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Spray-Dried Nipa Palm Vinegar Powder: Production and Evaluation of Physicochemical, Nutritional, Sensory, and Storage Aspects

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Abstract: Nipa palm vinegar (NPV) is a naturally fermented vinegar derived from the nipa palm (*Nypa fruticans* Wurmb) sap. This work optimized production of spray-dried nipa palm vinegar powder. The influence of the various drier air inlet temperatures (150, 170, and 190 °C) and maltodextrin DE10 carrier concentrations (15 and 20% *w/v*) in the feed, on the characteristics of the product powder was investigated. Nipa palm vinegar powder (NPVp) was evaluated in terms of the following responses: physicochemical and nutritional properties, sensory acceptability, and storage stability. All processing variables affected the responses. Based on product desirability as the optimization criterion, spray-drying with a hot air inlet temperature of 170 °C with a 15% *w/v* maltodextrin DE10 in the feed was optimal. The nutritional characteristics of the product made under the above identified optimal conditions were (per 100 g dry product): a calorific value of 366.2 kcal; 1.3 g protein; 88.1 g carbohydrate; 0.96 g fat; 883.9 mg potassium; 12.7 mg vitamin C; and 105 mg gallic acid equivalent (GAE) phenolics content. The product, vacuum-packed and heat-sealed in aluminum laminated polyethylene bags, could be stored at 25 °C for at least 180 days without noticeable loss in quality.

Keywords: nipa palm vinegar; nipa palm vinegar powder; *Nypa fruticans* Wurmb; spray-drying of nipa palm vinegar



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1. Introduction

Nipa palm (*Nypa fruticans* Wurmb) is a high-yielding sugar palm found commonly in mangrove ecosystems in Southeast Asia and Oceania [1–3]. In Thailand, nipa palm occurs in southwestern coastal regions bordering the Gulf of Thailand [4]. Nipa palm trees are tapped for the sugar-rich nipa palm sap [1,5] that is consumed fresh, and fermented to wine. The wine may be distilled to produce other alcoholic beverages, or further fermented to produce nipa palm vinegar (NPV) [1,6]. Production of NPV from fresh sap involves a two-stage fermentation. In the first stage, yeasts ferment the carbohydrates and sugars to alcohols and carbon dioxide. In the second aerobic fermentation, acetic acid bacteria convert the ethanol to acetic acid [4,5].

NPV is used as a food condiment and also consumed locally between meals and before bedtime [4,7]. NPV is also used as a food preservative [1]. Various health benefits are ascribed to NPV based mostly on in vitro studies [4,5,7,8]. NPV is mostly a regional product. Its utility as a food condiment can be enhanced by drying it to a stable, easy to distribute, NPV powder.

Spray drying is generally the method of choice for liquid foods as it is rapid and less damaging to the sensory and nutritional characteristics. Spray drying involves atomizing

the liquid into fine droplets in a hot gas stream to produce a dry powder [9]. Compared to the liquid form, a dry product has a reduced volume and weight and superior storage stability over extended periods [10]. Spray drying is also widely used for other highly heat-sensitive biological products such as pharmaceutical proteins and enzymes because the drying is rapid and, therefore, the activity loss is minimal [11]. A carrier agent such as the polysaccharide maltodextrin (MD) is often added to liquid products prior to spray drying. Maltodextrin acts as a filler, thickener, and texturizing agent. Maltodextrin is edible and generally recognized as safe (GRAS) according to the classification of the United States Federal Drug Administration (FDA) [10,12,13].

Fruit juices [12–15] and certain vinegars are commonly spray dried. The latter include the Chinese black vinegar [16,17], coconut water vinegar [18], and bamboo vinegar [19]. Unlike some other vinegar powders, nipa palm vinegar powder is not commercially available. No work has been reported on the effects of spray drying on nutritional and sensory characteristics of NPV, the organoleptic characteristics of the nipa palm vinegar powder (NPVp) in foods and beverages, and the storage stability of NPVp.

The present work aimed to identify the suitable conditions (drying air temperature, maltodextrin concentration in the NPV) for producing spray-dried NPV powder with acceptable sensory and nutritional attributes. The storage stability of the dried product was assessed as an important commercial characteristic.

2. Materials and Methods

2.1. Chemicals and Materials

The chemicals and reagents were of analytical grade and purchased from Sigma-Aldrich (St. Louis, MO, USA) unless specified otherwise. Maltodextrin with a dextrose equivalent (DE) value of 10 was obtained from Perfect Natural Food Powder and Flavor 2002 (Thailand) Co., Ltd. (Kratumban, Samutsakorn, Thailand).

All materials used in preparing consumable products were of food grade or higher quality. Hygienic practices in food preparation were followed.

2.2. Nipa Palm Vinegar

The nipa palm (*Nypa fruticans* Wurmb) vinegar product was obtained from a local market (Kanapnak sub-district, Pak Phanang district, Nakhon Si Thammarat province; 8°12'25.1'' N latitude, 100°14'51.7'' E longitude) in Thailand. The vinegar had been naturally fermented using a traditional local method, which involved incubating nipa palm sap from cut stalks in terracotta containers at room temperature (25–30 °C) for 40 days. The microorganisms found naturally in the raw sap were the fermenting agents [4]. The nipa palm vinegar lots were obtained from the same supplier. Fermented nipa palm vinegar had a pH of 2.67 ± 0.02 and an acetic acid content of $4.42 \pm 0.02\%$ *w/v* (g/100 mL).

