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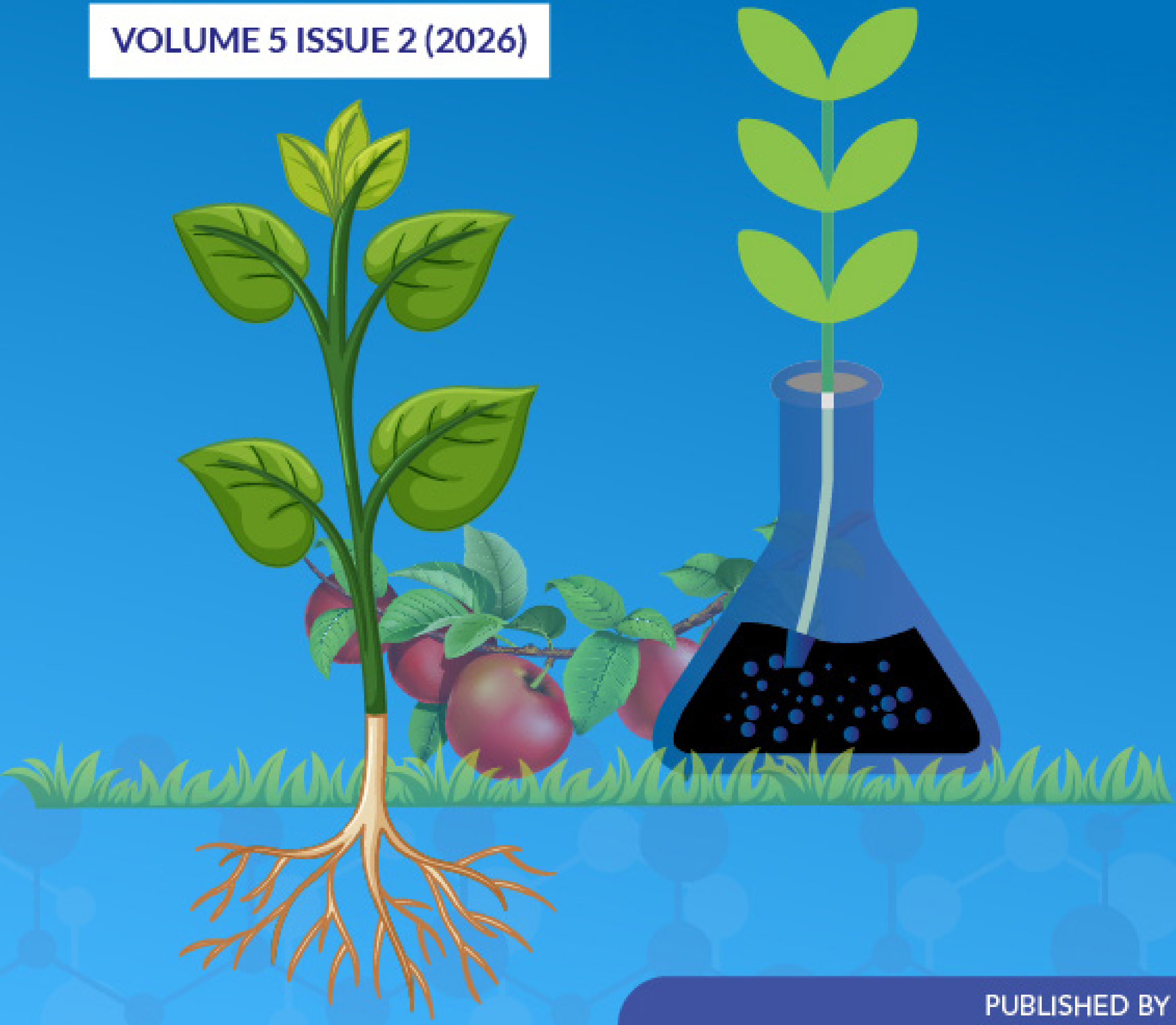

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Effect of Freeze-Drying on the Nutritional Composition of Watermelon–Orange–Mango Composite Fruit Powder

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Composite Puree, Freeze-Drying, Fruit Powder, L-Citrulline, Mangonutritional Composition, Orange, Watermelon

ABSTRACT

This study investigated the effect of freeze-drying on the nutritional composition of a watermelon–orange–mango composite fruit powder. Individual watermelon, orange and mango purees were produced and used to formulate six composite fruit blends. The formulations were subjected to sensory evaluation, and the most acceptable formulation, comprising 50% watermelon, 30% orange and 20% mango, was selected for freeze-drying. The selected composite puree was freeze-dried at an initial freezing temperature of −45 °C under vacuum, and the nutritional composition of the fresh puree and resulting freeze-dried fruit powder (FDFP) was determined. Freeze-drying reduced moisture content from 83.45 ± 3.34% in the fresh composite puree to 3.55 ± 3.34% in the FDFP. Correspondingly, measured concentrations of ash, protein, crude fibre, fat and carbohydrate increased from 0.74, 0.96, 0.74, 0.50 and 13.61% to 2.76, 4.39, 2.89, 0.78 and 85.63%, respectively. Most vitamins and all minerals analysed showed higher measured concentrations in the FDFP, although folic acid decreased from 4.10 to 0.53 mg/100 g and vitamin B12 was not detected in either sample. The concentrations of several amino acids also increased following freeze-drying, with L-citrulline increasing from 118.05 ± 3.86 to 250.01 ± 0.03 mg/100 g and arginine from 8.67 to 181.14 mg/100 g. The results indicate that freeze-drying effectively converted the high-moisture composite puree into a low-moisture fruit powder while retaining measurable nutritional constituents. However, the apparent increases in nutrient concentrations should principally be interpreted as concentration effects resulting from water removal rather than synthesis or necessarily complete nutrient retention. The findings demonstrate the potential of freeze-drying as a preservation and value-addition technology for watermelon–orange–mango composite fruit products.

INTRODUCTION

Fruits constitute an important component of a healthy and balanced diet because they provide carbohydrates, dietary fibre, vitamins, minerals and other nutrients required for normal physiological functions. They also contribute to dietary diversity and provide important micronutrients that support human health. However, fruits are among the most perishable agricultural commodities because of their high moisture content, active metabolic processes and susceptibility to microbial and physiological deterioration. After harvest, respiration and other biochemical activities continue, while the high moisture content of fresh fruits provides favourable conditions for chemical reactions and microbial growth. These characteristics contribute to their relatively short shelf life and make appropriate postharvest handling, preservation and processing essential for maintaining their quality and nutritional value (Grover & Negi, 2023).

Postharvest losses of fruits constitute an important challenge in developing countries, including Nigeria, where limitations in harvesting practices, transportation, storage infrastructure and processing facilities can increase deterioration along the supply chain. In a study of orange fruit in Nigeria, Obayelu *et al.* (2022) reported measurable postharvest losses among producers, wholesalers and retailers and identified storage practices and other supply-chain factors as important determinants of loss. The authors recommended improved harvesting, storage and postharvest management practices to reduce losses. Such losses not only reduce the economic returns obtained from fruit production but can also limit the availability of nutrient-rich fruits for consumption and processing. The development of appropriate technologies for extending fruit shelf life is therefore important for improving food utilization, reducing postharvest losses and increasing the value derived from fruit crops.

Watermelon (Citrullus lanatus), sweet orange (Citrus sinensis) and mango (Mangifera indica L.) are important tropical and subtropical fruits with considerable nutritional and economic value. Although each fruit possesses its own characteristic nutritional profile, their combination provides an opportunity to develop a composite product containing nutrients derived from three complementary fruit sources. The use of such a composite formulation may also provide a practical means of utilizing different fruits that become abundant during their respective production seasons.

