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Research article

Comparative evaluation of nutritional composition, phytochemicals and sensorial attributes of lyophilized vs conventionally dried *Grewia asiatica* fruit pulp powder

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Abstract: *Grewia asiatica*, commonly known as phalsa, is a widely cultivated fruit in Pakistan, valued for its nutritional and medicinal benefits. Being a perishable fruit with limited shelf life and rapid spoilage, a preservation approach is needed to extend fruit shelf life and product development. In this context, we aimed to extend the shelf life of *G. asiatica* fruit pulp using conventional and lyophilization drying and to evaluate their impact on nutritional composition, phytochemicals attributes, sensory evaluation and product shelf life for fruit pulp powder stored at ambient conditions for 3 months (0, 45th and 90th day). For the study, ripe *G. asiatica* fruit sourced from local farms were subjected to cleaning followed by drying using conventional and lyophilization processes. The resulting fruit powder was packed in sealed foil bags and stored at room temperature for 3 months, subjected to nutritional properties, phytochemicals, antioxidant capacity, and sensory evaluation on the 0, 45th, and 90th storage days. The results showed that both techniques increased the shelf life of powder. However, lyophilization resulted in better retention of vitamin C, antioxidant activity, and better free radical scavenging activity in fruit powder than conventional drying. Color parameters and sensory evaluation were also affected by drying and storage conditions as an advancement of storage resulted in decreased consumer acceptability. These findings demonstrate that lyophilization effectively preserves nutritional and phytochemical qualities of *G. asiatica*

powder, making it a viable preservation approach for prolonging fruit shelf life while maintaining its health promoting compounds and functional properties. The lyophilized *G. asiatica* fruit pulp powder may have potential use in the food industry as an additive in ready-to-use products to enhance nutritional attributes, color, and better consumer acceptability for dried powders.

Keywords: drying; Lyophilization; *G. asiatica* powder; storage stability; nutritional loss shelf life

1. Introduction

Grewia asiatica, commonly known as phalsa, is an indigenous berry of South Asia, renowned for its rich nutritional profile and higher phytochemical properties. *G. asiatica* is locally admired for its tangy flavor, consumed fresh and is used in multiple food products. Traditionally consumed for its taste and health benefits, it is a significant source of vitamins and antioxidants and has a high fiber content and bioactive compounds that have to therapeutic potential [1]. Being a perishable food crop, *G. asiatica* also contains an appreciable amount of essential minerals such as potassium, phosphorus, sodium, calcium, cobalt, chromium, nickel, iron, and zinc. Despite all the promising benefits of the berry, its cultivation is scarce and it has a short shelf life. Moreover, the preservation of these nutritional and phytochemical attributes during storage poses challenges, especially when aiming to maintain berry quality over an extended storage period. This very reason causes hindrances in reaching distant markets. The berry should be preserved in a way that health benefits can be attained all year round, along with the preservation of the nutrients in the berry and allied berry-based products [2].

The *G. asiatica* berry pulp powder can be used effectively in food and nutraceutical industries, due to its rich nutritional profile and attributes. The berry pulp powder has potential as a natural colorant due to its deep purple color and is a flavor enhancer in many common food products like gummies, smoothies, yoghurt, baked goods, candies, and confectioneries, providing a vibrant hue and tangy flavor. It can also be used as an active functional ingredient in various products such as dietary supplements, health drinks, vitamin waters, and energy bars to improve nutrient content. Nowadays, consumers are also inclined to choose natural and healthy food products. A total of 50% of berries are processed and preserved worldwide [3]. According to (MNFSR, 2020), Pakistan produces 4308 tons of phalsa and its production is increasing yearly, and post-harvest losses of berries are recorded as 50% [4]. This berry has a concise shelf life of about one to two days, so chances of post-harvest losses are greater among other fruits [5].

Food preservation involves processes and techniques to prevent food spoilage, extend shelf life, and maintain nutritional value, texture, and product flavor. Drying, one of the oldest food preservation techniques, involves removing moisture from food, which can inhibit the growth of bacteria, yeast, and molds that are responsible for food spoilage [6]. The major drying method for food includes sun drying, air drying, oven drying, dehydration, and freeze drying that has effect on product attributes with special reference to nutrient content. Freeze drying, also called lyophilization, removes moisture by freezing and then reducing the surrounding pressure to enable frozen water to sublimate directly from solid to gas in different foods. This method is highly effective at preserving nutritional content of food. In lyophilization, food is the first frozen solid, which preserves its cellular structure and nutritional content followed by placing frozen food in a vacuum chamber where pressure is lowered, and the temperature is slightly increased where ice converts directly into

water vapor without passing through the liquid phase. Afterwards, desorption takes place, where the product temperature is raised slightly to remove any remaining bound moisture molecules [7, 8].

Lyophilization is considered one of the best drying methods to preserve bioactive components of fruits and has commercial application for the development of healthy products. Lyophilized berry powders are particularly considered beneficial for enhancing nutritional value of processed food products [9]. The lyophilization process involves freezing berries and removing water content through sublimation. This method helps in preserving heat-sensitive bioactive components, vitamins, phenolics, and flavonoids, which might degrade during other drying processes. Furthermore, the health benefits of berries have been studied extensively. According to a study by the researchers in [10] on the relative distribution of phenolic component classes in *G. asiatica*, flavanols made up most of the group (44.3%). Of the total phenolics, anthocyanins made up 28%. Flavanols, flavones, and anthocyanins are all abundant in *G. asiatica* [11]. Considering the scenario, we prepare *G. asiatica* powder using conventional dehydration and freeze-drying methods for application in different food products. Additionally, we focus on nutritional and phytochemical stability of pulp powder during storage at ambient temperature. Our results provide valuable insight into the storage stability and consumer acceptability of *G. asiatica* pulp powder for drying techniques.

2. Material and methods

The present study conducted to observe nutritional changes, antioxidant and physiochemical stability of lyophilized and conventionally dried *G. asiatica* pulp powder during storage at ambient temperature for three months.