The nipa palm vinegar was transferred to a clean container and filtered through a fine-mesh sifter (40 mesh, 420 μm) to remove any suspended matter and stored at 4 °C until use.

2.3. Spray Drying of Nipa Palm Vinegar

Different concentrations of maltodextrin DE10 (10, 15, and 20% *w/v* (g/100 mL)) were mixed with nipa palm vinegar in different experiments. The resulting solution was filtered through a fine-mesh sifter (40 mesh, 420 μm) and used as the feed for spray drying.

A pilot-scale spray drier with a rotary atomizer (model SDE-10, JCS Technic Line Co., Ltd., Samutsakorn, Thailand) was used in all work. The cylindrical drying chamber made of stainless steel had an internal diameter of 0.9 m and a total height of 2.3 m. The variables studied were the maltodextrin concentration (10, 15, and 20% *w/v*) in the feed and the inlet air temperatures (150, 170, and 190 °C). The outlet air temperature was held at 90 °C. The feed flow rate was controlled at 500 mL h^{-1} . The atomization air flow rate was always 5 $\text{m}^3 \text{h}^{-1}$ and the fan blower speed was 13,000 rpm. The spray dried product (NPV powder) was sifted through a 60-mesh (250 μm) sifter, collected, weighed, and kept in

sealed containers for analysis. Based on the above noted dimensions of the drying chamber and the flow rate of the atomization air, the product residence time (or contact time with the drying air) in the drying chamber was always well below 42 s.

Preliminary spray drying trials revealed no powder recovery in the cyclone when feed contained 10% *w/v* of maltodextrin as the solid particles stuck to the walls of the drying chamber. Therefore, the maltodextrin level in the redesigned experiments ranged from 15% *w/v* (low, coded value = −1) to 20% *w/v* (high, coded value = 1).

2.3.1. Experimental Design

Based on preliminary studies, the experiments involved two factors: the inlet temperature (X_1) and the maltodextrin concentration (X_2). The factor X_1 was studied at three levels: coded values of −1 (low), 0 (medium), and 1 (high). The factor X_2 was studied at two levels with coded values of −1 (low) and 1 (high) (Table S1). The six experimental trials involving various combinations of factors were generated using Multilevel Categorical Design (Table S2) implemented via the Design-Expert[®] software (trial version 13; Stat-Ease, Minneapolis, MN, USA; www.statease.com, accessed on 24 May 2022). The powder collected for each set of drying condition was used to calculate the product yield and analyzed for physicochemical properties.

2.3.2. Selection of the Optimal Point

The optimization process entailed identifying the best independent factor values (i.e., X_1 and X_2) to obtain a product with the desired characteristics. The desirability function was used to optimize the independent variables, with individual responses being combined to provide the total desirability. The Design-Expert software was used for the numeric optimization. Each variable was optimized numerically using the desirability function (range 0–1). The goal was to maximize the product output yield and bulk density while minimizing the moisture content and water activity. The constraints adopted for the estimating the overall desirability are shown in Table S3.

2.4. Physicochemical Characterization

2.4.1. Product Yield

The product yield Y (%) of spray-dried NPV powders was calculated using the following equation:

$$Y (\%) = \left(W_f / W_i \right) \times 100\% \quad (1)$$

where W_i was the mass of total solids fed (g) to the spray drier and W_f was the mass of the spray-dried NPV powder (g) recovered.

2.4.2. Bulk Density

The bulk density (BD , g mL^{−1}) of NPV powder was determined according to the method of Goula and Adamopoulos [20]. An exact mass (2 g) of NPV powder was transferred to a 10 mL graduated cylinder. The cylinder was held on the vortex vibrator for 1 min and the volume of the powder was measured. The bulk density was calculated as the known mass divided by the measured volume.

2.4.3. Total Soluble Solids, Moisture Content, and Water Activity

The total soluble solids (TSS , °Bx) was measured using a refractometer (ATAGO DR-A1, Tokyo, Japan).

The moisture content was determined using the oven drying method [21]. A precisely weighed (2 g) sample of the NPV powder was oven-dried (105 °C) to a constant weight. Triplicate samples were measured.

The water activity was measured using a water activity meter (Aqualab Series 3TE; Decagon Devices, Pullman, WA, USA). Measurements were performed at room temperature.

2.4.4. The pH and Acetic Acid Determination

The pH was determined using the Method 981.12 [21] with a pH meter (Mettler-Toledo, LLC, Columbus, OH, USA).

The acetic acid concentration of NPV powder was determined by colorimetric titration [4,22]. The titrant was 1 M sodium hydroxide, and the indicator was 1% (*w/v*, g/100 mL) phenolphthalein solution. NPV powder (10 g) was added to a 250 mL Erlenmeyer flask and dissolved in 20 mL distilled water. Then, three drops of 1% (*w/v*) phenolphthalein solution were added and mixed. The titrant volume at the endpoint when the color turned red was used to calculate the percentage of acetic acid in the sample. Each mole of sodium hydroxide consumed was equivalent to 1 mol acetic acid in the sample.

