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Dehumidified Air-Assisted Spray-Drying of Cloudy Beetroot Juice at Low Temperature

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Abstract: This paper discusses the physicochemical properties of powders obtained by spray drying of cloudy beetroot juice, using dehumidified air in variants with or without carriers. The inlet air temperature was 130 °C or 90 °C, and the addition of the carriers was at a ratio of juice to carrier solids of 3:2. In the obtained powders, the following physicochemical properties were determined: water content and water activity, apparent density, loose and tapped density, porosity, flowability, particle size and morphology, and the content and retention of betalains. It was possible to dry cloudy beetroot juice without the use of carriers at low temperatures (90 or 130 °C). The 100% beetroot powders were characterized by satisfactory physicochemical properties, often better than those with carriers (including lower hygroscopicity and higher color saturation and yield). A lower loss of betalains was found for the powders with the addition of carriers. The best process yields were obtained for the powder without carriers at 130 °C and 90 °C.

Keywords: spray-drying; dehumidified air; glass transition; powder recovery; betalains; beetroot

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1. Introduction

Beetroot (*Beta vulgaris* L.) is a part of the *Chenopodiaceae* family and is considered as one of the ten vegetables with the best antioxidant properties because of its high polyphenol content (50–60 mol/g solids) [1,2]. Beetroot is mostly recognized for its characteristic color that it owes to betanins, which belong to the group of purple betacyanins. Betacyanins, in turn, belong to the group of betalaines, which also include yellow betaxanthins, the most important of which are vulgaxanthins [3,4]. Betacyanins are susceptible to high temperatures, which suggests that the application of low-temperature or non-thermal methods will result in products with high contents of these dyes. Moreover, during heat treatment, red dyes can turn into yellow dyes and there is also a high probability of Maillard reaction products being produced, which result from a series of chemical reactions that occur most often under the influence of heat between amino acids and reducing sugars. [5].

In the food industry, betalaines are mostly used as a natural dyes; however, their use is limited because of their susceptibility to high temperature and light. Currently, the main source of commercially used betalains dyes is beetroot [6]. Dyes are commonly used in the form of powders and the most popular method to obtain food powders is spray-drying, due to the short, single-step operation that enables the production of powders from liquid feeds. Moreover, this method is suitable for materials that may lose their properties due to high temperatures following brief contact with hot air [7]. However, during spray-drying there is a possibility of stickiness, which may occur when the drying materials are rich in low-molecular-weight sugars (glucose, fructose) because of their low glass transition temperatures (T_g). There are many approaches that make it possible to avoid this phenomenon, but the most common is the addition of carriers of high T_g [8]. However, the growing trend of "clean labels" among consumers makes it necessary to seek different approaches. One that is currently gaining more recognition in the literature is the use of dehumidified air as a drying medium. Dehumidified air makes it possible to lower the drying temperature and, as a consequence, prevent the stickiness problem. This method makes it possible to decrease the addition of carriers as well and, at the same time, enhance the product yield and powder properties. Jedlińska et al. (2019) successfully spray-dried rapeseed and honeydew honey at inlet/outlet temperatures of 75/50 °C with such a dehumidified air-assisted method [9]. It was possible to reduce carrier content to 20% thanks to the use of dehumidified air, while the addition of carriers in most honey powders is about 50%. Chasekioglou et al. (2017) spray-dried olive mill wastewater using dehumidified air at an inlet temperature of 110–130 °C and they observed significantly higher product recovery (87–94%) than with traditional spray-drying (<3%) [10]. Powders obtained using dehumidified air are characterized by lower water content and higher bulk density and their particles have smoother surfaces. Moreover, lowering drying temperatures reduces the risk of degradation of thermolabile compounds, such as betalaines [11]. Samborska et al. (2019) obtained honey powder with undeteriorated biological activity through the application of spray-drying with low-humidity air at 75/50 °C inlet/outlet temperatures [12].

The aim of the study was to develop 100% powders (without carriers) from cloudy beetroot juice using dehumidified air-assisted spray-drying at low temperatures (inlet air temperature: 90 °C and 130 °C). The research included the determination of the powder recovery and of selected physicochemical properties of the obtained powders, which were compared for powders without carriers and those with carriers (maltodextrin, milk powder, nutriose, and kleptose).

