples with different superscripts within the same column were significantly ($p<0.05$) different. Key: FDFP = freeze-dried fruit powder; SDFP = spray-dried powdered powder.

protein + 9 x % fat + 4 x %carbohydrate) expressed in kcal/100g as reported by Onyeike *et al.* (2003).

Vitamins determination

The vitamin composition of the test composite puree and fruit powders in Table 4 were determined according to the methods described by Hassan and Hassan (2008) as follows:

Vitamin A precursor (Beta-carotene): The β-carotene in each of the samples (i.e., test composite puree, freeze-dried fruit powder, and spray-dried fruit powder, respectively) was extracted by dissolving 1 g of each in 50 ml of methanol followed by incubation for 2 hours under dark at room temperature for complete extraction. The mixture was then mixed with 5 ml of hexane and transferred into a separator funnel. The upper on-aqueous layer was separated through the funnel and the volume made up to 10ml with hexane and then passed through sodium sulphate layer in a filter funnel in order to remove any moisture from the layer. The absorbance of the layer was subsequently measured at 436mm using hexane as a blank (Ranganna, 1999). The *(3-carotene* was calculated using the formula:

β-carotene (mg/100g) = Absorbance (436mm) x V x D x 100 x 100 (7)

W X Y

Where: V is the total volume of extract, D is the dilution factor, W is the weight of the sample, and is the percentage dry matter content of the sample.

The β-carotene contents were expressed as retinol equivalents (RE) by multiplying each value by a factor of 0.167 (FAO/WHO, 2005).

Ascorbic acid (Vitamin C)

Vitamin C contents of the samples were determined according to the official titrimetric method described by AOAC Official Method 967.21(2012) using 2-6-dichlorophenol indophenol (DCPIP). By this procedure, 2g of each powder or the test composite puree was mixed with 100 ml of 0.1M metaphosphoric acid and 10 ml of each solution titrated with DCPIP. The end point of titration was determined by the change of color to pink. Ascorbic acid content was expressed as mg Ascorbic acid/100 g sample as follows:

Ascorbic Acid (mg/100g) = (Volume of DCPIP x Titration Factor x Dilution Factor) / Sample Weight (8)

Where: Volume of DCPIP (mL) is the volume of DCPIP used to titrate the sample, Titration Factor (mg/mL) is the amount of ascorbic acid equivalent to 1 mL of DCPIP (usually provided with the DCPIP reagent), Dilution Factor is the Dilution of the sample (if any) while, Sample Weight (g) is the weight of the sample taken for analysis.

Standard calculation

Ascorbic Acid (mg/100g) = (V x TF x DF) / (SW x 100) (9)

Where: V is the volume of DCPIP (mL), TF is the Titration Factor (mg/mL), DF is the Dilution Factor, and SW is the sample weight (g).

Tocopherol

Saponification and extraction of tocopherols were performed according to Górnaś *et al.* (2014). Essentially, 0.2 g of each powder or the test composite puree was dissolved firstly in 2.5 mL of absolute ethanol followed by the addition of 0.05 g of pyrogallol and then 0.25 mL of aqueous potassium hydroxide (600 g/L) in capped Pyrex glass test tubes. The test tubes were capped tightly immediately and mixed (10 sec) by gentle swirling and then incubated at 80˚C for. 25 min. After incubation, the samples were rapidly cooled in an ice-water bath for 5 min, and 2.5 mL of sodium chloride (10 g/L) was added to each and mixed for 5 seconds. The tocopherol content of each was extracted by mixing with 2.5 mL of *n*-hexane: ethyl acetate (9 :1 v/v) and shaken for 15s.The organic layer was separated by centrifugation (1000 ×*g*, at 4ºC, 5 min) and transferred to a round-bottom flask. Residual tocopherol was re-extracted twice following the protocol above. The combined extracts for each sample were dried by evaporation using a vacuum rotary evaporator. The dried extracts were dissolved in 0.5 ml methanol and filtrated through a syringe filter (0.22 *μ*m) into vials. Samples were injected directly after preparation into the RP-LC and SFC systems.

RP-LC (Reversed-Phase Liquid Chromatography), a type of liquid chromatography that separates compounds based on their hydrophobicity and SFC (Supercritical Fluid Chromatography), a type of chromatography that uses a supercritical fluid (e.g., CO_2) as the mobile phase were used to define and describe the conditions for the estimation of tocopherol as detailed above.