Watermelon is a high-moisture fruit that contains carbohydrates, vitamins and minerals and is particularly notable for its L-citrulline content. L-citrulline is a non-protein amino acid that occurs naturally in watermelon and is one of the nutritional constituents that distinguishes

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the fruit from many other commonly consumed fruits. A recent comprehensive review of watermelon nutrition reported that the fruit contains several essential nutrients, including potassium, magnesium, calcium, phosphorus and vitamins, while L-citrulline is present in appreciable quantities in the edible portion (Baião *et al.*, 2025). L-citrulline is metabolically related to L-arginine and nitric oxide synthesis, making its occurrence in watermelon of particular nutritional interest.

Sweet orange (Citrus sinensis) is one of the most widely consumed citrus fruits and is commonly eaten fresh or processed into juice and other products. Citrus fruits generally contain high proportions of water and provide dietary fibre, carbohydrates, vitamins and minerals. Sweet orange is particularly recognized as a source of vitamin C and also contributes other micronutrients, including folate, thiamine and potassium (Aslan *et al.*, 2024). An updated review of Citrus sinensis similarly describes the fruit as containing vitamins and other nutritionally important constituents, with its composition varying according to cultivar, maturity, growing environment and postharvest conditions. The inclusion of orange in a composite fruit formulation can therefore contribute substantially to the micronutrient profile of the resulting product.

Mango (Mangifera indica L.) is another nutritionally important tropical fruit widely consumed in fresh and processed forms. Mango pulp contains carbohydrates, amino acids, vitamins and minerals and has considerable nutritional value. A detailed review of the nutritional composition of mango reported that the pulp contains a variety of nutrients and other functional constituents, with its composition differing among cultivars and fruit tissues (Pleguezuelo *et al.*, 2021). In addition, Sarkar *et al.* (2022) demonstrated that mango contains appreciable vitamins, carotenoids, organic acids and minerals and that processing conditions can influence the levels of these constituents. Thus, mango can contribute complementary nutrients to a watermelon–orange–mango composite formulation.

The nutritional characteristics of these three fruits provide a sound basis for their combination into a composite fruit product. Watermelon contributes moisture, minerals and L-citrulline; orange contributes vitamin C and other micronutrients; while mango contributes carbohydrates, vitamins, minerals and amino acids. However, the high moisture content responsible for the freshness and palatability of these fruits also contributes to their rapid deterioration. Converting the fruits into a more stable form, such as a powder, may therefore provide an alternative means of extending their usability beyond the period of fresh-fruit availability.

Drying is one of the established methods of preserving fruits because removal of water reduces the moisture available for microbial growth and many deteriorative reactions. Conversion of fruit puree into powder can also reduce product weight and volume and facilitate packaging, transportation, storage and incorporation into other food products. Nevertheless, the drying method employed is important because heat, oxygen exposure, processing time and other operating conditions can affect the nutritional and physicochemical characteristics of the resulting product. Reviews comparing drying technologies have shown that different drying methods can produce substantially different effects on the quality characteristics of fruits and vegetables (Sagar & Kumar, 2010; Aversa *et al.*, 2020).

Freeze-drying, also known as lyophilization, is a dehydration technique in which water is removed from a frozen product principally through sublimation under reduced pressure. The process generally involves freezing, primary drying, during which ice is sublimated, and secondary drying, during which residual moisture is removed. Because the principal removal of water occurs from the frozen state, freeze-drying can reduce the thermal exposure experienced by food materials compared with many conventional drying processes. The method is therefore widely investigated for high-value food materials in which preservation of nutritional and quality attributes is important (Yao *et al.*, 2023).

However, freeze-drying should not be regarded as a process that invariably preserves every nutrient completely. The freezing rate, product temperature, chamber pressure, shelf temperature, drying time and characteristics of the food matrix can influence the final quality of a freeze-dried product. Yao *et al.* (2023) emphasized that optimization of freeze-drying conditions is important for improving product quality and process efficiency. Similarly, Silva-Espinoza *et al.* (2020) demonstrated that changes in freezing rate, shelf temperature and pressure affected the physicochemical characteristics and nutrient-related quality attributes of freeze-dried orange puree. In their study, vitamin C retention was influenced by the processing conditions, demonstrating that the nutritional response to freeze-drying depends on the specific process parameters and food matrix.

Recent research further indicates that the effect of drying on nutritional constituents is nutrient-specific. For example, Sarkar *et al.* (2022) investigated the effect of drying on vitamins, carotenoids, organic acids and minerals in mango and demonstrated that drying could alter the levels of individual nutritional constituents. Consequently, evaluation of the nutritional composition of a fresh fruit puree and its freeze-dried counterpart is necessary to determine the actual changes associated with a particular drying process rather than assuming uniform nutrient retention.

The production of a watermelon–orange–mango composite powder may therefore provide a useful approach to fruit preservation and value addition. The composite formulation combines fruits with different nutritional characteristics and may provide a convenient, shelf-stable ingredient for use in beverages and other food products. More importantly, conversion of the composite puree into powder could provide an avenue for extending the period during which the nutrients contained in these fruits can be utilized, while reducing some of the limitations associated with the storage and transportation of highly perishable fresh fruits.

Despite the increasing application of freeze-drying to fruits and fruit-based products, there remains a need for information on the nutritional response of composite fruit matrices containing watermelon, orange and mango. The nutritional composition of a composite puree cannot necessarily be predicted from studies conducted on individual fruits because interactions among the constituent fruits, their relative proportions, moisture content and processing conditions may influence the composition of the final product. In particular, information on the effect of freeze-drying on the proximate composition, vitamins, minerals and amino acid profile of a watermelon–orange–mango composite puree is limited. The evaluation of L-citrulline and L-arginine is also relevant because watermelon is an important dietary source of L-citrulline and these amino acids are specifically included among the nutritional constituents investigated in the present study. Recent work has also demonstrated the importance of reliable analytical methods for determining L-citrulline in watermelon cultivars, further supporting the need for quantitative assessment of this constituent (Tran *et al.*, 2024).

Therefore, this study was undertaken to produce watermelon–orange–mango composite puree, determine the most acceptable formulation and convert the selected composite puree into powder by freeze-drying. The nutritional composition of the fresh composite puree and freeze-dried composite powder was evaluated based on proximate composition, vitamins, minerals and amino acids, including L-citrulline and L-arginine. The study is expected to provide information on the suitability of freeze-drying as a preservation and value-addition technology for watermelon, orange and mango and contribute to the development of a more stable fruit product that can facilitate improved utilization of seasonal fruits and reduction of postharvest losses.

Aim and Objectives of the Study

Aim of the Study

The aim of this study was to produce watermelon–orange–mango composite fruit powder by freeze-drying and evaluate the effect of the process on its nutritional composition.

Objectives of the Study

The specific objectives of the study were to:

- Produce individual and composite watermelon, orange and mango fruit purees and determine their nutritional compositions.
- Evaluate the sensory acceptability of the formulated watermelon–orange–mango composite purees and select the most acceptable formulation for freeze-drying.
- Produce powder from the selected watermelon–orange–mango composite puree using freeze-drying under specified processing conditions and evaluate its nutritional quality characteristics.
- Determine and compare the nutritional composition of the fresh composite puree and freeze-dried composite fruit powder in order to evaluate the effect of freeze-drying on their nutritional composition.

Scope of the Study

This study was limited to the development of watermelon (Citrullus lanatus), orange (Citrus sinensis) and mango (Mangifera indica L.) individual and composite fruit purees and their conversion into fruit powder by freeze-drying. The study covered the determination of the nutritional composition of the individual fruits and formulated composite purees, sensory evaluation of the composite formulations, and selection of the most acceptable formulation for freeze-drying. The selected composite puree was subsequently freeze-dried under specified processing conditions to produce fruit powder, which was evaluated for its nutritional quality. The nutritional composition of the fresh selected composite puree was compared with that of the resulting freeze-dried powder to determine changes associated with the freeze-drying process.