2.1. Chemicals, reagents, and experimental material procurement

The chemicals and reagents required for experiments were procured from local reputable suppliers. Sodium acetate was sourced from AUS Chem Pvt Ltd. while 2,2-diphenyl-1-picrylhydrazyl (DPPH) obtained from Sigma-Aldrich. Methyl red was supplied by Hangzhou Meite Chemical Co., Ltd, and cyanidin 3-glucoside, along with Folin-Ciocalteu's phenol reagent, was purchased from Thermo Fisher Scientific, USA

2.2. Preparation of conventional and lyophilized G. asiatica powder

For the research study, fresh *G. asiatica* berries were procured from Nagana fruit farm, Multan, Pakistan. Before harvest, the berries were carefully examined for maturation, color consistency, conformity to physical contaminants, and size uniformity. The botanical confirmation of *G. asiatica* was done by the Institute of Botany, Bahauddin Zakariya University Multan. Thereafter, the collected fruits were cleaned and properly rinsed with tap water to remove adherent particles. Then, the *G. asiatica* berries were cleaned, washed, and dried on open trays to remove any remaining water droplets. The pulp and seed were separated using a stainless-steel knife, and mixture was frozen at −18 °C in a freezer (HDF-325INV-Haier). The first group of *G. asiatica* berry pulp was air dried at 60 °C (SLN 75, POL-EKO® A. Polok-Kowalska sp.k, Poland) until a constant weight was obtained. In the 2nd group, frozen *G. asiatica* berry pulp was subjected to lyophilization using CHRIS Alpha 1-4LD lyophilizer [12]. The lyophilization process conducted at −55 °C for 36 hrs ensured moisture

removal while maintaining integrity of bioactive compounds. Post-lyophilization, dried *G. asiatica* powder was collected and sealed in aluminum foil bags to protect from environmental contamination. Furthermore, sealed bags were stored at room temperature in a light-free environment to prevent photodegradation and maintain quality of dried powder followed by being subjected to analysis. To evaluate the effects of the dehydration process and storage, the *G. asiatica* pulp powder was subjected to nutritional and phytochemical analysis. The lyophilized berry pulp powder was analyzed immediately post-lyophilization to assess the immediate impact of the drying process, and further analyses were performed after 45 days and at termination of storage to determine any changes in nutritional, physiochemical, phytochemical, and sensorial attributes of the berry powder.

2.3. Nutritional analysis of conventional and lyophilized G. asiatica powder

The conventional and lyophilized *G. asiatica* berry powder were analyzed for nutritional composition (moisture, ash, crude fiber, crude protein, crude fat, and nitrogen-free extract) using [13]. The samples were ran in triplicate at different storage intervals *i.e.* 0, 45th, and 90th day of lyophilization storage. For analysis, lyophilized and dried fruit pulp were ground using an electric grinder to make it a fine powder at uniformity.

2.3.1. Moisture analysis

The moisture content of dried samples plays an important role in food storage and quality. The samples were assessed for moisture content using the methods of association of official analytical chemists [13]. For moisture analysis, an empty dish with a lid was placed in an oven for 3 hours at 105 °C. After thorough heating, the dish was placed in a desiccator to cool in a moisture-free environment. A total of 10 grams of sample were added to the dish and again placed in the oven for 3 hours at 105 °C. After cooling in a desiccator, the dish was weighed, and moisture (wet basis) content was calculated using Formula 1:

$$\text{Moisture (\%)} = \frac{W1-W2}{W1} \times 100 \quad (1)$$

W_1 = initial weight (g) of the sample; W_2 = weight (g) of the sample after drying.

2.3.2. Crude proteins analysis

The protein contents of *G. asiatica* berry pulp powder were determined using the Kjeldahl method, following the guidelines of [13]. For the experiment, 2 g of sample was taken in an aliquot and placed in a Kjeldahl digestion flask followed by adding 40 mL concentrated sulfuric acid mixed with 2 g of salicylic acid and mixed thoroughly. Then, 2 g of digestion mixture was added, agitated, and left standing for 5 min. This mixture was heated gently during the process of digestion to avoid excessive frothing. Next, the sample was distilled with 200 mL of water at 25 °C. Ammonia was released during distillation and trapped in a receiving flask with a known volume of sulfuric acid. Titration was done in the presence of methyl red. The nitrogen content was measured using Formula 2:

$$\text{Nitrogen (\%)} = \frac{(V1-V2)\times N\times 1.4007}{W} \quad (2)$$

The nitrogen content then converted to protein content using a conversion factor of 6.25, which is standard for most food products.

2.3.3. Ash analysis

The ash content of fresh and lyophilized *G. asiatica* fruit was determined using the protocol in [13]. For analysis, 3 g of sample was taken in a dry clean weighed porcelain crucible. The porcelain crucible was then placed in a muffle furnace. The sample was exposed to 550 °C until the ash grey color was obtained, which was an indicator of organic matter combustion. Thereafter, the crucible was placed in a desiccator for cooling. The crucible was weighed again, and the ash was calculated using Formula 3:

$$\text{ASH (\%)} = \frac{W2-W1}{W0} \times 100 \quad (3)$$

W_1 = weight (g) of the empty crucible; W_2 = weight (g) of the ash in crucible, W_0 = weight(g) of sample.

2.3.4. Crude fat content

The crude lipid of the dried berry pulp powder was determined using the Soxhlet extraction technique by following the procedure of (AOAC, 2000). For the analysis, 3 g of fruit pulp powder was placed in a thimble, which was then placed in an extractor. N hexane was used as a solvent extractor, and solvent was condensed in condenser. The process was repeated for 6 hours until the siphon tube was clear. The solvent was removed through a rotary evaporator and dried in an oven at 105 °C for 30 min. The flask was then placed in a desiccator. The crude fat was calculated using Formula 4:

$$\text{Crude Fat (\%)} = \frac{W-W2}{W0} \times 100 \quad (4)$$

W_2 is the weight of the flask with residual fat, W_1 represents the weight of the empty flask, and W_0 is the weight of the sample.

2.3.5. Crude fiber analysis

For the fiber determination, the sample was diluted using 1.25% sulfuric acid in the conical flask and then placed on a heating mantle for boiling for half an hour with continuous stirring of the solution. The contents were then filtered and rinsed with distilled water. The resultant filtrate was diluted with 1.25% sodium hydroxide and brought to boiling for 30 min with continuous stirring. The resultant solution was filtered again, and the residue was weighed to calculate the fiber content.