Conditions for estimation of tocopherol by SFC

Column: C18 packed column (150 x 4.6 mm, 5 µm), Mobile phase: CO2: EtOH (95:5 v/v), Flow rate: 2mL/min, Pressure: 200 bar, Temperature: 30°C and Detection: UV (280nm). Sample preparation: tocopherol was extracted from samples using hexane, Standard preparation: α and β-tocopherol standards were used, Calibration curve: the peak area was plotted vs concentration for each tocopherol isomer, Sample analysis: 20 µL of the sample extract was injected and the peak areas measured.

Estimation:

The calibration curve equation is y = ax + b.

Where: y = peak area (Integrated area under the chromatographic peak), x = concentration of tocopherol standard (mg/ml), a = slope, b = intercept.

Tocopherol quantification equation

Tocopherol (mg/g) = Peak area x slope x correction factor (accounts for mobile phase composition) / Injection volume x sample weight.

Tocopherol content calculation

Tocopherol content (mg/100g) = (Tocopherol amount/ sample weight) x100 (10)

Minerals determination

The selected mineral elements in Table 5 were analyzed using the method described by Fashakin *et al.* (1991). For each of the fruit powders and the test composite puree, a 0.5 g sample was held in a muffle furnace, and the resultant ash was mixed with 10 ml each of concentrated NHO_3 and HCL, respectively, in digestion flasks. Each mixture was digested for 10 min, followed by filtration through Wattman #1 filter paper. Each filtrate was made up to 50ml with distilled water. Calcium, sodium, iron, potassium, and magnesium were measured using an atomic absorption spectrophotometer (Perkin Elmer Analyst 400, USA).

Experimental Conditions (AAS)

Instrument: Perkin-Elmer 503, Wavelength: Element-specific (e.g., Ca: 422.7 nm, Mg: 285.2 nm)**,** Lamp current: 15 mA**,** Slit width: 1 nm**,** Flame type: Air-acetylene, Fuel flow: 5 L/min**,** Oxidant flow: 10 L/min.

Operational Temperature

Flame temperature: 2000-3000°C, Furnace temperature (for graphite furnace AAS): 100-3000°C

Quantification Equation (AAS)

C = (A / S) x DF (11)

Where: C is the Concentration of mineral element (mg/L or µg/mL), A = Absorbance reading, S = Slope of calibration curve (L/mg or mL/µg), and DF = Dilution factor.

Quantification Equation (ICP-AES)

C = (I / S) x DF (12)

Table 5. Mineral composition of fresh, freeze-dried & spray-dried puree mixture (composite) (mg/100g).

Product	Calcium	Iron	Phosphorus	Potassium	Sodium	Selenium	Iodine	Magnesium
Fresh Sample	22.59±1.031[a]	0.89±0.751[a]	7.22±1.301[a]	92.13±1.213[a]	15.82±1.203[a]	0.42±1.143[a]	0.005±1.032[a]	6.97±1.043[a]
FDFP	21.89±1.021[a]	0.88±0.681[a]	6.92±1.301[a]	90.93±1.043[a]	14.89±1.233[a]	0.38±1.043[a]	0.003±1.043[a]	5.94±1.203[a]
SDFP	19.54±1.213[b]	0.53±0.531[b]	4.25±1.131[b]	62.16±1.013[b]	12.83±1.243[a]	0.16 ±1.203[a]	0.001±1.240[a]	3.93±1.240[a]

Average results from at least triplicate determinations. Key: FDFP = Freeze-dried fruit powder; SDFP = Spray-dried fruit powder.

Table 6 Amino acid (AA) composition of fresh, freeze-dried & spray-dried puree mixture (composite) (mg/100g).

Amino acid	Glutamic acid	Phenyl alanine	Tyrosine	Tryptophan	Threonine	Valine	L-Citrulline	Arginine
Fresh Sample	27.27±0.631[a]	11.87±0.081[b]	8.47±0.781[b]	9.83±0.083[a]	10.07±0.071[b]	10.78±0.701[b]	118.05±0.731[a]	188.67±0.758[b]
FDFP	30.20±0.681[a]	40.87±0.181[a]	38.47±0.701[a]	39.83±0.081[b]	40.07±0.069[a]	41.78±0.780[a]	250.01±0.783[b]	181.14±0.785[a]
SDFP	14.94±0.601[b]	38.93±0.076[a]	37.13±0.780[a]	17.67±0.080[c]	38.32±0.087[a]	39.06±0.708[a]	181.43±0.738[c]	179.81±0.781[a]

Average results from at least triplicate determinations. Key: FDFP = freeze-dried fruit powder; SDFP = spray-dried fruit powder.