2.4.5. Color Analysis

The electronic color parameters (L^* , a^* , and b^*) of NPV powder were measured using the Hunterlab Miniscan/EX instrument (10° standard observer, illuminant D65; Hunter Associates Laboratory, Inc., Reston, VA, USA). The device had been calibrated to a white and black standard. For the measurements, the NPV powder sample was weighed and placed on a transparent plastic plate of the instrument and the Hunter color parameters L^* , a^* , and b^* were measured. L^* denoted lightness in the dark–light spectrum range (black = 0, white = 100), a^* was a hue in the red–green color range where negative values indicated green and positive values indicated red, and b^* was a hue in the yellow–blue spectral range where negative values indicated blue and positive values indicated yellow [13,23].

2.5. Nutritional Characteristics

Nutritional composition of the NPVp was analyzed according to AOAC [21] method as follows: total fat (Method 948.15); cholesterol (Method 976.26); protein (Method 981.10); total sugar (Method 925.35); dietary fiber (Method 985.29); the mineral profiles of calcium (Ca), sodium (Na), and iron (Fe) (Method 984.27); and ash (Method 923.03). The amounts of vitamin A (retinol), vitamin B1 (thiamin), and vitamin C (ascorbic acid) were quantified using adaptations of published methods [24]. Carbohydrate content was determined by the method of Sullivan and Carpenter [25]. The calorific value was obtained by using a bomb calorimeter. The total phenolic content in the NPV samples was measured using the method of Chatatikun and Kwanhian [8] with gallic acid as the standard.

2.6. Sensory Evaluations of NPV and NPV Powder

The sensory analyses of the NPV and the NPV powder had been reviewed and approved by the Human Research Ethics Committee of Walailak University, Thailand (approval no. WUEC-20-048-01). A sensory evaluation involving 59 untrained panelists was carried out in a sensory laboratory (Center for Scientific and Technological Equipment 3, Walailak University, Nakhon Si Thammarat, Thailand). The freshly prepared samples of spray-dried NPV powder and the original NPV were evaluated by the panelists.

The recipe for sensory evaluation consisted of Recipe 1 (R1), Recipe 2 (R2), Recipe 3 (R3), Recipe 4 (R4), Recipe 5 (R5), and Recipe 6 (R6). The original NPV was used as R1 (control R1), and the rehydrated NPV powder was used as R2 (NPVp-3 product, control R2). For sensory evaluations, 200 g NPV or NPVp was mixed with 800 g of drinking water (sample: water mass ratio of 1:4) to produce a drink.

In the beverage recipes (R3 and R4), the original NPV was used for R3 and NPV powder was reconstituted as liquid vinegar as R4. For the preparation of the beverage, the NPV (200 g) was mixed with 200 g of honey (Thai Honey, Thailand; <http://www.thaihoney.net>, accessed on 24 May 2022) and 600 g of carbonated water (Singha Soda, Thailand; <https://www.singhacorporation.com/singhasoda/>, accessed on 24 May 2022). The mass ratio of NPV:honey:carbonated water was 1:1:3. The ingredients of R4 were the same as R3, but 200 g of NPVp was added instead of NPV.

For the food recipes (R5 and R6), vinegar was used to increase the acid taste of the food. In recipe R6, 60 g NPVp was added to 1 L of sour soup (Thai sour fish soup made

with sea mullet). While the ingredients for Recipe 5 were the same as for Recipe 6, NPVp was replaced with 60 g of the original NPV.

The sensory attributes evaluated by the panelists for each sample were: color, odor, taste, and overall acceptability. Each sample (15 mL) was served in a random order in white plastic cups coded with three digits. To minimize any residual effects, the panelists were instructed to rinse their mouth with water and take all necessary time to score the samples. The participants were asked to score each sensory attribute on a 9-point scale (1 = dislike very much to 9 = like very much) [26].

2.7. Assessment of Storage Stability of NPV Powder

Spray-dried NPV powder (30 g) (product NPVp-3, Figure 1A) was packed in aluminum-laminated polyethylene bags and heat-sealed under vacuum (Figure 1B). The sealed samples were kept at room temperature (25 ± 2 °C, 75% relative humidity) for 180 days. Entire bags were sampled at 30-day intervals for measuring the pH, the moisture content, water activity, color parameters, and microbial counts. Triplicate samples were analyzed.



Figure 1. The appearance of spray-dried NPV powder product NPVp-3 (A); the sealed aluminum-laminated polyethylene package containing the NPV powder for stability assessments (B).

Microbial Counts

Each NPV powder sample (10 g) was homogenized with 90 mL of sterile 0.1% *w/v* (g/100 mL) peptone solution (Himedia, Mumbai, India). Serial dilutions (9 mL) were prepared using sterile peptone solutions for plating. A portion (1 mL) of the appropriate dilution was plated in duplicate on potato dextrose agar media (Merck, Germany) which had been aseptically acidified with 10% *w/v* (g/100 mL) tartaric acid (Ricca Chemical Co., Arlington, TX, USA). The plates were incubated at 25 °C for 5 days for the enumeration of yeasts and molds [27,28].

2.8. Statistical Analysis

All analyses were performed on triplicate samples. The results were presented as mean values (\pm standard deviation). The results were analyzed by one-way ANOVA (Tukey's test). The mean values at 95% significance level ($p < 0.05$) were taken as a significant difference.