2.3.6. Nitrogen-free extract analysis

The nitrogen-free extract of the *G. asiatica* berry pulp powder was calculated by adding percentages of all nutritional components and subtracting by 100.

$$\text{NFE} = 100 - (\%\text{Moisture} + \%\text{Crude Protein} + \%\text{Crude Fiber} + \%\text{Ash}) \quad (5)$$

2.3.7. Mineral analysis

The minerals (sodium, potassium, magnesium, zinc, iron, phosphorus, and calcium) content of conventional vs freeze dried *G. asiatica* fruit pulp samples were determined using the procedure in [13]. For mineral determination, 0.5 g sample was digested using HNO_3 and perchloric acid at a ratio of 7:3 using a hot plate until a colorless solution of about 1–2 mL was obtained. The digested sample was filtered using Whatman filter paper. Thereafter, the digested sample was diluted to 50 mL for mineral analysis. Sodium, potassium, and calcium were calculated on a Flame Photometer-410 (Sherwood Scientific Ltd., Cambridge)while magnesium, manganese, phosphorus, and zinc were determined through an Atomic Absorption Spectrophotometer (HITACHI Z-8230 polarised Zeeman) following the procedure outlined in [14].

2.3.8. Determination of Vitamin C

The vitamin C level of dried *G. asiatica* **s**amples was determined using the iodine redox titration method as per the guidelines in [15]. The reagents used were potassium iodide, potassium iodine, and diluted HCL. The starch solution was used as an indicator. When iodine solution was added to the vitamin C solution, the vitamin c was oxidized to deoxy-ascorbic acid. Titration with starch solution was done until a blackish blue color was achieved.

2.4. Antioxidant activity determination:

The antioxidant activity of *G. asiatica* dried berry pulp powder was assessed using total phenolics, total anthocyanins, and free radical scavenging activity analysis.

2.4.1. Total phenolics contents

The total phenolic contents of dried *G. asiatica* powder were determined as described in [16]. For determination of TPC, 1 mL extract was subjected to conventional vs lyophilized powder placed in a test tube. Then, 10 mL of deionized water and 1.0 mL of Folin-Ciocalteu phenol reagent were added to the test tubes and shaken vigorously for thorough mixing. The same procedure was repeated for preparation of gallic acid standard solution (1 mL). A total of 20% sodium carbonate (2 mL) was added to the mixture, extracted for 5 min, and the solution was stored in a dark place for 1 hour. After the hour, the extract was evaluated for absorbance using a spectrophotometer at 750 nm. Total phenolic contents were measured as GAE per 100 g of initial weight sample.

2.4.2. Total anthocyanins

The total anthocyanin content was analyzed through a pH differential method by following the procedure in [17]. For analysis, pH 1 and 4.5 buffers were prepared, and anthocyanin extracts were diluted in both buffers separately. Thereafter, the absorbance was measured at 520 nm and 700 nm for buffers 1 and 2, respectively. Afterwards, the total anthocyanin content was calculated using the Formula 6.

$$\text{Total anthocyanin content(mg/L)} = \frac{AXMWXDX1000}{EXL} \quad (6)$$

2.4.3. 2,2-diphenyl-1-picrylhydrazyl assay

The radical scavenging activity of dried *G. asiatica* berry pulp powder was assessed using the DPPH method as described in [18]. For the analysis, various concentrations of dried *G. asiatica* pulp powder ranging from 50–100 µL were made and transferred to labeled test tubes. Thereafter, the methanol was used to adjust the volume of the test tubes to 100 µL. Then, 5 mL of DPPH reagent of 0.1 mM concentration was poured into the test tubes and, the tubes were vortexed. The test tubes were then left standing for 20 min at 27 ℃. In the control sample, methanol was used in place of extract and the shift in absorbance was read at 517 nm. The formula for Free radical scavenging activity is:

$$\text{Antioxidant activity (\%)} = \frac{Blank\ sample\ reading - actual\ sample\ reading}{blank\ sample\ reading} \times 100 \quad (7)$$

2.4.4. Ferric reducing antioxidant power assay

The ferric reducing antioxidant power (FRAP) assay is a colorimetric assay used to determine ferric iron reduction using FRAP reagents [19]. For the analysis, lyophilized *G. asiatica*, FRAP reagents were mixed in the ratio of 1:1:10 as ferric chloride, sodium acetate buffer, and 2,4,6-tripyridyl-s-triazine (TPTZ). Thereafter, the powder was mixed in 0.02 mL prepared FRAP reagent and was left standing for 30 min in a light-free space to attain optimum absorbance of tested samples using a spectrophotometer at 593 nm followed by calculations.

2.5. Physiochemical properties of G. asiatica powder

2.5.1. Color measurement

The color of *G. asiatica* berry pulp powder was subjected to conventional vs lyophilization measured at different storage intervals using a lab calorimeter by following the procedure in [20]. The color parameters, including lightness, redness, and yellowness described as L*, a*, and b*, were assessed.

2.5.2. Brix analysis using refractometer

The brix of the *G. asiatica* berry pulp powder was determined using a refractometer (Abbe Refractometer - AR2008) by following the procedure in [21].

2.6. Sensory evaluation

To check quality and consumer acceptability of the *G. asiatica* berry pulp powder, sensory profiling was done using the procedure in [22]. The sensory panel comprised 12 members aged between 20 and 35 years, and panelists were asked to evaluate the overall quality of the powders on a 9-point hedonic scale for scoring, where 1 represented like extremely, 5 represented neither like nor dislike, and 9 stood for an extremely dislike for attributes, including color, taste, aroma, consistency, and overall acceptability of dried powder samples.

2.7. Statistical analysis

All experiments were performed thrice, and the resultant data was subjected to statistical analysis through the statistical package for social sciences (SPSS, 20). The results were represented as means and standard deviations. To test the significant difference, analysis of variance (ANOVA) was used, and the Tucky post hoc test used for comparing the means of different parameters by following the procedure in [23].

3. Results

The lyophilized vs conventional dried *G. asiatica* powders were assessed for nutritional stability, physical properties, and sensory evaluation to establish a powder that can be used as a functional food product for its health benefits throughout the year.

3.1. Effect of storage on lyophilized G. asiatica nutrition content

3.1.1. Proximate analysis

The results regarding proximate analysis of *G. asiatica* pulp powder that were prepared by drying (conventional vs lyophilized) and the storage presented (Table 1) depicted significant differences for moisture and ash after storage termination (90^{th} day). Initially, the moisture for conventionally air-dried (CAD) samples was 5.11% ± 0.61, which increased slightly over time, reaching 5.81% ± 0.66 after the 90^{th} day. Contrary to this, lyophilized *G. asiatica* pulp (LPP) powder had a lower initial moisture at 3.38% ± 0.22 with a slight increase over the storage duration (90 days) to a maximum extent of 3.82% ± 0.28. The ash content in CAD reduced from 1.27%±0.03 to 1.15% ± 0.5 over storage time, whereas lyophilized phalsa berry powder maintained a stable ash at 1.17%± 0.03. However, a slight variation was observed at 1.15% ± 0.02 after the 90^{th} day of study. The protein and fat content of powder prepared by the conventional vs lyophilized processes showed non-significant difference. The crude fat in CAD was reduced slightly while LPP samples remained stable from 0.45% ± 0.12 to 0.41% ± 0.08. The crude fiber content showed less degradation in lyophilized berry powder. The nitrogen-free extract (NFE) varied from 72.46 ± 0.30 to 74.46 ± 0.81 in the CAD sample, while lyophilized powder varied from 72.88 ± 0.78 to 73.84 ± 1.05. Overall, both techniques performed better in terms of drying but lyophilization performed better for retaining nutrient quality during storage of *G. asiatica* powder, reporting higher initial retention and slow degradation of nutrients like protein and fiber, compared to conventional drying.