The study was restricted to the selected watermelon, orange and mango fruits, the formulated composite puree proportions, the sensory attributes evaluated, the specified freeze-drying conditions, and the nutritional quality parameters included in the experimental analyses. Phytochemical profiling, antioxidant activity, detailed functional properties and other quality characteristics not specified in the study objectives were outside the scope of the study.

LITERATURE REVIEW

Watermelon, Orange and Mango as Raw Materials for Fruit Blends

Watermelon (Citrullus lanatus), sweet orange (Citrus sinensis) and mango (Mangifera indica L.) are nutritionally important fruits with complementary compositional and sensory characteristics. Their high moisture contents, however, contribute to rapid deterioration and limit their storage life, creating a need for processing approaches that improve stability while retaining nutritional and sensory quality.

Watermelon is characterized by high moisture content and relatively low fat content, while red-fleshed cultivars are notable for lycopene and L-citrulline. The concentration of these constituents varies with cultivar, maturity and growing conditions. Watermelon is therefore relevant to fruit-product development both as a source of nutrients and as a high-moisture fruit suitable for blending and dehydration. .

Sweet orange contributes soluble sugars, organic acids, vitamin C and citrus bioactive compounds. Citrus fruits contain carotenoids, flavonoids, limonoids and terpenes, while citric acid contributes substantially to their characteristic acidity. These characteristics make orange particularly useful in fruit formulations where sweetness, acidity, flavour and nutritional composition need to be balanced (Saini *et al.*, 2022).

Mango pulp is characterized by appreciable soluble solids, sugars, pectin, vitamins, minerals, carotenoids and phenolic compounds. Its relatively high pulp content, sweetness, colour and characteristic aroma make mango suitable for blended fruit products. Nutritional composition varies considerably with cultivar, maturity and fruit part (Lebaka *et al.*, 2021). (PubMed Central

The combination of these fruits can therefore provide complementary sweetness, acidity, flavour, colour and nutritional characteristics. However, the quality of a composite puree depends on the proportions of the component fruits. Changes in formulation can alter soluble solids, acidity, colour, viscosity and consumer acceptance. Consequently, formulation and sensory evaluation are important preliminary steps before selecting a blend for dehydration.

Composite Fruit Purees and Sensory Acceptability

Fruit blending is commonly used to obtain desirable combinations of flavour, colour, sweetness and acidity and to improve the utilization of highly perishable fruits. For a watermelon-orange-mango product, watermelon can contribute a mild flavour and high moisture content, orange can provide acidity and characteristic citrus flavour, while mango can contribute sweetness, pulp solids and characteristic colour and aroma.

Sensory evaluation is therefore necessary to determine whether the nutritional and technological advantages of a formulation correspond with consumer acceptability. This is particularly important because the most nutritionally favourable formulation is not necessarily the most acceptable to consumers. Research on mixed fruit pulps has demonstrated that formulation and drying method can influence physicochemical and sensory attributes simultaneously. For example, Silva Júnior *et al.* (2022) found that freeze-dried mixed fruit pulp showed favourable retention of several quality attributes and achieved high sensory acceptance.

For the present study, sensory evaluation serves a specific purpose: selection of the most acceptable watermelon-orange-mango composite puree for subsequent freeze-drying. This provides a logical link between formulation development and powder production.

Freeze-Drying of Fruit Purees

Freeze-drying, or lyophilization, is a dehydration process in which water is removed predominantly by sublimation from a frozen product under reduced pressure. The process consists principally of freezing, primary drying and secondary drying. Because the material remains frozen during most water removal, thermal exposure is generally lower than in conventional hot-air drying.

Freeze-drying is particularly attractive for fruit products because it can produce porous, low-moisture materials with relatively good retention of colour, structure, nutrients and other quality characteristics. However, the process is considerably more energy intensive and slower than many conventional drying methods. Bhatta *et al.* (2020) identified nutrient retention and structural preservation as important advantages, while also highlighting the high energy requirement associated with freeze-drying.

More recent evidence confirms that freeze-drying performance depends on raw-material composition, freezing conditions, chamber pressure, shelf temperature, drying time and storage conditions. Uwineza and Zhang (2026) emphasized these factors as important determinants of both process efficiency and final food quality.

Effect of Freeze-Drying on Nutritional and Quality Characteristics

The principal objective of dehydration is to reduce water content sufficiently to improve product stability while minimizing undesirable changes in nutritional and sensory quality. Freeze-drying can reduce moisture and water activity and produce a porous structure that facilitates rehydration. Nevertheless, the extent to which nutrients are retained depends on the composition of the fruit matrix and the conditions employed during freezing, primary drying, secondary drying and storage.

Vitamin C is particularly relevant because it is susceptible to oxidation and processing-related degradation. Studies involving fruit products indicate that freeze-drying can provide comparatively favourable retention of heat-sensitive nutrients, although retention is not complete and varies among food matrices. In freeze-dried orange puree, Silva-Espinoza *et al.* (2021) demonstrated that formulation and carrier composition influenced the retention and bioaccessibility of vitamin C, phenolic compounds and β-carotene.

The effect of freeze-drying on physical properties is also important. The sublimation of ice produces a porous structure, which may improve wetting, solubility and rehydration. In mango powder, Zotarelli *et al.* reported that freeze-dried powder exhibited favourable wetting and solubility characteristics, while the reconstituted pulp retained colour characteristics comparable with the original pulp.

Similarly, research on mixed fruit pulp has shown that drying method can influence moisture, water activity, soluble solids, acidity, colour, nutritional constituents and sensory characteristics. Silva Júnior *et al.* (2022) reported comparatively favourable retention of ascorbic acid, carotenoids and phenolic compounds in freeze-dried acerola-ceriguela mixed pulp and also observed favourable sensory acceptance. (Wiley Online Library)

Recent work further supports the relevance of freeze-dried fruit powders as convenient food ingredients. Rafiquzzaman *et al.* (2025) reported that freeze-dried guava, amla and jamun juice powders retained measurable amounts of vitamin C, carotenoids and phenolic compounds and exhibited good consumer acceptance after formulation.

Relevance of Freeze-Drying to Watermelon-Orange-Mango Composite Powder

The available literature demonstrates the potential of freeze-drying for converting fruit purees and juices into stable powders while retaining important nutritional and quality characteristics. However, published studies have predominantly investigated individual fruits or fruit combinations other than watermelon, orange and mango. Studies have examined freeze-dried orange puree, mango powder and other mixed fruit pulps, but evidence concerning a watermelon-orange-mango composite puree remains limited.

Literature Gap

The reviewed literature demonstrates that watermelon, orange and mango are nutritionally valuable fruits containing different combinations of vitamins, minerals, carotenoids, phenolic compounds and other bioactive constituents. The fruits also possess complementary physicochemical and sensory characteristics that provide a reasonable basis for their use in a blended fruit product. Previous research has established that freeze-drying can be used to convert fruit juices and purees into stable powders while maintaining favourable physicochemical, nutritional and functional characteristics. Studies involving orange puree, mango powder and mixed fruit pulps have demonstrated the importance of moisture, water activity, soluble solids, acidity, colour, vitamin C, carotenoids, phenolic compounds, antioxidant activity and rehydration behaviour in evaluating dried fruit products (Silva-Espinoza *et al.*, 2021; Zotarelli *et al.*, 2022; Silva Júnior *et al.*, 2022).