Where: C = Concentration of mineral element (mg/L or µg/mL), S = Slope of calibration curve (L/mg), DF = Dilution factor.

Calibration Curve Equation

$$y = mx + b \quad (13)$$

Where: y is Absorbance (AAS) or Emission intensity (ICP-AES), x is the Concentration of mineral element standard (mg/L or µg/mL), m is slope (S), b is the Intercept, and DF accounts for sample dilution.

Amino acids determination

The amino acid compositions of the composite fruit purees are shown in Table 6. The amino acids were determined by the method described by Liu *et al.* (2021) using High-Performance Liquid Chromatography (HPLC). Briefly, the amino acids were separated using reverse-phase HPLC with C18 column and mobile phase (acetonitrile: water, 80:20). The amino acids were detected using fluorescence detection (excitation: 340 nm, emission: 450 nm).

L-citrulline and L-arginine determination

Extraction and Quantification

L-citrulline and L-arginine extraction and quantification were as described by Jayaprakasha *et al.* (2011).

Extraction: Frozen puree samples were thawed at room temperature, and the powdered samples were weighed as 0.2 g +/− 0.01 g aliquots. Phosphoric acid (1.2 mL, 0.03 M) was added to samples before vertexing for 1 min. Samples were sonicated (30 min), left at room temperature (10 min), and then centrifuged for 20 min at 4°C, 5700× *g*; (Eppendorf, Model 5417R). A mL aliquot of supernatant was filtered (17 mm nylon syringe filter, F2513-2, Thermo Scientific) into amber HPLC vials and held at −80°C until HPLC analysis.

Quantification by HPLC: L-Citrulline and L-Arginine concentrations were determined using the method of Jayaprakasha *et al.* (2011) with modifications. Filtered samples (5 µL) were injected onto a High-Performance Liquid Chromatograph (Hitachi Elite LaChrom) equipped with a photodiode array detector and auto sampler. A Gemini 3u C18, 110 A, 250 × 4.6 mm. 00G-4439-EO column and guard column (C18 4 × 2.0; AJO-4286, Security Guard Cartridge), (Phenomenex, CA, USA) held at 25°C and a mobile phase of 15 mMphosphoric acid, 0.5 mL/min was used for peak separation. External standards of arginine and citrulline (Sigma) were used to verify and quantify these amino acid peaks.

Table 7. Phytochemical composition of fresh, freeze-dried & spray-dried puree mixture (Composite) (mg/100g)\.

Phytochemicals	Carotenoids	Total phenols	Alkaloids	Tannins	Total saponins	Phytosterols	Flavonoids	Cardiac glycosides	Oxalate	Phytate
Fresh sample	6.29±1.203^{b}	251.76±1.210^{b}	10.61±1.203^{a}	32.54±1.240^{a}	1.10±1.211^{b}	20.77±1.215^{b}	26.07±1.203^{a}	1.06±1.230^{b}	36.85±1.241^{b}	2.51±1.231^{b}
FDFP	16.29±1.241^{a}	261.76±1.213^{a}	21.61±1.240^{b}	42.54±1.234^{b}	21.10±1.212^{a}	30.77±1.202^{a}	36.07±1.213^{b}	21.06±1.202^{c}	46.85±1.242^{a}	12.51±1.232^{a}
SDFP	13.68±1.201^{a}	259.03±1.233^{a}	3.00±1.243^{a}	17.67±1.206^{c}	19.98±1.213^{a}	28.37±1.243^{a}	14.83±1.223^{c}	9.09±1.223^{a}	44,62±1.243^{a}	11.23±1.233^{a}

Average results from at least triplicate determinations. Keys: FDFP = freeze-dried fruit powder; SDFP = spray-dried fruit powder

Phytochemicals analysis

The phytochemical content of the dried and fresh samples of the fruits in Table 7 was determined according to the methods described by AOAC (2012). 10 g of the fruit powders (10 ml of fresh samples) was added to l00 mLs of distilled water (at normal room temperature) inside a conical flask and plugged with cotton wool. After 24 hours (12 hours for fresh juice), the mixture was filtered using cheese cloth and then through Whatman No. 1 filter paper. The filtrate was then concentrated using a rotary evaporator (Iweala and Okeke, 2005).