3.1.2. Mineral analysis

The mineral (phosphorus, potassium, calcium, zinc, magnesium, and iron) content of conventional and lyophilized dried *G. asiatica* berry pulp powder is presented in Table 2, showing significant differences for zinc and potassium and non-significant differences for the other minerals. In conventionally dried phalsa berries powder, a significant change in P, K, and Mg after the 90^{th} day of storage was observed, while after lyophilization, all mineral content showed prominent stability over 90 days of storage. Overall, mineral retention in LPP appeared to be slightly better compared to CD

G. asiatica berries, suggesting that the freeze-drying technique is more effective for preserving mineral composition of *G. asiatica* berries. In CD, phosphorus content slightly decreased from 24.20 ± 0.1 to 23.27 ± 0.57 mg/100 g after the 90th storage day. Similarly, the potassium content decreased from 372.27 ± 0.76 to 346.11 ± 6.05 mg/100 g over the same period. The calcium content decreased from 134.034 ± 2.66 to 129.70 ± 1.45 mg/100 g, while zinc reduced from 1.22 ± 0.11 to 1.02 ± 0.03mg/100g. Magnesium decreased from 0.96 ± 0.07 to 0.92 ± 0.51, and iron decreased from 1.80 ± 0.05 to 1.71 ± 0.03. In case of lyophilized *G. asiatica* berry pulp powder, phosphorus remained stable from 24.70 ± 0.35 at the beginning of storage and decreased to 24.07 ± 0.47 at storage termination. Similarly, the potassium level showed a minor decrease from 373.10 ± 2.39 to 364.33 ± 10.97, whereas the calcium content in lyophilized *G. asiatica* powder was initially at 136.83 ± 0.35 and decreased to 135.07 ± 0.85 mg/100 g at storage termination. The zinc content decreased from 1.44 ± 0.06 to 1.34 ± 0.09, and magnesium remained relatively stable during storage from 1.09 ± 0.50 to1.07 ± 0.51, while the iron level in the powder showed a change from 1.84 ± 0.46 to 1.79 ± 0.05.

Table 1. Proximate composition of conventional and lyophilized dried *G. asiatica* berry pulp powder at different storage intervals.

Dehydration method	Storage days	Moisture	Ash	Crude protein	Crude fats	Crude fiber	Nitrogen free extract
Conventional drying	0	$5.11^{a} \pm 0.61$	$1.27^{ns} \pm 0.03$	$1.62^{ns} \pm 0.08$	$0.44^{ns} \pm 0.08$	$19.10^{b} \pm 0.89$	$72.46^{a} \pm 0.30$
	45	$5.37^{a} \pm 0.55$	$1.17^{ns} \pm 0.19$	$1.58^{ns} \pm 0.11$	$0.41^{ns} \pm 0.08$	$18.00^{b} \pm 0.66$	$73.47^{a,b} \pm 1.49$
	90	$5.81^{a} \pm 0.66$	$1.15^{ns} \pm 0.51$	$1.52^{ns} \pm 0.13$	$0.39^{ns} \pm 0.08$	$16.67^{c} \pm 0.15$	$74.46^{b} \pm 0.81$
Lyophilization drying	0	$3.38^{b} \pm 0.22$	$1.17^{ns} \pm 0.03$	$1.95^{ns} \pm 0.21$	$0.45^{ns} \pm 0.12$	$20.17^{a} \pm 0.89$	$72.88^{a} \pm 0.78$
	45	$3.46^{b} \pm 0.33$	$1.16^{ns} \pm 0.03$	$1.92^{ns} \pm 0.21$	$0.43^{ns} \pm 0.07$	$19.20^{b} \pm 0.66$	$73.83^{a,b} \pm 0.68$
	90	$3.82^{b} \pm 0.28$	$1.15^{ns} \pm 0.02$	$1.88^{ns} \pm 0.19$	$0.41^{ns} \pm 0.08$	$18.90^{b} \pm 0.72$	$73.84^{a,b} \pm 1.05$

Data are "Means ± Standard deviation". Superscript letters (^{a,b}) show the statistical differences at ($p < 0.05$) within the columns and "ns" represents non-significant differences within column for that parameter, and the start day of the experiment (0 day) refers to the samples after immediate treatment of the samples with drying techniques.

Table 2. Mineral (mg/100g) composition of conventional and lyophilized *G. asiatica* berry pulp powder at storage intervals (0, 45th, and 90th).

Drying method	Storage days	Phosphorus	Potassium	Calcium	Zinc	Magnesium	Iron
Conventional drying	0	$24.20^{ns} \pm 0.1$	$372.27^{a} \pm 0.76$	$134.034^{ns} \pm 2.66$	$1.22^{b} \pm 0.11$	$0.96^{ns} \pm 0.07$	$1.80^{ns} \pm 0.05$
	45	$23.57^{ns} \pm 0.5$	$357.17^{b} \pm 6.65$	$131.41^{ns} \pm 1.06$	$1.06^{c} \pm 0.04$	$0.93^{ns} \pm 0.58$	$1.76^{ns} \pm 0.06$
	90	$23.27^{ns} \pm 0.57$	$346.11^{c} \pm 6.05$	$129.70^{ns} \pm 1.45$	$1.02^{c} \pm 0.03$	$0.92^{ns} \pm 0.51$	$1.71^{ns} \pm 0.03$
Lyophilization	0	$24.70^{ns} \pm 0.35$	$373.10^{a} \pm 2.39$	$136.83^{ns} \pm 0.35$	$1.44^{a} \pm 0.06$	$1.09^{ns} \pm 0.50$	$1.84^{ns} \pm 0.46$
Drying	45	$24.37^{ns} \pm 0.46$	$370.27^{a} \pm 4.65$	$136.01^{ns} \pm 0.07$	$1.39^{a} \pm 0.06$	$1.08^{ns} \pm 0.01$	$1.82^{ns} \pm 0.05$
	90	$24.07^{ns} \pm 0.47$	$364.33^{b} \pm 10.97$	$135.07^{ns} \pm 0.85$	$1.34^{a} \pm 0.09$	$1.07^{ns} \pm 0.51$	$1.79^{ns} \pm 0.05$

Data are "Means ± Standard deviation". Superscript letters (^{a,b}) show the statistical differences at ($p < 0.05$) within columns and "ns" represents non-significant differences within a column for that parameter, whereas the start day of the experiment (0 day) refers to the samples after immediate treatment of the samples with drying techniques.