However, there remains limited published evidence specifically addressing the development and freeze-drying of a watermelon-orange-mango fruit formulation and the relationship between formulation composition and the resulting physicochemical, nutritional, functional and sensory properties. Existing studies have largely focused on individual fruits or other fruit combinations. Consequently, there is a need for research that evaluates the selected fruit combination under defined formulation and freeze-drying conditions.

The present study therefore addresses this gap by investigating watermelon, orange and mango as components of a blended fruit product and evaluating the characteristics of the resulting product following freeze-drying. The study is expected to provide information on the suitability of the selected fruits for formulation, the influence of freeze-drying on product quality, and the potential of the resulting dried product as a convenient fruit-based food or beverage ingredient. The study therefore contributes evidence on the feasibility of using watermelon, orange and mango as a composite fruit matrix for freeze-dried powder production and on the extent to which freeze-drying influences its nutritional quality.

MATERIALS AND METHODS

Materials

Sources of raw materials and preliminary handling

Exactly 5 kg each of fully matured watermelon, oranges and mangoes fruits were purchased from railway local market in Makurdi, Benue State, Nigeria. The fruits were transported to the Horticulture Department of the Joseph SarwuanTarka University, Makurdi, Benue State, Nigeria for identification and confirmation of cultivar authenticity by a qualified horticulturist. They were promptly transported to the laboratory in plastic baskets and washed with tap water to remove dirt and other adhering. The cleaned fruits were placed in clean plastic baskets and kept in a household refrigerator prior to use within 12 h for product development.

Fruit Purees Production

The general flow for fruit puree production is shown in Figure 1 using the method described by Mateescu *et al.* (2022) while mango puree production flow chart is shown in in Figure 2.

Orange and Watermelon puree production are similar to that of mango as shown in flow charts in Figures 3 and 4. The purée of blanched mango pieces was obtained by crushing blanched mango in the presence of 0.2 M citric acid buffer (pH 5.2) blanched at 90 °C for 4 min in closed plastic containers. The analyses of the nutrients were carried on the purées 30 min after crushing.

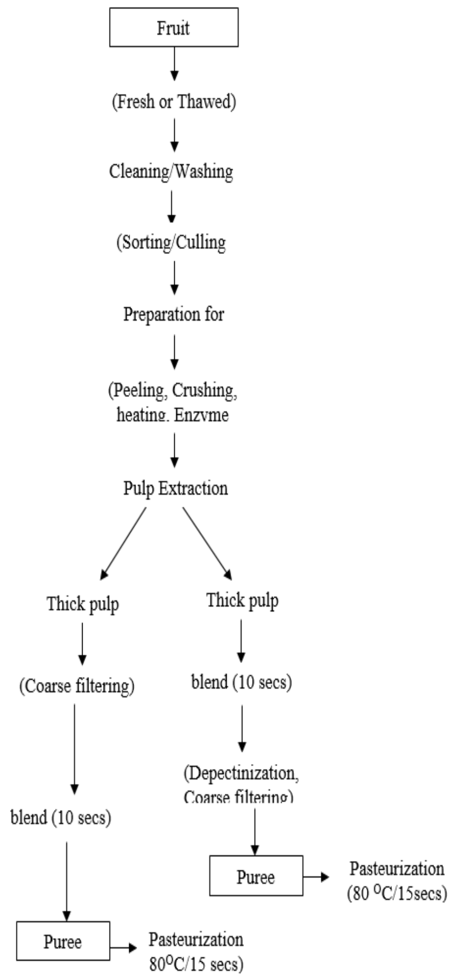

Figure 1: General flow for fruit puree production
Source: Mateescu et al. (2022)

Watermelon fruit puree production

The flow chart for the production of watermelon puree is shown in Figure 2 using the method described by Akinwande & Ojo (2023). 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 s into smooth pastes which were pasteurized at 70 oC for 15 s 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.

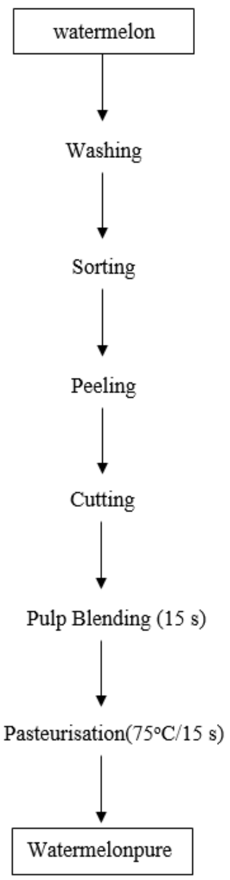

Figure 2: Flow chart for watermelon fruits puree production
Source: Akinwande & Ojo (2023)

Production of orange fruit puree

Orange fruit puree was produced as described by Sharma &Anand (2022). Essentially, as shown in Figure 3, 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 pasteurised at 70 °C for 15 seconds in 250 mL glass beakers with aluminium foil covers. The pasteurised orange puree was rapidly cooled in an ice bath and promptly stored in the refrigerator prior to use for mixed puree formulation.

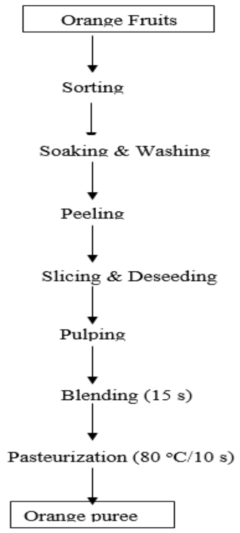

Figure 3: Flow chart for the production of orange fruit puree
Source: Sharma &Anand (2022).

The production of the mango fruits puree was by the method of Labaky *et al.* (2020)
as provided in Figure 4. The mango fruits were sorted, washed and blanched by immersion in a boiling hot water bath maintained at 98 oC for 5 min. The blanched mango fruits were then cooled in running tap water, peeled using stainless steel knives and the fleshy mesocarp sliced to obtain pieces which 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.

The purée of blanched mango pieces was obtained by crushing blanched mango in the presence of 0.2 M citric acid buffer (pH 5.2) blanched at 90 °C for 4 min in closed plastic containers. The analyses of the nutrients were carried on the purées 30 min after crush

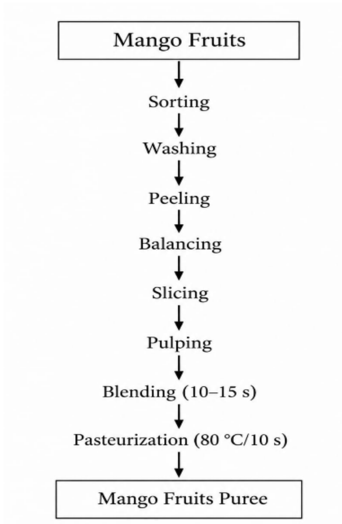

Figure 4: Flow chart for mango puree production
Source: Labaky et al. (2020)

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-digits 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

Fruit Powders Production

The most organoleptically accepted composite fruits puree containing 50 % watermelon, 30 % orange and 20 % mango was subjected to freeze drying as described in the next sections.

Freeze drying operations

Freeze drying of the composite fruits puree was as described by Varghese *et al.* (2014) 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 litres 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 250g 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 2min and the powder packaged in air tight aluminum pouches which were then stored on dry shelves in glass desiccators containing activated silica.