2. Materials and Methods

2.1. Materials

Beetroot (*Beta vulgaris* L.) of the Wodan variety was purchased from a local market in Warsaw (Poland) and stored in a cold place at 4–5 °C. Skimmed milk powder (M) (Mlekovita, Błonie, Poland), nutriose FM06 (N) (Roquette, Lestrem, France), maltodextrin DE15 (MD) (Pepees, Łomża, Poland), and kleptose (K) (Roquette, Lestrem, France) were applied as carriers. The chemicals used for betalain determination were obtained from Sigma Aldrich.

2.2. Preparation of Juice and Feed Solutions

Cloudy juice (J) was prepared by squeezing beetroot with a NS-621CES squeezer (Kuvings, Daegu, Korea). The juice had an extract content of 8 °Bx. The symbols applied to describe the compositions of the seven variants of feed solution are presented in Table 1. Three variants (J130, J90, and J90c) were spray-dried without the carrier. The other four (LM130, JN130, JMD130, and JK130), with carriers, were prepared through the addition of carriers at a weight ratio of juice to carrier solids of 3:2. Among the variants without the carrier, one variant (J90c) was spray-dried after concentration of the juice to 50 °Bx on a Rotavapor R-124 evaporator (BUCHI, Flawil, Switzerland) at a bath temperature lower than 70 °C and with an initial pressure of 130 hPa (antifoaming agent was used).

2.3. Spray-Drying

A Mobile Minor Laboratory spray-dryer (GEA, Skanderborg, Denmark) was used. Feed solutions were pumped at a feed ratio speed of 0.20 $mL \cdot s^{-1}$, inlet/outlet temperatures were respectively 130/80 °C or 90/55 °C, depending on the variant (as presented in Table 1), and the disc rotation speed was 26,500 rpm. The spray-dryer was equipped with an air dehumidification system (cooling unit and condensation–adsorption unit), as described by Jedlińska et al. (2019), that made it possible to perform drying at an inlet air humidity of 1 $g \cdot m^{-3}$ [9].

After drying, the amount of obtained powder was measured and the powder recovery (%) was calculated as the ratio of solids content in the powder and in the feed solution.

Table 1. Variants of cloudy beetroot juice spray-dried with or without carriers at different temperatures.

Variant	Carrier	Inlet/Outlet Air Temperature (°C)
J130	No carrier added	130/80
JM130	Milk powder	130/80
JN130	Nutriose	130/80
JMD130	Maltodextrin	130/80
JK130	Kleptose	130/80
J90	No carrier added	90/55
J90c	No carrier added	90/55

2.4. Powder Characterization

2.4.1. Morphologies of the Particles

The powder particles' morphologies were described based on pictures taken with a TM3000 scanning electron microscope (Hitachi, Tokyo, Japan; magnification 1000×). Prior to analysis, samples were prepared by placing them on double-sided sticky tape and metalizing their surfaces by coating them with a layer of gold using a Cressington 108auto automatic coater (EO Elektronen-Optik-Service GmbH, Dortmund, Germany).

2.4.2. Particle Size Distribution

The sizes of the particles of the powders were examined with laser diffraction using an 1190 instrument (CILAS, Orleans, France). Measurements were made in the wet dispersed phase in ethyl alcohol with an obscuration of 5–10%.

2.4.3. Water Content and Water Activity

Water content (WC) was determined with the oven method, with 1 g of powder dried at 105 °C/4 h. Water activity (a_w) was measured with a Hygro Lab C1 (Rotronic, Bassersdorf, Switzerland) at 25 °C.

2.4.4. Glass Transition Temperature

Modulated differential scanning calorimetry (MDSC) was performed on a Q200 differential scanning calorimeter (TA Instrument, New Castle, DE, USA) to determine the glass transition temperature (T_g). Beforehand, powders were dehydrated at 50 °C for 24 h at reduced pressure in order to remove residual moisture. The cell was purged with 50 mL·min^{-1} dry nitrogen and calibrated to baseline on an empty oven and for temperature using standard pure indium. An empty, sealed aluminum pan was used as a reference test. Then, 10–15 mg of the samples was hermetically sealed in the aluminum pans (volume: 30 μL) and scanned from −40 to 120 °C at a constant cooling rate of 1 °C/min, with an amplitude of ±1 °C and a 60 s period of modulation. Curves were analyzed with respect to the total, reversible, and non-reversible heat flow (Górska et al., 2017). The glass transition was estimated as the midpoint of a vertical shift in the reversing transition curve using TA Instruments Universal Analysis software.