3.2. Vitamin C level, total anthocyanin, and total phenolics of lyophilized and conventional dried G. asiatica pulp powder

The vitamin C, total anthocyanin, and total phenolics of CAD and LP *G. asiatica* berry pulp powder showed significant differences due to treatments and storage (Table 3). At 0 days, the lyophilized berry pulp samples had a greater vitamin C content (5.22 ± 0.14 mg/100 g) than traditionally dried samples (3.66 ± 0.28 mg/100 g). Both drying processes resulted in a decline of vitamin C during storage especially after the 90th day; however, lyophilized berry samples maintained higher levels of vitamin C (4.10 ± 0.18 mg/100 g) than traditionally dried samples (2.45 ± 0.42 mg/100 g). This implies that lyophilization drying minimizes nutritional breakdown owing to drying at lower temperatures and better maintains vitamin C. During storage, the anthocyanins are susceptible to deterioration. At the beginning of the study, the anthocyanin concentration of lyophilized samples (4.86 ± 0.11 mg/100 g) was greater than that of traditionally dried samples (4.11 ± 0.10 mg/100 g). Both techniques demonstrated a decrease in anthocyanin content over 90 days. However, compared to traditionally dried samples (3.37 ± 0.18 mg/100 g), lyophilized samples preserved greater amounts (4.18 ± 0.13 mg/100 g). This suggests that lyophilization preserves anthocyanins well and that antioxidant capabilities of phenolic compounds are essentially maintained. LP had a greater total phenolic content (332.06 ± 8.54 mg GAE/100 g) than CAD (313.67 ± 10.26 mg GAE/100 g) right after dehydration. The phenolic content decreased after 90 days with both techniques; however, the lyophilized samples maintained a higher phenolic content (289.33 ± 4.51 mg GAE/100 g) compared to the traditionally dried samples (261.01 ± 14.04 mg GAE/100 g). This showed that lyophilization protects phenolic chemicals better, perhaps because drying causes less oxidation and heat breakdown.

Table 3. Vitamin C levels, total anthocyanin, and total phenolics content of lyophilized and conventional dried G. *asiatica* berry pulp powder at different storage intervals.

Drying method	storage days	Vitamin C	Total anthocyanin	Total phenolics
Conventional drying	0	$3.66^{c} \pm 0.28$	$4.11^{c} \pm 0.10$	$313.67^{c} \pm 10.26$
	45	$2.80^{d} \pm 0.65$	$3.78^{d} \pm 0.07$	$288.06^{d} \pm 19.97$
	90	$2.45^{e} \pm 0.42$	$3.37^{e} \pm 0.18$	$261.01^{e} \pm 14.04$
Lyophilization	0	$5.22^{a} \pm 0.14$	$4.86^{a} \pm 0.11$	$332.06^{a} \pm 8.54$
Drying	45	$4.17^{b} \pm 0.16$	$4.47^{b} \pm 0.18$	$304.33^{b} \pm 7.57$
	90	$4.10^{b} \pm 0.18$	$4.18^{c} \pm 0.13$	$289.33^{d} \pm 4.51$

Data are "Means ± Standard deviation". Superscript letters (^{a,b}) show the statistical differences at ($p < 0.05$) within a column, whereas the start day of the experiment (0 day) refers to the samples after immediate treatment of the samples with drying techniques.

3.3. Free radical scavenging activity and DPPH assay of phalsa berry pulp powder

The results of antioxidant activity of *G. asiatica* berry pulp powder subjected to drying techniques, including conventional and lyophilization measured over time (0, 45th and 90th day), are reported (Figures 1 and 2), showing significant differences due to treatments and storage. Antioxidant activity is quantified using assays, including FRAP (Ferric Reducing Antioxidant Power) and DPPH (2,2-diphenyl-1-picrylhydrazyl). The FRAP levels were significantly higher in lyophilized phalsa pulp (29.78 mM Fe/g) compared to conventional air after drying (16.43 mM Fe/g).

DPPH showed higher radical scavenging activity (249.38) than conventional drying (196.11). A decline in antioxidant activity was observed in both methods over 90 days; however, lyophilized samples consistently retained higher FRAP values (26.25 mM Fe/g at 45th day and 22.81 mM Fe/g at the 90th day compared to conventionally dried samples (13.36 and 9.98-mM Fe/g at 45 and 90 days, respectively). Lyophilized samples maintained significantly higher DPPH values (229.97 at 90 days) than conventionally dried samples (167.04 at 90th day).

3.4. Color attributes of powder

The color of dried *G. asiatica* berry powder can significantly influence consumer behavior and preferences [24]. The color parameters of *G. asiatica* were evaluated in terms of their luminosity (L*), redness (a*), yellowness (b*), Hue (H), and chrome values. In case of CAD, the luminosity (L*) value increased slightly over the storage period, causing a noticeable change in natural color of *G. asiatica* berry berry, while LPP retained more lightness from 33.77 ± 0.59 to 34.43 ± 0.47 when compared with CAD, which showed L* from 31.20 ± 0.77 to 32.43 ± 0.47 after 90 days. The redness (a*) of lyophilized *G. asiatica* pulp powder remained relatively stable, starting at 40.60 ± 0.49 and slightly decreasing to 39.73 ± 0.12, while in the CAD method, the a* value declined substantially from 29.30 ± 0.57 to 27.84 ± 0.13. Similarly, yellowness (b*) showed minimal fluctuation in LPP with minimal color degradation over the storage duration. The chroma (C) values decreased from 29.88 ± 0.56 to 28.65 ± 0.13 in CAD *G. asiatica* berry pulp powder, which was slightly higher than LPP. Similarly, the Hue (H) value in LPP showed a relatively stable red hue, retaining the maximum color properties compared to LPP (Table 4).