Proximate Analysis

Moisture Content

Moisture content was determined by oven-drying approximately 2 g of sample at 105°C to constant weight, and the percentage moisture was calculated from the weight loss (AOAC, 2012).

$$\%\ \text{Moisture} = \frac{W_1 - W_2}{W_0} \times 100$$

Where: W1 = Weight of crucible and sample before drying, W2 = Weight of crucible and sample after drying, W0 = Original Weight of Sample

Crude Protein

Protein content was determined using the Kjeldahl method involving acid digestion, distillation, and titration of liberated ammonia. Nitrogen content was multiplied by a conversion factor of 6.25 to obtain crude protein

$$\text{Protein Content } \% = \frac{\text{Titre (less blank)} \times \text{Molarity} \times 0.014 \times 100 \times 6.25}{W_0} \quad (14)$$

Where: W0 = Original weight of Sample

Crude Fibre

Samples were subjected to acid digestion, washed, dried, ashed in a muffle furnace, and crude fibre calculated from the loss in weight after ashing.

$$\text{Crude fibre } \% = \frac{W_1 - W_2}{W_0} \times 100 \quad (15)$$

Where: W1 = Weight of crucible + ash, W2 = Weight of crucible+ residue, WO =Initial weight of sample

Ash Content

Ash content was determined by incinerating the sample in a muffle furnace at 550°C until a constant weight was obtained.

$$\text{Ash content } \% = \frac{W_1 - W_2}{W_0} \times 100 \quad (16)$$

Where: W1= weight of empty ashing dish (before ignition), W2= weight of the ashing dish containing the ash (after ignition), W0 = Original weight of the sample.

Crude Fat

Fat was extracted using petroleum ether in a Soxhlet apparatus, and the extracted oil was determined gravimetrically.

$$\text{Crude fat content (\%)} = \frac{D-C}{B-A} X\ 100 (17)$$

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

Total Carbohydrate

Total carbohydrate was calculated by difference after subtracting the percentages of moisture, protein, fat, fibre and ash from 100.
100 - (% protein + % fat + % crude fibre + % moisture + % ash)

Energy Value

Energy values were estimated using the Atwater conversion factors (4 kcal/g for protein and carbohydrate; 9 kcal/g for fat).
Atwater's Conversion factor (4 x % protein + 9 x % fat + 4 x %carbohydrate) expressed in kcal/100 g

Vitamin Determination

β-Carotene (Provitamin A)

β-Carotene was extracted with methanol and hexane and quantified spectrophotometrically. Values were converted to retinol equivalents.

$$\beta - \text{carotene(mg/100g)} = \frac{\text{Absorbance (436mm) x V x D x 100 x 100}}{\text{W X Y}}$$

V = total volume of extract, D = dilution factor, W = weight of sample, Y = 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

Vitamin C

Ascorbic acid was determined by titration with 2,6-dichlorophenol indophenol (DCPIP) according to AOAC (2012).

Tocopherol (Vitamin E)

Tocopherols were extracted following saponification and quantified using reversed-phase liquid chromatography (RP-LC) and supercritical fluid chromatography (SFC).

Mineral Analysis

Minerals determination

The selected mineral elements were analyzed using the method described by Fashakin *et al.* (1991). For each of the fruit powders and the test composite puree, 0.5g sample was as held in a muffle furnace and the resultant ash mixed with 10 mL each of conc. $HNO3$ and conc. 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 was measured using Atomic Absorption Spectrophotometer (Perkin Elmer Analyst 400, USA).

Atomic Absorption Spectroscopy (AAS) Conditions

Mineral element analysis was performed using an atomic absorption spectrophotometer (Perkin-Elmer Model 503). Element-specific hollow cathode lamps were operated at a wavelength appropriate for each analyte (e.g., calcium at 422.7 nm and magnesium at 285.2 nm). The lamp current was set at 15 mA, with a slit width of 1.0 nm. Measurements were carried out using an air–acetylene flame, with a fuel flow rate of 5 L min^{-1} and an oxidant flow rate of 10 L min^{-1}. The flame temperature ranged between 2000 and 3000 °C. For analyses conducted using graphite furnace AAS, furnace temperatures were programmed between 100 and 3000 °C depending on the analytical step.

Quantification and Calibration

The concentration of mineral elements determined by AAS was calculated using Equation
C = (A / S) x DF
Where: C is the concentration of the mineral element (mg L^{-1} or µg mL^{-1}), A is the measured absorbance, S is the slope of the calibration curve (L mg^{-1} or mL μg^{-1}), and DF is the dilution factor.
For inductively coupled plasma–atomic emission spectroscopy (ICP-AES), mineral concentrations were calculated using Equation
Equation (ICP-AES): C = (I / S) x DF
Where: C represents the concentration of the mineral element (mg L^{-1} or µg mL^{-1}), I is the emission intensity, S is the slope of the calibration curve (L mg^{-1}), and DF is the dilution factor.
Calibration curves for both AAS and ICP-AES were constructed using standard solutions of known concentrations and expressed by the linear regression equation
$y = mx + b$
Where: y denotes absorbance (for AAS) or emission intensity (for ICP-AES), x is the concentration of the mineral element standard (mg L^{-1} or µg mL^{-1}), m is the slope of the calibration curve, and b is the intercept.

Determination of Amino Acids

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 & L-Arginine determination

L-Citrulline and L-Arginine extraction and quantification were as described by Jayaprakasha *et al.* (2011).Frozen puree samples 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 vortexingfor 1 min. Samples were sonicated (30 min), left at room temperature (10 min), and thencentrifuged for 20 min at 4 °C, 5700× g; (Eppendorf, Model 5417R). A 1 mL aliquot ofsupernatant was filtered (17 mm nylon syringe filter, F2513-2, Thermo Scientific) into amber HPLC vials and held at −80 °C until HPLC analysis.

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, SecurityGuard Cartridge), (Phenomenex, CA, USA) held at 25 °C and a mobile phase of 15 mM phosphoric 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.

Statistical Analysis

All the experiments were conducted in triplicate samples and the data were the mean of the three replications. All data obtained were statistically analysed 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, 1985).

RESULTS AND DISCUSSION

Results

Table 2 : Effect of Freeze-drying at a temperature of -45 oC and feed rate 400 mL/h) on the Proximate composition of the fruit (composite) puree (in mg/100g)

Parameter		Samples
	Fresh	**FDFP**
Moisture	83.45 ± 3.34	3.55 ± 3.34a
Ash	0.74 ± 0.03	2.76 ± 0.03a
Protein	0.96 ± 0.04	4.39 ± 0.04a
Crude Fiber	0.74 ± 0.03	2.89 ± 0.03a
Fat	0.50 ± 0.02	0.78 ± 0.02a
Carbohydrate	13.61 ± 0.56	85.63 ± 0.56a

Values are mean ± standard deviation (SD) of triplicate determinations. Samples with different superscripts within the same column were significantly different (p<0.05).
FDFP = Freeze-Dried Fruit Powder
All fresh and dried samples are composed of 20% mango, 30% orange, 50% watermelon freeze-dried powder

Table 3 : Effect Effect of Freeze-drying at a temperature of -45 oC and feed rate 400 mL/h) on the vitamin composition of the fruit (composite) puree (in mg/100g)

Parameter	Samples	
	Fresh	**FDFP**
Vitamin A (Retinol)	5.40 ± 3.34a	16.64 ± 0.67a
Vitamin B1 (Thiamine)	0.01 ± 0.03a	0.64 ± 0.03b
Vitamin B2 (Riboflavin)	0.02 ± 0.03b	0.52 ± 0.00a
Vitamin B3 (Niacin)	0.02 ± 0.03b	5.20 ± 0.21a
Vitamin B5 (Panthothenic acid)	0.02 ± 0.00b	0.27 ± 0.01b
Vitamin B6 (Pyridoxin)	0.01 ± 0.00a	4.15 ± 0.17b
Vitamin B9 (Folic acid)	4.10 ± 2.57b	0.53 ± 0.02b
Vitamin B12 (Cobalamin)	0.00 ± 0.00b	0.00 ± 0.00b
Vitamin C (Ascorbic acid)	5.25 ± 0.00b	23.42 ± 0.94a
Vitamin E (Tocopherol)	0.89 ± 0.04b	1.07 ± 0.04b
Vitamin K	0.01 ± 0.00b	4.20 ± 0.03a