3. Results and Discussion

3.1. Effects of Spray Drying Conditions

Success in spray drying depends critically on the characteristics of the feed, particularly on the concentration of the carrier [10]. The carrier in the present work was maltodextrin DE10. In addition, the inlet temperature of the drying air was important as too high a temperature could damage the product whereas an insufficiently high temperature would

result in a poorly dried sticky product [10]. Preliminary studies were carried out to define the suitable ranges of inlet air temperature and the carrier concentration. The maltodextrin content had to be significantly higher than 10% *w/v* to produce a non-agglomerating powder with the required colloidal stability. In keeping with prior work on spray drying of blueberry juice [29], a maltodextrin content of 10% *w/v* in the feed resulted in no recovery of the powder possibly because the material stuck to the walls of the spray drier. Therefore, two levels of maltodextrin DE10 concentrations (15 and 20% *w/v*) were tested in combination with three levels of air inlet temperature (150, 170, and 190 °C) (Table S1). For the factor combinations used in the six experimental runs (Table S2), the measured responses of the physicochemical properties of the products are shown in Table 1. The data shown (Table 1) are the average values of two independent replicates. The mean squares of average experimental results were subjected to analysis of variance (Table 2).

Table 1. Physicochemical properties of the spray-dried NPV powder formulations.

Product	Condition		Y (%)	BD (g mL ⁻¹)	MC (% w/w)	a _w	TSS (°Bx)	Color		
	T (°C)	MD (%)						L*	a*	b*
NPVp-1	150	15	53.37 ± 0.14	0.46 ± 0.00	2.56 ± 0.06	0.31 ± 0.01	19.50 ± 0.71	91.75 ± 0.06	0.18 ± 0.01	7.91 ± 0.01
NPVp-2	150	20	56.31 ± 0.44	0.47 ± 0.00	3.05 ± 0.07	0.36 ± 0.01	24.10 ± 0.14	91.95 ± 0.06	-0.27 ± 0.00	5.53 ± 0.04
NPVp-3	170	15	53.62 ± 0.11	0.47 ± 0.00	2.08 ± 0.04	0.23 ± 0.00	20.00 ± 0.00	93.57 ± 0.62	0.38 ± 0.01	10.12 ± 0.12
NPVp-4	170	20	58.29 ± 0.05	0.48 ± 0.01	3.03 ± 0.05	0.24 ± 0.00	23.70 ± 0.42	92.80 ± 0.13	0.49 ± 0.01	10.28 ± 0.02
NPVp-5	190	15	47.04 ± 0.44	0.43 ± 0.00	2.63 ± 0.04	0.23 ± 0.00	20.00 ± 0.00	91.53 ± 0.04	-0.09 ± 0.00	6.29 ± 0.03
NPVp-6	190	20	50.72 ± 0.02	0.46 ± 0.00	3.02 ± 0.03	0.24 ± 0.01	24.00 ± 0.00	92.14 ± 0.06	0.54 ± 0.00	6.21 ± 0.01

T, inlet temperature; MD, concentration (% *w/v*) of maltodextrin DE 10; Y, product yield; BD, bulk density; MC, moisture content, a_w, water activity; TSS, total soluble solids. Results are mean values ± standard deviations of measurements on duplicate samples of the final product.

Table 2. Analysis of variance (ANOVA) of physicochemical characteristic responses of spray-dried NPV powders.

Source of Variation	Y	BD	MC	a _w	TSS	Color		
						L*	a*	b*
Model	31.94 **	0.0005 **	0.2966 **	0.0054 **	10.19 **	1.16 **	0.2132 **	8.58 **
Inlet temperature (X ₁)	57.9 **	0.0008 **	0.094 **	0.0122 **	0.0433 NS	35.19 **	0.2295 **	18.59 **
Maltodextrin concentration (X ₂)	42.41 **	0.0007 **	1.12 **	0.0015 **	50.43 **	0.0099 NS	0.029 **	1.76 **
X ₁ × X ₂	0.7493 **	0.0002 **	0.0892 **	0.0006 **	0.21 NS	7.41 **	0.289 **	1.97 **
R ²	0.9974	0.9814	0.9903	0.9940	0.9864	0.9342	0.9999	0.9996

Y, product yield; BD, bulk density; MC, moisture content; a_w, water activity; TSS, total soluble solids.; R² values indicate the goodness of fit of the theoretical models (see Table S4) to the experimental data; NS, nonsignificant (*p* > 0.05); **, significant (*p* < 0.05).

3.1.1. Yield of NPV Powder

The product yield of the spray-dried NPVp samples (NPVp-1 to NPVp-6) ranged from 58.3 to 47.0% (Table 1). The inlet temperature and maltodextrin concentration both had substantial effects (*p* < 0.05) on the yield of the powdered product (Table 2). As the air inlet temperature was increased from 150 °C to 190 °C, the product yield reduced by 19.3%. This was consistent with similar findings reported for spray-drying of mountain tea extract [30], soluble sage (*Salvia fruticosa* Miller) [31], and a mandarin (*Citrus unshiu*) beverage [32]. The decreasing yield was associated with the melting of the powder at higher temperatures and deposition on the walls of the drying chamber [30–32]. In contrast to the temperature effect, a raising of the maltodextrin concentration in the feed from 15% to 20% *w/v* increased the product yield (Table 1). This was simply the effect of a higher concentration of soluble solids in the feed [30,31].