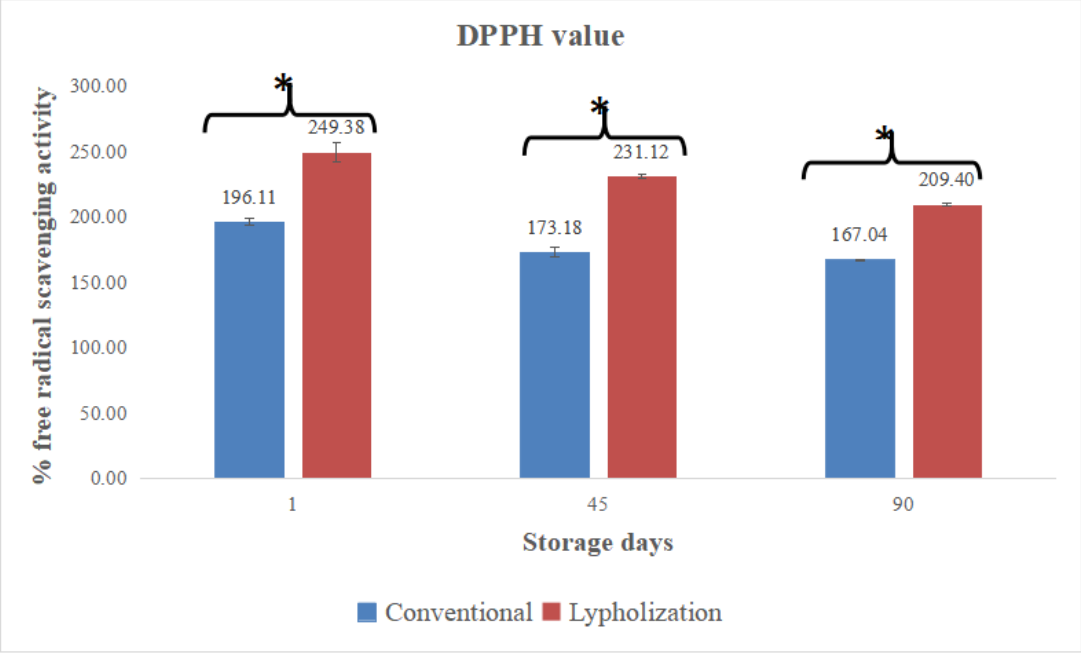

Figure 1. 2,2-Diphenyl-1-picrylhydrazyl (DPPH) of *G. asiatica* pulp powder subjected to conventional and lyophilized drying at different storage intervals. * indicates statistically significant differences among parameters across treatments and storage intervals.

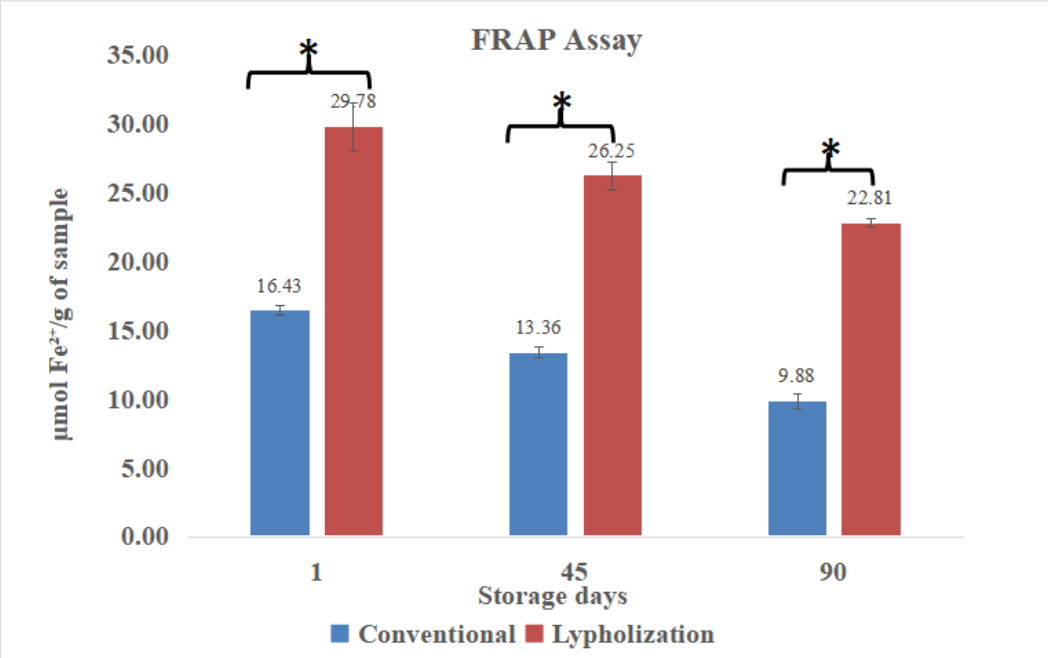

Figure 2. Ferric reducing antioxidant power (FRAP) assay of *G. asiatica* pulp powder subjected to conventional and lyophilized drying at different storage intervals. * indicates statistically significant differences among parameters across treatments and storage intervals.

Table 4. Color parameters of conventional *versus* lyophilized *G. asiatica* berry pulp powder at different storage intervals.

Drying method	Storage days	L* value	a* value	b* value	Chroma value	Hue value
Conventional drying	0	$31.19^{a} \pm 0.77$	$29.30^{c} \pm 0.57$	$5.83^{b} \pm 0.04$	$29.88^{a} \pm 0.56$	$22.52^{b} \pm 0.32$
	45	$31.62^{a} \pm 0.66$	$28.15^{c} \pm 0.18$	$5.99^{b} \pm 0.01$	$28.78^{a} \pm 0.17$	$24.02^{c} \pm 0.19$
	90	$32.43^{b} \pm 0.47$	$27.84^{d} \pm 0.13$	$6.78^{a} \pm 0.06$	$28.65^{a} \pm 0.13$	$27.40^{d} \pm 0.28$
Lyophilization drying	0	$33.77^{c} \pm 0.59$	$40.60^{a} \pm 0.49$	$4.61^{c} \pm 0.09$	$40.88^{c} \pm 0.51$	$12.96^{a} \pm 0.20$
	45	$33.75^{c} \pm 0.67$	$39.85^{b} \pm 0.13$	$4.35^{c} \pm 0.18$	$40.09^{c} \pm 0.15$	$12.48^{a} \pm 0.49$
	90	$34.43^{d} \pm 0.47$	$39.73^{b} \pm 0.12$	$4.73^{c} \pm 0.05$	$40.01^{c} \pm 0.13$	$13.58^{a} \pm 0.12$

Data are "Means ± Standard deviation". Superscript letters (^{a,b}) show the statistical differences at ($p < 0.05$) within a column, whereas the start day of the experiment (0 day) refers to the samples after immediate treatment of the samples with drying techniques.