Values are mean ± standard deviation (SD) of triplicate determinations. Samples with different superscripts within the same column were significantly different (p<0.05).
FDFP = Freeze-Dried Fruit Powder
All fresh and dried samples are composed of 20% mango, 30% orange, 50% watermelon freeze-dried powder

Table 4 : Effect of Freeze-drying at a temperature of -45 oC and feed rate 400 mL/h) on the mineral composition of the fruit (composite) puree (in mg/100g)

Parameter	Calcium	Iron	Phosphorus	Potassium	Sodium	Selenium (μg/100g)	Iodine	Magnesium
Fresh Sample	19.54± 1.213b	0.53± 0.531b	4.25± 1.131b	62.16± 1.013b	12.83± 1.243a	0.16± 1.203a	0.001± 1.240a	3.93± 1.240a
FDFP	22.59± 1.031a	0.89± 0.751a	7.22± 1.301a	92.13± 1.213a	15.82± 1.203a	0.42± 1.143a	0.005± 1.032a	6.97± 1.043a

Values are mean ± standard deviation (SD) of triplicate determinations. Samples with different superscripts within the same column were significantly different (p<0.05).
FDFP = Freeze-Dried Fruit Powder
All fresh and dried samples are composed of 20% mango, 30% orange, 50% watermelon freeze-dried powder

Table 5 : Effect of Freeze-drying at a temperature of -45 oC and feed rate 400 mL/h on the amino acid composition of the fruit (composite) puree (mg/100g)

Parameter	Samples	
	Fresh	FDFP
Isoleucine	8.79 ± 0.29	38.79 ± 3.34a
Leucine	11.72 ± 0.38	31.72 ± 0.03a
Lysine	9.28 ± 0.30	39.28 ± 0.04a
Methionine	4.80 ± 0.16	34.80 ± 0.03a
Phenylalanine	11.87 ± 0.39	40.87 ± 0.02a
Tyrosine	8.47 ± 0.28	38.47 ± 0.56a
Tryptophan	9.83 ± 0.32	39.83 ± 2.57a
Threonine	10.07 ± 0.39	40.07 ± 3.34a
Valine	10.78 ± 0.35	41.78 ± 0.03a
Histidine	9.04 ± 0.30	39.04 ± 0.04a
Arginine	8.67 ± 0.28	181.14 ± 0.03a
Alanine	7.58 ± 0.23	34.50 ± 0.02a
Aspartic acid	11.10 ± 0.36	47.40 ± 0.56a
Glutamic acid	27.27 ± 0.89	46.62 ± 2.57a
Glycine	4.14 ± 0.14	30.20 ± 3.34a
Proline	3.87 ± 0.13	23.87 ± 0.03a
Serine	3.31 ± 0.11	25.90 ± 0.04a
L-Citrulline	118.05 ± 3.86	250.01 ± 0.03a

Values are mean ± standard deviation (SD) of triplicate determinations. Samples with different superscripts within the same column were significantly (p<0.05) different.
FDFP = Freeze-Dried Fruit Powder
All fresh and dried samples are composed of 20% mango, 30% orange, 50% watermelon freeze-dried powder

Discussion

Proximate Composition

The proximate composition results showed a clear change in the composition of the watermelon–orange–mango composite puree following freeze-drying. Moisture content decreased markedly from 83.45 ± 3.34% in the fresh composite puree to 3.55 ± 3.34% in the freeze-dried fruit powder (FDFP). In contrast, the other proximate components showed increases in their measured concentrations. Ash increased from 0.74 to 2.76%, protein from 0.96 to 4.39%, crude fibre from 0.74 to 2.89%, fat from 0.50 to 0.78%, and carbohydrate from 13.61 to 85.63%.

The most pronounced trend was therefore the substantial reduction in moisture, accompanied by increases in the measured concentrations of the non-water components. Overall, the results changed from a high-moisture fresh puree to a predominantly solid, low-moisture powder.

Vitamin Composition

The vitamin results showed a predominantly increasing trend of measured concentrations following freeze-drying. Vitamin A increased from 5.40 to 16.64, vitamin B1 from 0.01 to 0.64, vitamin B2 from 0.02 to 0.52, vitamin B3 from 0.02 to 5.20, vitamin B5 from 0.02 to 0.27, vitamin B6 from 0.01 to 4.15, vitamin C from 5.25 to 23.42, vitamin E from 0.89 to 1.07, and vitamin K from 0.01 to 4.20.

However, vitamin B9 (folic acid) showed a contrasting trend, decreasing from 4.10 to 0.53 following freeze-drying. Vitamin B12 was not detected in either the fresh composite puree or the freeze-dried powder. Thus, the vitamin results did not show a uniform response to freeze-drying; most vitamins showed higher measured concentrations, whereas folic acid decreased and vitamin B12 remained undetected.

Mineral Composition

All the mineral elements determined in the study showed increases in their measured concentrations following freeze-drying. Calcium increased from 19.54 to 22.59, iron from 0.53 to 0.89, phosphorus from 4.25 to 7.22, potassium from 62.16 to 92.13, sodium from 12.83 to 15.82, selenium from 0.16 to 0.42, iodine from 0.001 to 0.005, and magnesium from 3.93 to 6.97.

The results therefore showed a consistent upward trend across all the minerals analysed. The magnitude of increase varied among the individual minerals, with potassium showing a notable increase in absolute concentration, while iodine and selenium showed relatively large proportional increases from their very low initial concentrations.

Amino-Acid Composition

The amino-acid results generally showed increased measured concentrations following freeze-drying. Isoleucine increased from 8.79 to 38.79 mg/100 g, leucine from 11.72 to 31.72 mg/100 g, lysine from 9.28 to 39.28 mg/100 g, methionine from 4.80 to 34.80 mg/100 g, phenylalanine from 11.87 to 40.87 mg/100 g, and valine from 10.78 to 41.78 mg/100 g.

L-citrulline increased from 118.05 ± 3.86 to 250.01 ± 0.03 mg/100 g, while arginine showed a particularly pronounced increase from 8.67 to 181.14 mg/100 g. Thus, the general pattern was an increase in the measured concentrations of the amino acids after freeze-drying, with L-citrulline remaining the most prominent amino acid among those reported.

Overall Nutritional Composition

Overall, freeze-drying changed the watermelon–orange–mango composite puree from a high-moisture fresh product into a low-moisture powder. The dominant pattern was a reduction in moisture from 83.45 to 3.55%, accompanied by increased measured concentrations of most proximate components, vitamins, minerals and amino acids.

The results therefore indicate that the major compositional effect of freeze-drying was dehydration and concentration of the solid fraction. However, the response was not identical for all nutrients, as demonstrated by the decrease in folic acid and the absence of detectable vitamin B12 in both samples.

Discussion of Results

Effect of Freeze-Drying on:

Proximate Composition

The pronounced reduction in moisture from 83.45 ± 3.34% in the fresh composite puree to 3.55 ± 3.34% in the freeze-dried fruit powder demonstrates the effectiveness of freeze-drying in removing water from the watermelon–orange–mango composite matrix. Freeze-drying involves freezing the food material followed by removal of ice through sublimation under reduced pressure. The resulting low moisture content is technologically important because reduction of water contributes to the production of a lighter and more stable dried product. Silva-Espinoza *et al.* (2020) similarly reported substantial water reduction during freeze-drying of orange puree and showed that freezing rate, chamber pressure and shelf temperature influenced the final moisture content of the product

The moisture value of 3.55% obtained in the present study is also consistent with the production of a low-moisture fruit powder. Rafiquzzaman *et al.* (2025), in their study of freeze-dried tropical fruit juice powders, demonstrated the feasibility of producing convenient fruit powders with desirable physicochemical and nutritional characteristics. Thus, the low moisture observed in the present study supports the effectiveness of the freeze-drying operation in converting the composite puree into a dried powder.