Tannins

1 ml of the extract was added to a 10 ml volumetric flask containing 4 ml water. At times zero minute, 0.3 ml of 5% NaNO was added to each volumetric flask. At 5 minutes, 0.3 ml of 10% $A1CL_3$ was added; at 6 minutes, 3 ml of 1 m NaOH was added. Each reaction flask was then immediately diluted with 2 - 4 mL of H_2O and mixed. Absorbance upon development of pink colorways was determined at 510 nm relative to a prepared blank. The total tannin content of the sample was expressed in milligrams of Gallic acid per 100 mL sample.

Phenolic

100 mg of the extract of the sample was weighed accurately into 100 ml of triple distilled water (TDW). 1 ml of this solution was transferred to a test tube, then 0.5 mL 2 N of Folin-iocalteu reagent and 1.5 mL 20% of Na_2CO_3 solution was added and ultimately, the volume was made to 8 ml with TDW followed by rigorous shaking and finally allowed to stand for 2 hours after which the absorbance was taken at 765 nm. These data were used to estimate the total phenolic content using a standard calibration curve obtained from various diluted concentrations of garlic acid (Abdelhady *et al*., 2011).

Saponins

20 g of the sample was placed in a conical flask, and 100 mLs of 20% aqueous ethanol was added and heated in a hot water (55°C) bath for 4 hours with continuous stirring. The mixture was filtered, and the residue was re-extracted with another 200 mL of 20% ethanol. The extract was reduced to 40 ml over a water bath at about 90°C. The concentrate was transferred into a 250 mL separator funnel, and 20 mL diethyl ether was added and shaken rigorously. The aqueous layer was recovered, while the ether layer was discarded. The purification process was repeated. 60 mL of n-butanol was added. The combined n-butanol extracts were washed twice with 10 ml of 5% aqueous sodium chloride. The remaining solution was heated in a water bath. After evaporation, the samples were dried in the oven to a constant weight; the saponin content was calculated according to Obadoni and Ochuko (2001).

Flavonoids

Total flavonoids were determined according to the Aluminum Chloride (AlCl3) Colorimetric Method described by Ahmed *et al.* (2022). 10 g of the sample was repeatedly extracted with 100 ml of 80% aqueous methanol at room temperature and 1 mL of extract mixed with 1 mL of 2% AlCl3 solution. 3 mL of methanol was added and incubated at room temperature for 30 minutes. Using a spectrophotometer, absorbance at 415 nm was measured, and the total flavonoids were calculated using a calibration curve with quercetin as a standard equation:

$$\text{Total Flavonoids (mg/g or mg/mL)} = (A \times DF \times CF) / (\varepsilon \times l) \quad (14)$$

Where: TF = Total Flavonoids (mg/g or mg/mL), A = Absorbance at 415 nm, DF = Dilution Factor, CF

= Calibration Factor (mg/mL), ε = Molar Extinction Coefficient (L/mol/cm), l = Path Length (cm).

Standard Curve: Using quercetin solutions (0-100 µg/mL), Absorbance: at 415 nm

Plot: Absorbance vs. Concentration, Calibration Factor (CF): Slope of standard curve / Molar mass of standard, Molar Extinction Coefficient (ε): ε = 37,600 L/mol/cm (for quercetin).

Alkaloids

The total alkaloids of the samples were determined using the High-Performance Liquid Chromatography (HPLC) method described by Liu *et al.* (2021). The sample was extracted with methanol, and the alkaloids were separated using HPLC with a C18 column and mobile phase (acetonitrile: water, 80:20).

Extraction: 5 g of the sample was weighed into a 250 ml beaker, and 200 ml of 10% acetic acid in ethanol was added, covered, and allowed to stand for 4 hours. Alkaloids were detected at 280 nm using a UV detector, and the total alkaloids were calculated using the peak area and calibration curve with standard alkaloids.