3.5. pH, brix, and water activity of G. asiatica berry pulp powder

The effect of drying, including conventional versus lyophilization on pH, Brix (total soluble solids), and water activity at different storage for *G. asiatica* berry pulp powder depicted in Table 5, showed significant differences for the drying method and storage interval. The key differences observed can impact the stability and quality of *G. asiatica* powder as a potential food ingredient. A rise in pH of conventional dried sample from 3.58 ± 0.07 to 3.90 ± 0.42 was observed whereas a

slight rise suggests a reduction in acidity, possibly due to the degradation of organic acids during storage. Also, the higher pH during storage could lead to microbial spoilage. The pH of lyophilized *G. asiatica* increased from 3.47 ± 0.14 to 3.71 ± 0.18 over the same period but remained lower than the conventionally dried sample. The consistently lower pH indicates better retention of acidic compounds, which may help preserve the tart flavor characteristic of a lower pH. On the other hand, the Brix value slightly decreased from 18.38 ± 0.24% to 17.58 ± 0.14% by the end of the study period in conventionally dried *G. asiatica*. Lyophilized *G. asiatica* powder showed an initial Brix value of 13.46 ± 0.42%, which decreased slightly to 12.84 ± 0.13% on the 90th day. In the CDP sample, the water activity increased from 0.51 ± 0.07 to 0.61 ± 0.05 over 90 days. The rise in water activity suggests an increased availability of free water. The lyophilized *G. asiatica* originally had reduced water activity of 0.43 ± 0.03 but increased to 0.49 ± 0.02 after 90 days of storage. The reduced initial water activity suggests that lyophilization efficiently decreases the quantity of free water, increasing microbial stability and prolonging shelf life of the developed powder.

3.6. Sensory evaluation of G. asiatica berry pulp powder subjected to conventional and lyophilization drying

The results of sensory evaluation of *G. asiatica* berry pulp powder for color, taste, aroma, texture, and overall acceptability presented in Table 6 reported significant differences due to drying methods and storage. At the start of storage, the attributes of powder subjected to LPP performed better than CDP in every category of sensory evaluation. The highest scores were recorded for color (9.14), taste (9.13), aroma (9.33), texture (9.14), and overall acceptability (9.50). While the sensory quality of both powders decreased over time, LPP continuously scored higher than CDP. When it came to color (8.15 vs. 7.47), taste (8.08 vs. 7.86), aroma (8.17 vs. 7.87), texture (8.37 vs. 8.10), and overall acceptability (7.77 vs. 7.50) after 90 days, LPP outperformed CDP. This pattern suggests that lyophilization preserved sensory properties better.

Table 5. Physiochemical attributes, including pH, brix, and water activity, of *G. asiatica* berry pulp powder subjected to conventional and lyophilization drying at various storage intervals.

Drying method	Storage days	pH	Brix	Water activity
Conventional drying	0	$3.58^{c} \pm 0.07$	$18.82^{c} \pm 0.24$	$0.51^{b} \pm 0.07$
	45	$3.74^{b} \pm 0.05$	$17.23^{b} \pm 0.74$	$0.57^{b} \pm 0.04$
	90	$3.90^{a} \pm 0.42$	$17.58^{a} \pm 0.14$	$0.61^{a} \pm 0.05$
Lyophilization drying	0	$3.47^{d} \pm 0.14$	$13.46^{d} \pm 0.42$	$0.43^{c} \pm 0.03$
	45	$3.53^{c} \pm 0.16$	$13.24^{d} \pm 0.26$	$0.45^{c} \pm 0.05$
	90	$3.71^{b} \pm 0.18$	$12.84^{e} \pm 0.13$	$0.49^{a} \pm 0.02$

Data are "Means ± Standard deviation". Superscript letters (^{a,b}) show the statistical differences at ($p < 0.05$) within a column, whereas the start day of the experiment (0 day) refers to the samples after immediate treatment of the samples with drying techniques.

Table 6. Sensory evaluation of conventionally and lyophilized *G. asiatica* berry pulp powder at various storage levels.

Treatments	Storage days	Color	Taste	Aroma	Texture	Overall acceptability
Conventionally	0	$7.90^{a} \pm 0.57$	$7.70^{a} \pm 0.82$	$8.10^{a} \pm 0.57$	$7.80^{a} \pm 0.63$	$7.90^{a} \pm 0.74$
Dried phalsa	45	$6.80^{b} \pm 0.42$	$6.60^{b} \pm 0.52$	$6.60^{b} \pm 0.52$	$6.20^{b} \pm 0.79$	$6.50^{b} \pm 0.71$
powder (CPD)	90	$6.70^{b} \pm 0.48$	$6.10^{b} \pm 0.88$	$6.30^{b} \pm 0.95$	$5.70^{b} \pm 1.25$	$60.0^{b} \pm 1.05$
Lyophilized dried	0	$8.80^{a} \pm 0.42$	$8.70^{a} \pm 0.48$	$8.80^{a} \pm 0.42$	$8.40^{a} \pm 0.84$	$8.70^{a} \pm 0.48$
phalsa powder	45	$8.60^{a} \pm 0.52$	$8.30^{a} \pm$ o.67	$8.40^{a} \pm 0.52$	$7.90^{a} \pm 0.99$	$8.10^{a} \pm 0.74$
(LDP)	90	$8.20^{a} \pm 0.63$	$7.90^{a} \pm 0.87$	$8.10^{a} \pm 0.57$	$7.60^{a} \pm 0.70$	$7.70^{a} \pm 0.48$

Data are "Means ± Standard deviation". Superscript letters (^{a,b}) show the statistical differences at ($p < 0.05$) within a column, whereas the start day of the experiment (0 day) refers to the samples after immediate treatment of the samples with drying techniques

4. Discussion

We found that lyophilization is significantly effective for removing moisture to extend product shelf life and prevent microbial growth. Non-significant differences in ash, fiber, and fat after a storage trial in LPP indicated effectiveness of the freeze-drying method. Moreover, lyophilization not only preserves nutrients more effectively but also contributes to higher overall energy content in the dried *G. asiatica* powder. In this context, the researchers in [1] reported that the contents of carbohydrates, protein, fat, and crude fiber of fresh *G. asiatica* were 39.74%, 17.41%, 11.19%, and 26.16%, respectively. There is limited literature available on the proximate analysis of freeze-dried *G. asiatica* berry powder. The variations in these parameters are due to the variety of berry, temperature, and storage conditions. Similarly, another study by the researchers in [25] reported a slight increase in protein and ash contents in lyophilized Guabiju (*Myrcianthes Pungens*) fruit when compared with in-natural Guabiju fruit that has not been subjected to any drying method.