The increase in ash from 0.74 to 2.76% should not be interpreted as the formation of new mineral matter during freeze-drying. Rather, it is more appropriately attributed to a concentration effect resulting from the removal of water. Ash represents the inorganic residue remaining after incineration; consequently, when a large proportion of water is removed, the remaining mineral constituents constitute a larger proportion of the total mass of the dried sample. Similar increases in the measured concentration of components of dried fruit powders have been reported by Athmaselvi *et al.* (2014) and Rafiquzzaman *et al.* (2025) Protein also increased from 0.96 to 4.39%, while crude fibre increased from 0.74 to 2.89%. These increases should similarly be interpreted as changes in measured concentration rather than actual synthesis of protein or fibre. Freeze-drying does not generate new protein or dietary fibre within the fruit matrix. Instead, removal of water reduces the sample mass and consequently increases the proportion of the pre-existing solid components when expressed per 100 g of product. Dilrukshi *et al.* (2021) reported the retention of measurable protein and fibre in freeze-dried fruit and vegetable products, supporting the observation that these components remain in the food matrix after dehydration.

The increase in fat from 0.50 to 0.78% was relatively small compared with the changes observed for protein, fibre and carbohydrate. This pattern is reasonable given the generally low fat content of watermelon, orange and mango. Since freeze-drying does not synthesise lipid, the observed increase is more appropriately attributed to concentration of the lipid fraction following water removal. The relatively small magnitude of the change may also reflect the low initial concentration of fat in the composite puree and the analytical characteristics of the composite matrix.

Carbohydrate exhibited the largest numerical increase, from 13.61 to 85.63%. This substantial change is expected because carbohydrates constitute an important part of the non-water fraction of fruits, including soluble sugars and other carbohydrate components. When water is removed, these constituents represent a much larger proportion of the remaining dry material. Freeze-dried fruit powders have similarly been reported to contain high measured carbohydrate proportions following dehydration (Rafiquzzaman *et al.*, 2025).

Nevertheless, the carbohydrate value requires careful interpretation because carbohydrate in proximate analysis is commonly calculated by difference. Its calculated value is therefore influenced by the measured values of moisture, ash, protein, fat and fibre. Consequently, the increase from 13.61 to 85.63% should not be interpreted as an 85.63% increase in the absolute quantity of carbohydrate. Rather, it represents a substantially greater proportion of carbohydrate in the dehydrated product.

Therefore, comparing the two products directly on a wet-weight basis can result in apparently large increases in nutrient concentrations simply because much of the water present in the fresh puree has been removed. The results consequently demonstrate principally the concentration of dry matter following dehydration, rather than the formation of new macronutrients.

Vitamin Composition

The vitamin results demonstrated that the response to freeze-drying was nutrient-specific. Although most vitamins showed higher measured concentrations in the freeze-dried powder, folic acid decreased from 4.10 to 0.53, while vitamin B12 was not detected in either sample. This indicates that freeze-drying should not be regarded as a process that uniformly increases or preserves all vitamins.

The apparent increases in most vitamins should initially be considered in relation to the large reduction in moisture. Vitamin C deserves particular consideration because it is susceptible to degradation, especially through oxidation. Freeze-drying may provide an advantage over more thermally intensive drying processes because it avoids prolonged exposure to high temperatures. Recent comparative research on kiwifruit found that freeze-drying provided favourable vitamin C retention relative to other drying methods (DOI: 10.1016/j.jfca.2026.108880). This provides support for the possibility that the relatively high measured vitamin C concentration in the present freeze-dried composite may reflect both concentration of the remaining solids and favourable preservation under the low-temperature drying conditions.

Silva-Espinoza *et al.* (2020) further demonstrated that vitamin C preservation during freeze-drying of orange puree depends on operating conditions, including shelf temperature and pressure. This finding is relevant to the present study because orange formed part of the composite formulation. The relatively high measured vitamin C concentration after freeze-drying may therefore be associated with retention of vitamin C under the processing conditions used, together with the concentration effect produced by moisture removal.

The study by Silva-Espinoza *et al.* (2021) also demonstrated that processing conditions and protective carrier materials can influence vitamin C and other bioactive compounds during freeze-drying of orange puree. This reinforces the view that the nutritional response of the present composite cannot be attributed to freeze-drying alone without considering the particular processing conditions and food matrix.

The increases in vitamins A, E and K can likewise be considered in relation to concentration of the solid fraction and the behaviour of fat-soluble vitamins during dehydration. However, the exceptionally large increases observed for some vitamins require caution. For example, vitamin B6 increased from 0.01 to 4.15, while vitamin K increased from 0.01 to 4.20. Such large differences cannot confidently be attributed solely to concentration without confirming the analytical units, moisture basis, dilution factors and laboratory calculations. Consequently, these values should be interpreted as increased measured concentrations rather than definitive evidence of complete nutrient retention.

The reduction in folic acid from 4.10 to 0.53 provides important evidence that not all vitamins were equally stable during freeze-drying. The contrasting behaviour of folate demonstrates that nutrient stability is compound-specific and may depend on molecular structure, susceptibility to degradation, interactions with the food matrix and the particular processing conditions. Thus, freeze-drying cannot appropriately be described as a process that universally increases or preserves all vitamins.

Vitamin B12 was not detected in either sample. This result indicates that vitamin B12 was below the detection capability of the analytical method or was otherwise not measurable under the analytical conditions used. Therefore, the result should preferably be reported as "not detected" rather than interpreted as absolute absence of vitamin B12.

Overall, the vitamin findings demonstrate that freeze-drying produced higher measured concentrations for most of the vitamins analysed, but the response was not uniform. This is consistent with evidence that nutrient stability during drying depends on the particular nutrient, food matrix and processing conditions. Comparative research has shown that freeze-drying may provide favourable retention of sensitive vitamins compared with some conventional drying methods, although complete preservation of every vitamin cannot be assumed.

Mineral Composition

All the mineral elements investigated showed higher measured concentrations after freeze-drying. Calcium increased from 19.54 to 22.59, iron from 0.53 to 0.89, phosphorus from 4.25 to 7.22, potassium from 62.16 to 92.13, sodium from 12.83 to 15.82, selenium from 0.16 to 0.42, iodine from 0.001 to 0.005, and magnesium from 3.93 to 6.97.

The general increase in mineral concentration can primarily be explained by removal of water during freeze-drying. Mineral elements are non-volatile components of the fruit matrix under the conditions used for sublimation of ice. Consequently, the mineral constituents remain associated with the dried solids while water is removed. The resulting powder therefore contains a greater concentration of minerals per unit mass than the original high-moisture puree.

The increase in potassium from 62.16 to 92.13 and magnesium from 3.93 to 6.97 is consistent with this concentration effect. However, these changes should not be interpreted as evidence that freeze-drying generated potassium or magnesium. Rather, they indicate that the pre-existing mineral constituents represented a greater proportion of the final dried material. Research comparing drying methods has similarly demonstrated differences in the measured mineral composition of fruit powders. Freeze-dried strawberry powder, for example, has been reported to retain relatively high concentrations of potassium and magnesium compared with air-dried samples (DOI: 10.1007/s11694-025-03430-3).