The interactive effect of the variables on yield was positive (*p* < 0.05; Table 2). The experimental data agreed well with the predictive model (Table S4) as evidenced by R² of 0.9974 (Table 2). Similar positive interactive effects of drying air temperature and maltodextrin content in the feed, on product yield were reported by others [13,14,33].

3.1.2. Bulk Density

A high bulk density reduces the volume for a given mass of product, reducing cost of packaging and shipping. In addition, powders with high bulk densities have superior flow properties compared to agglomerated powders with a low bulk density [10]. The bulk density of the NPV powders varied from 0.43 to 0.48 g mL⁻¹ (Table 1). These values were higher compared to data reported for spray-dried instant soluble sage [31] and spray-dried whey protein mixes [34] made under conditions similar to those of the present work. Both the inlet temperature and maltodextrin concentration significantly ($p < 0.05$) affected the bulk density of NPVp (Table 2). The experimental data of bulk density agreed well with the predictive model ($R^2 = 0.9814$; Table 2) shown in Table S4.

3.1.3. Moisture Content and Water Activity

Dry products generally have a good storage stability if the moisture content is less than 5% [20,31,35]. In the present study, the moisture content of the NPV powders was in the range of 2.1–3.1% (Table 1). Individually, both the experimental factors (X_1 , X_2) significantly ($p < 0.05$) affected the moisture contents of the NPV powders (Table 2). In addition, the moisture content was significantly affected ($p < 0.05$) by the interactive effect of the factors (Table 2). A similar behavior was reported during drying of amla juice [13]. A high drying temperature increased the rate of heat transfer into the drying particles, resulting in faster evaporation of water [36]. A higher concentration of the carrier in the feed resulted in a higher moisture content in the product (Table 2), although the moisture content was always well below 5%. A similar effect of the carrier concentration was previously reported for drying of amla juice [13].

The water activity of fruit and vegetable juice powders typically ranges from 0.2 to 0.6 [10]. The NPVp samples in the present work had water activities in the range of 0.23–0.36 (Table 1). Food with a water activity of less than 0.6 is generally regarded as microbiologically stable [37], although deterioration may occur through chemical reactions such as oxidation. The effects of the factors (X_1 , X_2) on water activity were comparable to their effects on the moisture content as discussed earlier in this section. An increased concentration of the carrier increased the water activity of the product, and this effect was statistically significant ($p > 0.05$) (Table 2). Similar effects of carriers have been reported in spray drying of various fruit juices [28,32,34].

3.1.4. Total Soluble Solids

A significantly high total soluble solids were observed in the product if the feed contained 20% *w/v* maltodextrin instead of 15% *w/v* maltodextrin (Table 1). This was understandable because the total soluble solids in the feed were higher if the carrier concentration was higher. The total soluble solids were not significantly affected by the inlet temperature of the drying air as the minimum temperature used was satisfactory for drying (Table 2). Similarly, the interactive effect of factors on total soluble solids was not significant (Table 2).

3.1.5. Color

All NPV powders had an off-white general appearance (Figure 1A; Table 1). In numerical terms, the color was characterized by the parameters L^* , a^* , and b^* (Table 1). The L^* lightness scale ranged from 0 (dark) to 100 (light), with 50 as the midpoint. Thus, the NPV powders had L^* values of 91.5–93.6 (Table 1), indicating that they all were well toward the lighter end of the scale. The low positive value of a^* (−0.27 to 0.54; Table 1) revealed a hue slightly more green ($a^* = -100$) than red ($a^* = 100$). Furthermore, the low positive b^* (5.53–10.28, Table 1) indicated that the color was slightly yellowish ($b^* = -100$) rather than blue ($b^* = 100$). The product NPVp-3 is depicted in Figure 1A.

The a^* and b^* color values of the NPV powders were significantly affected ($p < 0.05$) by the inlet temperature and maltodextrin content, but maltodextrin concentration had no effect on L^* values (Table 2). Other work has shown similar trends of the drying temperature

affecting the lightness (L^*) of the spray dried product [37]. The hue of the product was largely influenced by the white color of the native maltodextrin carrier.

3.2. Optimization of Design

The goal of the optimization step was to identify the optimum values of the independent variables for producing a product with the desired properties. The desirability function was created by optimizing independent variables and combining the individual responses to produce an overall desirability. The numeric optimization was implemented using the Design-Expert software. The values of the product yield, bulk density, moisture content, and water activity were set to the target values for generating the optimized product (Table S3).

The highest value of overall desirability (0.921, Figure 2) could be obtained in a product that combined a high product yield and bulk density with low moisture content and water activity. The product with a desirability of 0.921 was the NPVp-3 produced using an inlet air temperature of 170 °C and a feed with 15% w/v of maltodextrin. For NPVp-3, the predicted values of the product yield, bulk density, moisture content, and water activity were 53.62%, 0.47 $g\ mL^{-1}$, 2.08% w/w , and 0.23, respectively. The NPVp-3 had the measured attributes values that were within 5% of the predicted values, thus validating the optimized model (Table S4).