Phosphorus and magnesium levels in *G. asiatica* were relatively stable in both samples subjected to drying treatments, whereas K, Fe, and Zn content showed minimal loss during freeze-drying. However, there was a prominent decrease in phosphorus using the CAD technique compared with the freeze-drying method of drying. Similarly, the researchers in [26] also reported that mineral content was higher in lyophilized *G. asiatica* pulp powder when compared with lyophilized *G. asiatica* seed powder. The results indicated that freeze-drying was better able to retain nutritional, physiochemical, and sensory qualities of *G. asiatica* berries.

The therapeutic potential of natural foods depends on the presence of bioactive constituents, mostly due to polyphenols, anthocyanins, and flavonoids. *G. asiatica* is considered to have potent therapeutic potential due to the presence of anthocyanins and phenolic compounds considered beneficial for human consumption. However, a substantial proportion of these bioactive compounds is destroyed during drying and processing, and in this regard, a drying process like freeze drying could be a potential strategy to preserve the bioactive of fruit and fruit-based products. Accordingly, the researchers in [27] reported that freeze-dried berries had the highest percentage of total phenols (2.56 ± 0.02), while hot air-dried berries showed significant reduction in total phenolic contents except for cranberries. It was concluded that lyophilization helps to retain nutritional and phytochemical properties in different berries. Additionally, the researchers in [28]

stated that freeze drying resulted in the dominant retention of anthocyanins in blueberries by retaining kuromanin, which is the most abundant anthocyanin. Furthermore, temperature sensitivity of anthocyanin was observed when low kuromanin levels were reported in high temperature drying methods. Concerning nutritional value, vitamin C is the most labile of all water-soluble vitamins. This is because it is sensitive to heat, light, and ultraviolet radiation and is susceptible to oxidation in the presence of oxygen from the environment (Hou et al., 2019). The highest levels of vitamin C were reported in frozen dried samples compared with other drying methods. However, freeze dried berry samples 50 °C exhibited 20% loss within 20 hours. The researchers in [29] reported that the increase in drying temperature resulted in decreased polyphenol and antioxidant activity.

Sensory attributes, including taste, color, aroma, texture, and overall acceptability, showed significant improvement in LPP, with maximum color and taste retention. The maximum score in sensory attributes proves palatability and consumer satisfaction for dried phalsa pulp powder. These findings are supported by the researchers in [30], who identified the freeze drying technique as the most effective against black currant and sea buckthorn, with minimal losses of sensorial attributes in terms of color, taste, total sugars, and organic acid contents. Sensorial attributes, including color, texture, flavor, and appearance, showed significant differences when compared with conventional drying methods.

The findings by [31] demonstrate different drying conditions responsible for varying luminosity (L*), redness (a*), and yellowness (b*) depending on the drying method for food products. The literature reports that drying under high temperature causes significant deterioration in berries' color brightness and saturation along with sensorial parameters like aroma and texture. Similarly, the researchers in [32] demonstrated a slight reduction in the saturation index (C) of freeze-dried strawberry pulp and seeds, independent of the product thickness; however, the berries' processes with conventional drying showed a major decline in physiochemical attributes, including, color degradation, aroma, Brix, and total acidity of berries. The brightness of freeze-dried samples is mainly attributed to the presence of sugars in lyophilized berries [33]. These findings suggest that lyophilized *G. asiatica* powder had better nutritional and sensorial results due to a very low temperature and sublimation technique with a longer shelf life, as mentioned by [24].

Lyophilized berries are considered a promising approach to obtain a higher retention of predominant phytochemicals and nutritional attributes [34]. The comparison of parameters, including L*, a*, and b*, showed significant differences for lyophilized samples compared with conventionally dried samples [24]. We found that lyophilized *G. asiatica* powder after 90 days storage had a minimal decrease of L*, which indicates the presence of brown pigments and oxidation of heat-sensitive nutrients; however, the *G. asiatica* pulp dried through conventional drying showed a substantial reduction in the saturation index of *G. asiatica*. The chromaticity of *G. asiatica* pulp powder was compared, which indicated a maximum reduction in non-enzymatic browning in LPP; however, the browning index showed higher CDP, causing a significant change in the color tone of a sample, making variations in natural color. These findings correlate with the results in [35], where the researchers compared the browning index (BI) of lyophilized avocado paste with natural fresh avocado pulp and found a higher browning index of lyophilized avocado paste when temperature was controlled above 40 °C, suggesting that degradation of heat sensitive nutrients causes accelerated non-enzymatic browning. However, the browning index may be increased depending on the type of freezing and pressure employed on a sample, giving the *G. asiatica* berry pulp a brownish tint. Similar results were reported by the researchers in [36], who observed a considerable browning

index in freeze dried strawberries subjected to increased working pressure and heating temperature, affecting the quality of final product.

5. Conclusions

Our findings indicated that lyophilization of *G. asiatica* berry represents excellent nutritional profiles, bioactive compounds, vitamin C, and minerals, especially zinc and phosphorus, at different storage interval compared to conventional drying. Nutrient stability during storage can be made possible with improved storage conditions and optimizing preservation techniques. Freeze drying *G. asiatica* resulted in powder with better nutrients and consumer acceptability; thus, this powder could be applied in different processed foods, and preserve nutrient content of highly perishable phalsa fruit, resulting in less waste, increased shelf life, and sustainability of the developed powder product. Additionally, phalsa berry powder showed improved functional and physiochemical properties, which may open new avenues in product development in the form of high-quality food formulations and nutraceutical applications. Lyophilized *G. asiatica* berry powder is recommended to be packaged and sealed with aluminum foil in a light free environment with a temperature range of 20–25 °C for LPP for storage and preservation.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

Data availability

The datasets used and/or analyzed during the current study are available from the corresponding author upon reasonable request.

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Conflict of interest

The authors declare no conflicts of interest related to this research.

Author Contributions

The idea was conceived by MS, SA, and MTS, the research was supervised and reviewed by them whereas, SL conducted field research, carried out the laboratory analyses, and drafted the original document. The original draft was reviewed, supervised, and analyzed by HM.

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