The findings are also consistent with Zhang *et al.* (2014), who determined several mineral elements and free amino acids in freeze-dried strawberry and blueberry samples, demonstrating the persistence of mineral constituents following freeze-drying.

The magnitude of the increase differed among minerals. Calcium showed a relatively small increase from 19.54 to 22.59, whereas iodine and selenium showed proportionally larger changes because their initial concentrations were very low. The particularly large proportional increase in iodine should therefore be interpreted cautiously because the absolute concentrations remained extremely small.

Importantly, concentration should be distinguished from retention. A higher concentration of a mineral per 100 g of dried powder does not necessarily demonstrate that no amount of that mineral was lost during processing. Therefore, the mineral results indicate that freeze-drying produced a powder with higher measured concentrations of the mineral elements examined. The observed pattern is consistent with dehydration and concentration of the solid fraction and with published evidence demonstrating the persistence of mineral constituents in dried fruit matrices.

Amino-Acid Composition

The amino-acid results showed higher measured concentrations of most of the amino acids determined after freeze-drying. Isoleucine increased from 8.79 to 38.79 mg/100 g, leucine from 11.72 to 31.72 mg/100 g, lysine from 9.28 to 39.28 mg/100 g, methionine from 4.80 to 34.80 mg/100 g, phenylalanine from 11.87 to 40.87 mg/100 g, and valine from 10.78 to 41.78 mg/100 g. L-citrulline increased from 118.05 ± 3.86 to 250.01 ± 0.03 mg/100 g, while arginine increased from 8.67 to 181.14 mg/100 g.

The general increase in measured amino-acid concentrations can partly be explained by the removal of water and concentration of the non-water fraction. Amino acids are non-volatile constituents and are therefore expected to remain associated with the solid fraction during sublimation. Consequently, expressing the results per 100 g of dried powder can produce higher concentrations than those obtained for the corresponding fresh puree.

Yang *et al.* (2024), in research comparing integrated freeze-drying, conventional freeze-drying and hot-air drying of durian, reported that the types of amino acids remained largely unchanged during drying, although their concentrations were influenced by the drying process. The relatively high amino-acid contents observed in freeze-dried samples provide support for the persistence of amino acids during dehydration.

The presence of L-citrulline at a relatively high concentration is particularly relevant because watermelon is recognized as an important source of this amino acid. Ridwan *et al.* (2018) demonstrated the presence of L-citrulline and L-arginine in watermelon flesh and rind using a validated RP-HPLC method and reported considerably higher concentrations of L-citrulline than arginine in the watermelon samples examined .

In the present study, L-citrulline increased from 118.05 to 250.01 mg/100 g. The relatively high concentration may plausibly be related to the inclusion of 50% watermelon, 30% orange and 20% mango in the selected formulation. The watermelon component therefore provides a plausible explanation for the relatively prominent L-citrulline concentration observed in the composite powder. This interpretation is consistent with the established occurrence of L-citrulline in watermelon.

The most scientifically defensible interpretation for the very large increase in arginine, from 8.67 to 181.14 mg/100 g, is that, freeze-drying resulted in a higher measured concentration of arginine, rather than that the process generated or synthesised arginine. Amino-acid measurements can be affected by extraction efficiency, calibration, dilution factors, sample mass, analytical basis and chromatographic quantification. Ridwan *et al.* (2018) highlighted the importance of validated analytical procedures for accurate determination of L-citrulline and L-arginine in watermelon.

Overall, the amino-acid results demonstrate that the freeze-dried composite retained measurable amino acids, including essential amino acids and the watermelon-associated L-citrulline. The generally higher concentrations are consistent with dehydration and concentration of the solid fraction, but they should not be interpreted as evidence of biochemical synthesis of amino acids during freeze-drying.

Overall Nutritional Composition of the Composite Fruit

Taken collectively, the results demonstrate that freeze-drying substantially transformed the watermelon–orange–mango composite puree from a high-moisture fresh matrix into a low-moisture powder. The dominant change was the reduction in moisture from 83.45 to 3.55%, which was accompanied by higher measured concentrations of several proximate components, vitamins, minerals and amino acids.

The observed pattern is consistent with the technological principle of freeze-drying. By freezing the food matrix and subsequently removing ice through sublimation, the process can produce a dehydrated product without the prolonged high-temperature exposure associated with conventional thermal drying. Uwineza and Zhang (2026) reviewed the application of freeze-drying in the food industry and emphasized that nutritional and quality retention depends on factors such as freezing conditions, chamber pressure, shelf temperature, drying time and food-matrix characteristics.

The findings are also supported by Silva-Espinoza *et al.* (2020), who demonstrated that freeze-drying conditions influence water removal as well as the retention of vitamin C and β-carotene in orange puree. This is relevant to the present study because orange formed part of the composite formulation and because vitamin C showed a marked increase in measured concentration following freeze-drying.

Similarly, Wojdyło *et al.* (2018) found that freeze-dried berries generally retained vitamin C and several bioactive constituents better than hot-air-dried counterparts. These findings support the potential of freeze-drying to provide favourable retention of sensitive nutritional constituents when compared with more thermally intensive drying processes, although the response remains dependent on the specific food material and processing conditions.

The present findings also support the potential use of the freeze-dried watermelon–orange–mango powder as a convenient food ingredient. Conversion of the puree into a low-moisture powder can facilitate handling, transportation, storage and incorporation into other food products. Rafiquzzaman *et al.* (2025) similarly demonstrated the feasibility of producing freeze-dried tropical fruit juice powders with useful physicochemical, nutritional and consumer-acceptance characteristics.

Nevertheless, an important qualification applies to the interpretation of all the nutritional results. The substantial reduction in water content means that an increase in nutrient concentration per 100 g of product can occur even if some absolute quantity of that nutrient has been lost during processing. This distinction is particularly important for the unusually large increases observed for some vitamins and amino acids. Therefore, the present results demonstrate increased measured concentrations following freeze-drying, but additional dry-matter-basis comparisons or mass-balance studies would be necessary to establish the absolute retention of individual nutrients.

In conclusion, the nutritional results indicate that freeze-drying successfully achieved its primary technological purpose of producing a substantially dehydrated watermelon–orange–mango powder while retaining measurable nutritional constituents. The process therefore shows promise as a preservation and value-addition technology for the selected composite fruit formulation. However, the nutritional changes should principally be interpreted in terms of dehydration, concentration and nutrient-specific stability, rather than as the generation of additional nutrients during freeze-drying.

CONCLUSION

The study demonstrated the feasibility of producing a watermelon–orange–mango composite fruit powder by freeze-drying. The 50:30:20 watermelon–orange–mango formulation was selected as the most acceptable based on sensory evaluation. Freeze-drying reduced the moisture content of the composite puree from 83.45% to 3.55%, converting the highly perishable puree into a stable and convenient powder. The reduction in moisture resulted in apparent increases in the concentrations of ash, protein, crude fibre, fat and carbohydrate.

The freeze-dried powder also showed increased measured concentrations of most vitamins and minerals, although folic acid decreased and vitamin B12 was not detected. Several essential and non-essential amino acids were present, with L-citrulline increasing from 118.05 to 250.01 mg/100 g on the reported basis.

Overall, freeze-drying proved effective for producing a nutrient-rich, convenient watermelon–orange–mango fruit powder with potential applications in beverages and other food products.

Recommendations

Further studies should focus on storage stability, water activity, rehydration, solubility, hygroscopicity, colour, drying optimization, microbiological quality, and energy/economic efficiency. Nutrient results should also be verified against original laboratory reports and expressed on both wet- and dry-matter bases to distinguish concentration effects from actual nutrient retention or loss.

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