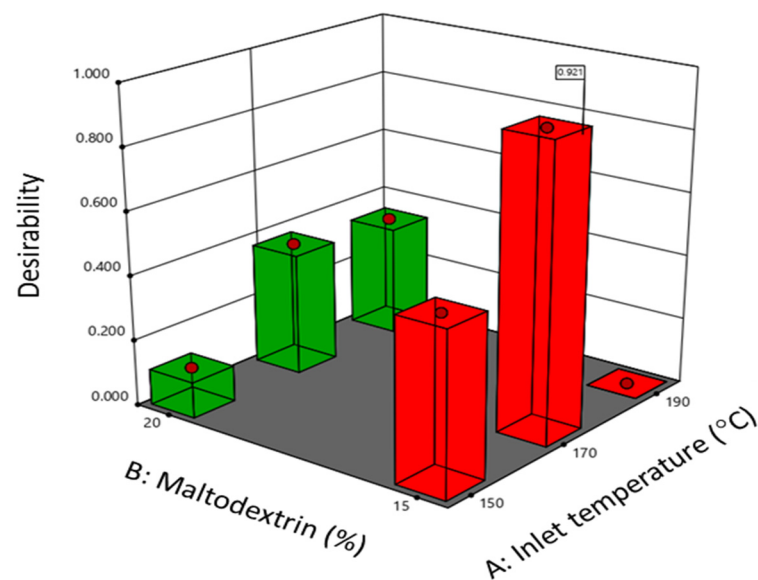


Figure 2. The overall desirability response of the products made using various combinations of the drying air inlet temperature ($X_1 = A$) and maltodextrin concentration ($X_2 = B$). The product with the highest desirability (0.921) had a drying temperature of 170 °C and 15% w/v of maltodextrin content. Design points are shown as red circles.

3.3. Nutritional Characteristics

A comparison of the nutritional parameters of the NPVp-3 powder with a commercial product is shown in (Table 3). The NPVp-3 powder had around 96% of the calorific content as the commercial product possibly because of its lower level (91.5% of commercial product) of total carbohydrate compared to the commercial product (Table 3). The protein content of NPVp-3 was ~27-fold greater compared to the commercial product and the total fat was also greater (~10-fold; Table 3) possibly due to natural variations of fat and protein in the vinegar, depending its original source.

Table 3. Nutritional analysis of the NPVp-3 product compared with a commercial vinegar powder sample.

Test Item	Unit/100 g ^{a,b}	NPVp-3 Powder	Commercial Vinegar Powder ^d
Energy	kcal	366.16 ± 3.92	380.9
Protein	g	1.33 ± 0.01	0.05
Crude fat	g	0.96 ± 0.013	0.1
Cholesterol	mg	Not detected	0
Carbohydrate	g	88.05 ± 1.88	96.2
Total sugars	g	14.16 ± 0.85	3.5
Total dietary fiber	g	0 ± 0.00	0.22
Ash	g	3.03 ± 0.01	-
<i>Minerals</i>			
Calcium (Ca)	mg	30.92 ± 0.71	25
Iron (Fe)	mg	0.54 ± 0.00	0.41
Sodium (Na)	mg	268.56 ± 0.12	79
Potassium (K)	mg	883.90 ± 0.35	-
Phosphorous (P)	mg	35.90 ± 0.03	-
Magnesium (Mg)	mg	21.50 ± 0.13	-
Zinc (Zn)	mg	0.26 ± 0.00	-
<i>Vitamins</i>			
Vitamin A	µg	0.00 ± 0.00	0.54
Vitamin B1	mg	Not detected	-
Vitamin B2	mg	Not detected	-
Vitamin C	mg	12.68 ± 0.10	0.27
<i>Other constituents</i>			
Total phenolics content	µg GAE ^c g ⁻¹	1049.76 ± 20.03	-
Acetic acid	% w/v	5.13	12

^a Samples were analyzed in triplicate. Data are average values ± standard deviations.; ^b Dry weight basis.; ^c GAE, gallic acid equivalent; ^d Values are based on the literature (Spice Barn[®] vinegar powder, Spice Barn, Inc.; https://spicebarn.com/vinegar_powder.htm (accessed on 24 May 2022)).

The NPVp-3 powder had similar levels of calcium and iron as the commercial product, but was considerably richer in the other minerals listed in Table 3. The total vitamin C in NPVp-3 powder was nearly 47-fold greater than in the commercial product, possibly because the spray drying conditions used preserved a higher proportion of this heat-labile vitamin compared to the processing conditions used in making the commercial product. The commercial product had a small amount of vitamin A (Table 3), but this was too low to be meaningful compared to ~900 µg daily requirement of this vitamin in adult males.

Gallic acid equivalent (GAE) is a commonly used measure of total phenolics in foods. Phenolics are antioxidants and also protect food against oxidative damage. The NPVp-3 powder had a high level of total phenolics (~1050 µg GAE g⁻¹; Table 3) compared to literature data (167 µg GAE mL⁻¹; [8]) for reported for NPV. The elevated level of phenolics was explained at least partly by the concentrating effect of spray drying.

Natural liquid vinegars typically have more than 4 g of acetic acid per 100 mL (FDA, <https://www.fda.gov/media/71937/download> (accessed on 24 May 2022)). The NPVp-3 powder had ~5% w/v acetic acid (Table 3) because much of it was lost by vaporization during drying.

3.4. Sensory Acceptance

The sensory evaluations of the beverage/food preparations containing the original NPV and the product NPVp-3 for color, odor, taste, and overall acceptability are shown in Figure 3. The six recipes (R1–R6) were scored by the 59 panelists as previously explained.