Equation:

Alkaloid Content (mg/g) = (PA / WA) × (CF / DF) (15)

Where: PA = Peak Area of alkaloid, WA = Weight of sample (g), CF = Calibration Factor (mg/mL), DF = Dilution Factor

HPLC Parameters: Column: C18, Mobile Phase: Methanol: Water (70:30), Flow Rate: 1-2 mL/min, Injection Volume: 10-20 µL, Detection Wavelength: 280 nm, Calibration Curve: Using solutions of alkaloid (atropine)

Plot: Peak Area vs. Concentration

Calculation: Calibration Factor (CF): Slope of standard curve / Molar mass of standard

Phytate

Phytate in the samples was determined using acid extraction followed by a colorimetric method for detection according to the molybdenum Method described by De Angelis *et al.* (2024).

Carotenoids

Carotenoids were analyzed by the method of reversed phase HPLC using a Waters 600 system equipped with UV–visible photodiode array detector, recording absorption spectra between 250 and 500nm as described by Chen *et al*. (2004). These parathions of all-*trans*-β-carotene and 13-*cis*-β-carotene were carried out in a C30 carotenoid column. The analytes were eluted from the column with different proportions of methanol (MeOH) and methyl tert-butyl ether (MTBE), with a flow rate of 1 mL/min. The gradient initial conditions were 75%(v/v) MeOH and 25%(v/v) MTBE. At 17 minutes, the ratio varied to 30% MeOH and 70% MTBE and at 26 minutes to 75% and 25% MeOH and MTBE, respectively, up to a finish at 30 minutes, with a flow rate of 1mL/min. The gradient allowed separations of all-*trans*-β-carotene and its geometrical isomers (9, 13 and 15-*cis*-carotene). The retention times for all-*trans*-β-carotene and 13-*cis*-β-carotene were approximately 21.20 min and 17.00 min, respectively. Other isomers were not detected. Quantification of all-*trans*-β-carotene was done based on linear calibration curves of eight standard solutions of all-*trans*-β-carotene and using response factors for quantification of the *cis*-isomers according to Bengtsson *et al*. (2008). The injection volume was 20 µL. Carotenoids were identified using retention times and UV/v is absorption spectra. The concentration of all-*trans*-β Carotene was expressed as micrograms per g dry matter, given as the mean of triplicate extractions. The percentage retention of all-*trans*-β-carotene was calculated as the ratio of all-*trans*-β-carotene in the treated sample to fresh sample x 100.

Cardiac glycosides

Cardiac glycosides were determined using HPLC (High-Performance Liquid Chromatography) after extraction with methanol or ethanol and purification by partitioning as described by Ikeda *et al.* (1991).

Oxalic acid

Total Oxalic Acid was determined using the method of AOAC (2012) as described by Savage *et al.* (2000). One-gram samples of "newly" ground freeze-dried food was weighed into a 250 mL beaker, and 50 mL 2 M HCL was added. The beakers were placed in a water bath at 80^{o}C for 15 minutes. The extract was allowed to cool and then transferred quantitatively to a 100 mL volumetric flask and made up to volume with 2 M HCl. Three extractions were carried out for each food sample.

Sample analysis: The extracts were then centrifuged at 3000 rpm, and 10 mL of the supernatant was filtered through a 0.45 mm cellulose acetate membrane (Satorius, Goettingen, Germany). A 5 µL sample was analyzed using a Waters Chromatography System, consisting of a Waters 717 plus auto sampler, Waters 600-MS Isocratic/Gradient Pump, and a Waters UV/VIS

detector set at 210 nm. Data capture and processing were carried out using the Millennium (ver 2.15) chromatographic software. The chromatographic separation was carried out using an Aminex Ion exclusion HPX-87H3007.8 mm analytical column attached to an Aminex Cation-H guard column, using an isocratic elution at 0.5 mL/min with 0.0125 M sulphuric acid (Analar, BDH, UK) as a mobile phase. The analytical column was held at room temperature, and the columns were equilibrated at a flow rate of 0.1 mL/min prior to use and in between sample sets. Before use, the mobile phase was filtered through a 0.45 m membrane and degassed using a vacuum. The oxalic acid peak was identified by comparison of the retention time to a range of common plant organic acid standards.

Standard calibration: Two standard curves were prepared in the range 1-20 mg/100 mL; one set of standard solutions was prepared by adding oxalic acid (Analar, BDH, UK) to 100 mL volumetric flasks and making up to volume with distilled water. This set was used to analyze the soluble oxalic acid content of the water extracts. The second set of standard solutions was prepared by diluting the standard oxalic acid to 100 mL with 2.0 M HCl. This set of standards was used to quantify the total oxalic acid content of the samples. All blank and standard solutions were filtered through a 0.45 mm cellulose acetate membrane syringe filter prior to analysis.

Phytosterol

The identification and quantification of the major and minor sterol components of the FP were according to the AOCS Ce 12–16 methods (AOCS, 2009) as described by Gomes *et al*. (2019). The sample was diluted in chloroform at a concentration of 1 mg/mL. Then, 0.5 mL of this solution was added to 0.5 mL of internal reference solution composed of 1 mg/mL α-cholesterol in chloroform. The sample and internal reference were dried under nitrogen. After drying, the sample was derivatized with 500 µL of a 1:3:9 (v/v/v) trimethylchlorosilane: hexamethyldisilane: pyridine solution and analyzed by GC. The gas chromatograph Agilent 6850 Series GC System (Santa Clara, CA, USA) was equipped with an automated liquid sampler (1 µL injections), split injector (1:50 split ratio), a fused silica low-polarity capillary column [Rxi-5HT (poly (5% diphenyl-95% dimethyl) siloxane; length, 30 m; intern diameter, 0.25 mm, and 0.25 µm film thickness, Restek], and FID. The oven program for the determination of sterols was set isothermally at 265°C. Helium was used as carrier gas at a flow rate of 1 mL/min. The temperatures of the injector and detector were 300°C. The quantitative determination was done using the internal reference of α-cholesterol. The data were expressed as the total percentage of sterols in the sample, by the ratio of the internal reference, and the percentage of the area of each sterol in the sample. Peak identification was performed by calculation of the retention time and comparison to the reference chromatograms.

Functional properties

Bulk density

Bulk densities of the freeze dried (FD) and spray dried (SD) fruit powders samples were determined as described by Onwuka (2018). Essentially, 50 g of each powder was poured into a 100 ml capacity glass measuring cylinder followed by gentle tapping to a constant volume. Bulk density of each was then calculated as follows:

$$\text{Bulk Density (g/ml)} = \frac{\text{wt of sample}}{\text{volume of sample after tapping (ml)}} \quad (16)$$

Specific Gravity (SG)

Specific gravities of the fruit samples were determined using density bottles. Each sample was poured into a 50 ml density bottle and weighed. The mass of each less weight of the density bottle was divided by the volume of the density bottle to get the density. The specific gravity was then calculated as density of sample relative to density of water at the same temperature as follows:

Density of Sample x X (g/ml)

Density of water = 0.998 (g/ml).

And

$$\text{X} = (W_2 - W_1\,(g))/Vml \quad (17)$$

Where: W_2 = Weight of sample + density bottle, Wi = Weight of density bottle, V = Volume of the density bottle (50ml).

Viscosity (cP)

Viscosity of the fruit samples were measured using a Brookfield viscometer DV-II+ Pro, USA (Wong *et al.,* 2015). 250 mL of samples were added with spindle no.2 at 25°C, and the reading measured at a speed of 150 rpm.

Water absorption capacity (WAC)

The WAC of the samples was determined using the method of Onwuka (2018). One gram of the sample was dispensed into a weighed centrifuge tube with 10 ml of distilled water and mixed thoroughly. The mixture was

allowed to stand for 1 hour prior to centrifugation at 3,500 rpm for 30 minutes. The excess water (unabsorbed) was decanted, and the tube inverted over an absorbent paper to drain dry. The weight of water absorbed was determined by difference. The water absorption capacity was calculated as:

$$\text{WAC}(\%) = \frac{\text{Volume of Water used} - \text{Volune of free water}}{\text{Weight of sample used}} x\ 100 \qquad (18)$$

Oil absorption capacity (OAC)

The OAC of the fruit samples was determined using the method adapted from Que *et al. (*2008) with slight modifications. One gram of the dried fruit powder was weighed into a clean, dry centrifuge tube, and 10 milliliters of refined vegetable oil (soybean oil) was added to the tube containing the sample. The mixture was vortexed for 1 minute to ensure thorough mixing of the powder and oil, and the mixture was allowed to stand at room temperature for 30 minutes to enable the powder to absorb the oil. The mixture was centrifuged at 4,000 rpm for 25 minutes to separate the oil-absorbed powder from the unabsorbed oil, and the supernatant oil was carefully decanted without disturbing the sediment. The tube with the oil-absorbed powder residue was weighed and recorded.