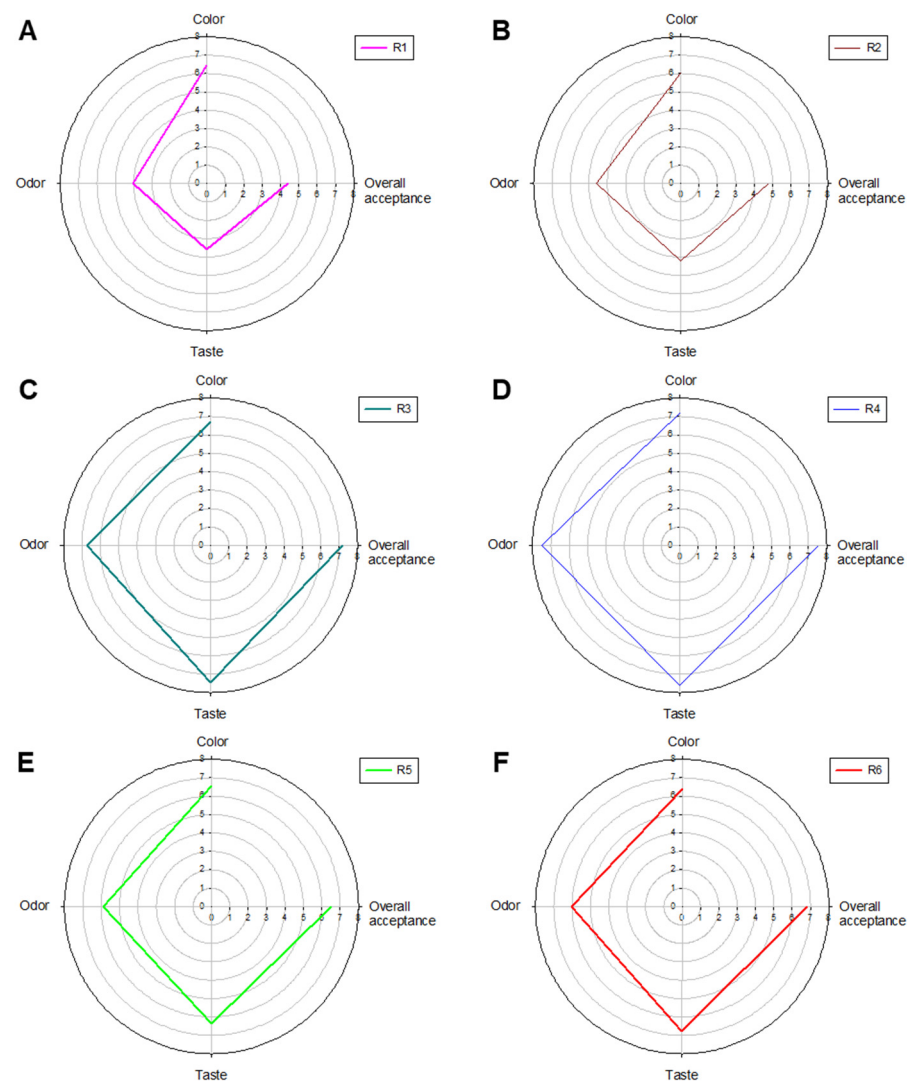


Figure 3. Spider charts of sensory evaluation scores of the original NPV and the NPVp-3 powder used as a beverage and food additive: (A) Recipe R1 containing the original NPV (control R1) 200 g in 800 g of drinking water; (B) Recipe R2 containing 200 g of rehydrated NPV powder (control R2) in 800 g of drinking water; (C) Recipe R3 containing the original NPV (200 g), 200 g honey, and 600 g carbonated water; (D) Recipe R4 containing NPVp-3 (200 g), 200 g honey, and 600 g carbonated water; (E) Recipe R5 containing the original NPV (60 g) in 1 L of sour soup (Thai sour sea mullet fish soup); and (F) Recipe R6 containing NPVp-3 (60 g) in 1 L of sour soup (as above). The data are mean values \pm standard deviations ($n = 59$). Each score was generated by 59 individuals using a 9-point hedonic scale (1 = dislike very much; 9 = like very much).

The test materials containing the original NPV were used as controls (R1, Figure 3A; R3, Figure 3C; R5, Figure 3E). The various drink/food recipes are explained in the caption of Figure 3. The recipes contained either the original NPV, or NPVp-3, mixed with: drinking water (R1, R2); honey and carbonated water (R3, R4); and Thai sour fish soup (R5, R6). The recipes containing NPVp-3 (R2, Figure 3B; R4, Figure 3D; R6, Figure 3F) used the same ingredients as in the control preparations containing the original NPV (Section 2.6).

The sensory scores of color, odor, taste, and overall acceptability for the recipes R1 and R2 (Figure 3A,B) were not substantially different. R1 and R2 received overall acceptance scores of 4.41 ± 1.63 and 4.73 ± 1.72 , respectively. NPVp-3 recipe R2 received a little higher rating than the recipe R1 that contained the original NPV. The difference was explained by the more intense vinegary odor and the characteristic sour flavor of NPV due to its higher content of acetic acid.







In the beverage recipes R3 and R4 (Figure 3C,D), the recipe R4 made with NPVp-3 scored somewhat higher than R3 (made with the original vinegar) for all the scored attributes. Overall, the tasters better accepted the beverages made with NPVp-3.

Original NPV (Recipe R5) and NPVp-3 powder (Recipe R6) were used to season the fish soup (Figure 3E,F). The sensory parameter ratings for both were fairly comparable, but R6 scored somewhat higher than the original NPV. Although the differences were small, the tasters preferred the beverage recipe R4 (Figure 3D) based on NPVp-3 more than the same product used as a food seasoning (R6, Figure 3F).

3.5. Product Stability during Storage

Variations in the relevant physicochemical parameters of NPVp-3 stored for various periods at 25 °C are shown in Table 4. The off-white appearance of the samples barely changed during 180 days of storage (Table 4). In terms of the electronic color parameters (L^* , a^* , b^* ; Table 4), there were minor changes: a slight but fairly consistent increase in a^* value with the length of storage, and a similar increase in the b^* value. The sample lightness (L^*) was always within 94.1 and 95.1 (Table 4).

Table 4. Effect of storage duration on appearance, pH, moisture content, water activity, color, and microbial contents (molds and yeasts) of the product NPVp-3 during storage at 25 °C.

Property	Storage Duration (Days) ^A					
	0	30	60	90	120	180
Appearance						
pH	3.20 ± 0.01 ^{ab}	3.18 ± 0.01 ^a	3.18 ± 0.01 ^a	3.21 ± 0.01 ^b	3.21 ± 0.00 ^b	3.21 ± 0.00 ^b
Moisture (% w/w)	2.61 ± 0.01 ^a	2.62 ± 0.02 ^a	2.61 ± 0.01 ^a	2.61 ± 0.01 ^a	2.62 ± 0.02 ^a	2.62 ± 0.01 ^a
Water activity	0.23 ± 0.00 ^a	0.23 ± 0.00 ^a	0.23 ± 0.00 ^a	0.23 ± 0.00 ^a	0.24 ± 0.00 ^a	0.24 ± 0.00 ^a
Color						
L^*	94.13 ± 0.18 ^a	95.07 ± 0.13 ^{bc}	95.04 ± 0.05 ^{bc}	94.94 ± 0.07 ^{bc}	94.87 ± 0.04 ^{bc}	94.64 ± 0.07 ^b
a^*	0.37 ± 0.03 ^a	0.36 ± 0.01 ^a	0.42 ± 0.06 ^{ab}	0.49 ± 0.04 ^{ab}	0.54 ± 0.03 ^{bc}	0.58 ± 0.02 ^c
b^*	10.03 ± 0.04 ^a	10.26 ± 0.20 ^{ab}	10.44 ± 0.15 ^{abc}	10.53 ± 0.02 ^{bc}	10.67 ± 0.01 ^{bc}	10.78 ± 0.11 ^c
Molds (CFU g ⁻¹)	00.00 ± 0.00 ^a	00.00 ± 0.00 ^a	00.00 ± 0.00 ^a	00.00 ± 0.00 ^a	00.00 ± 0.00 ^a	00.00 ± 0.00 ^a
Yeasts (CFU g ⁻¹)	00.00 ± 0.00 ^a	00.00 ± 0.00 ^a	00.00 ± 0.00 ^a	00.00 ± 0.00 ^a	00.00 ± 0.00 ^a	00.00 ± 0.00 ^a

All analyses were in triplicate. Data are average values ± standard deviations.; ^A Different superscript lowercase letters within a row indicate a significant difference ($p < 0.05$).

The moisture content of the product remained stable (2.62% w/w; Table 4) over the full duration of storage. Similarly, the water activity remained within 0.23–0.24 throughout (Table 4). NPV powder maintained its initial water activity level throughout the study. The pH values were quite stable over the entire storage period (Table 4), although marginally higher in samples stored for 90 days and longer. A stable acidity (pH) and water activity level are particularly important in ensuring a long shelf-life of a food product. Stable values of these parameters indirectly suggest a lack of microbial growth [38].

The microbial count of the product did not change, and the product was essentially sterile (Table 4). Absence of microbial growth was consistent with the low water activity (≤ 0.24 ; Table 4) of the product, as microbes do not proliferate if the water activity is less than 0.5 [10].

4. Conclusions

The NPV powder with the highest desirability could be produced using a drying air inlet temperature of 170 °C in combination with a nipa palm vinegar feed containing 15% w/v maltodextrin DE10. This product (NPVp-3) had nearly 47-fold higher vitamin C content than a commercial vinegar powder but only ~43% of the acidity (acetic acid)

compared to the commercial product. The NPVp-3 powder was stable at room temperature during 180-days of storage. The excellent stability was explained by the low water activity ($a_w = 0.23$) of the product, indicating highly effective drying by the method used. A relatively high content of the highly heat-sensitive vitamin C in the product indicated that the spray-drying treatment minimized damage to the product due to the short contact time (residence time in the drying chamber was less than 42 s) of the product with the drying air. The NPVp-3 was judged to be a good food seasoning and beverage ingredient.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/fermentation8060272/s1>. Supplemental material for this article is available at: Table S1. The levels of the various factors in experimental design. Table S2. The factor values used in producing the various powdered NPV products. Table S3. The constraints used in estimating the overall desirability. Table S4. The statistical models and comparison of predictions with the measured values.

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Data Availability Statement: All data relating to this study are included within this article. Data will be made available on reasonable request.

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