Calculation: The amount of oil absorbed was calculated by subtracting the initial weight of the powder from the weight of the oil-absorbed powder residue using the formula:

OAC (g oil/g sample) = (Weight of oil-absorbed powder - Initial weight of powder) / Initial weight of powder (19)

RESULTS AND DISCUSSION

The results of this study reveal significant differences between freeze-dried fruit powders (FDFP) and spray-dried fruit powders (SDFP) in terms of nutritional composition, functional properties, and sensory attributes. Freeze-drying preserved higher levels of key nutrients such as vitamin C, with FDFP containing 5.60 mg/100 g compared to 5.40 mg/100 g in SDFP. Similarly, carotenoid content in FDFP (16.29 mg/100 g) was significantly higher than in SDFP (13.68 mg/100 g), consistent with previous findings by Silva-Espinoza *et al*. (2019) and Muizniece-Brasava *et al*. (2020) that freeze-drying better retains heat-sensitive compounds.

These differences in nutritional retention and functional properties directly influence the usability and consumer preference for these powders. The superior nutrient preservation in FDFP makes it more desirable for health-conscious consumers and applications where maintaining high levels of vitamins and antioxidants is crucial, such as in dietary supplements or functional foods. Additionally, freeze-dried powders often have a finer texture and better solubility, enhancing their ease of incorporation into smoothies, beverages, and culinary applications. In contrast, while SDFP may offer advantages such as cost-effectiveness and higher bulk density, its slightly lower nutritional content and potential textural variations may affect its appeal in premium health-oriented markets. Understanding these distinctions can guide manufacturers in selecting the appropriate drying method based on product goals and consumer expectations.

The proximate analysis revealed significant differences between the two drying methods, which could have important implications for consumer preferences and food product formulation. FDFP had higher protein (0.78%) and fat (2.76%) contents compared to SDFP (0.31% and 1.11%, respectively), likely due to the gentler freeze-drying process minimizing nutrient degradation (Natumanya *et al*., 2021). These differences suggest that freeze-dried products may be more appealing to consumers seeking nutrient-dense foods, particularly in health-focused markets. However, the slightly higher carbohydrate content in SDFP (6.15% vs. 5.51% in FDFP) might offer advantages in applications requiring higher energy content, such as meal replacements or sports nutrition products.

Functional properties also play a crucial role in food processing and consumer experience. The lower bulk density of FDFP (0.31 g/cm^3) compared to SDFP (0.44 g/cm^3) enhances its reconstitution properties, making it more suitable for instant beverages or rehydrated food products (Jeyanth *et al*., 2020). This characteristic could be particularly beneficial for the development of convenient, high-quality powdered foods that dissolve quickly and easily.

From a health and wellness perspective, phytochemical analysis further supports the advantages of FDFP. Its higher antioxidant levels, with total phenols at 261.76 mg/100 g compared to 259.03 mg/100 g in SDFP, suggest that freeze-dried products retain more bioactive compounds (Latinović *et al*., 2024; Liu *et al*., 2021). This enhanced nutritional profile may be attractive to health-conscious consumers and could be leveraged in marketing claims for functional foods and nutraceuticals.

Finally, sensory evaluation demonstrated a clear consumer preference for FDFP, with significantly higher scores for appearance, taste, and overall acceptability ($p<0.05$). The superior sensory attributes of FDFP, likely due to better nutrient retention and a more appealing texture (Lim *et al*., 2021), highlight its potential for premium food products where taste and texture are key selling points. These findings indicate that freeze-drying may be the preferred processing method for manufacturers aiming to develop high-quality, consumer-appealing products.

Conclusion

This study demonstrated that freeze-drying is a superior method for producing high-quality fruit powders from

watermelon, orange, and mango purees. It preserves higher levels of essential nutrients, bioactive compounds, and functional properties compared to spray drying. Freeze-dried powders also exhibited greater sensory appeal, confirming their potential as a viable solution to postharvest losses and as a convenient, nutritionally dense food product. These findings support the application of freeze-drying in the production of value-added functional foods, contributing to food security and sustainable agricultural practices.

CONFLICTS OF INTEREST

The authors declare that they have no conflict of interest.

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