2.4.5. Hygroscopicity

The hygroscopicity of powders was measured as the gain in weight of about 1 g sample incubated at a relative humidity of 75% (over saturated NaCl solution). Samples were weighed before placing them in the desiccator and again after 1, 2, 5, 24, 48, and 72 h. Hygroscopicity was presented graphically as the increase of water content (expressed as u[g/g solids]) in a sample during the measurement.

2.4.6. Loose Bulk Density and Tapped Density

For loose bulk density (D_L), an empty cylinder of 25 mL was weighed and then weighed again with 25 mL of the powder. For tapped density (D_T), the measurement was made by tapping the sample 100 times in a 25 mL cylinder on a shock volume meter STAV 2003/Engelsmann AG (Ludwigshafen, Germany).

2.4.7. Apparent Density

The measurement of apparent density (D_{ap}) was made using a Stereopycnometer (Quantachrome—Boynton Beach, FL, USA) helium pycnometer.

2.4.8. Bulk Porosity and Flowability

Porosity (ε_L) was calculated as the ratio of difference of the apparent density and loose bulk density to the apparent density. Flowability was expressed as a Hausner's ratio (HR)—the ratio of the bulk tapped density to the bulk loose density.

2.4.9. Color

The color measurement was carried out using a Minolta CR5 (Japan) chromatometer with the measurement geometry d/0° in the color system CIE Lab. Color saturation was calculated as chroma ($C^* = \sqrt{a^2 + b^2}$). The parameter $H^* = \text{atang}(b^*/a^*)$ was also calculated. The color of the powders was illustrated using an appropriate program (http://hclwizard.org/hclcolorpicker/, accessed on 25 May 2021) and based on the H*, L*, and C* parameters.

2.4.10. Betalains

The betalain measurement was carried out for the cloudy juice and obtained powders using Nilsson's method. A sample of juice was 0.5 g, while the sample of powder was weighed so that its mass corresponded to the amount of dry substance contained in 0.5 g of raw material. Then, the sample was filled with buffer and shaken in a Heidolph Multi Reax Vortex Mixer laboratory shaker for 10 min at maximum speed. The sample was placed in a centrifuge (Sigma Laboratory Centrifuge model 4–15) for 5 min at 600 rpm. The solution was then filtered and its absorbance was measured at 476, 538, and 600 nm in a spectrophotometer (Thermo Spectronic Helios Gamma—Thermo Fisher Scientific, Walthan, MA, USA) using a phosphate buffer solution as a standard. The retention of betalain was calculated as taking into account the betalain content in the dry matter of the juice as 100%. The results were expressed as the contents of betalains (BL), betanins (BN), and vulgaxanthins (V). Moreover, the retention for obtained cloudy beetroot juice powders was calculated by taking in to consideration the betalain content in the juice before drying and the dry substance content of the juice and powders.

2.5. Statistical Methods

The results were analyzed using Statistica 13.1. One-way analysis of variance (ANOVA) was performed at a significance level of $\alpha = 0.05$ and homogeneous groups were determined using Tukey's test. Pearson correlation analysis and correlation coefficient (r) calculation were performed for two groups of properties: (1) powder recovery, glass transition temperature, water content, water activity, median diameter, bulk loose density, bulk tapped density, Hausner ratio, apparent density, and loose bed porosity; and (2) color parameters and betalain content. Hierarchical cluster analysis was done separately for the same two groups of parameters.

3. Results and Discussion

3.1. Powder Recovery

The highest powder recovery (R_P) was observed for the variants obtained without a carrier at the temperatures of 130 °C and 90 °C (J130 and J90), while the lowest was observed for the powder with milk powder as a carrier (JM130) (Table 2). The obtained results showed no significant influence from temperature, the presence of the carrier and its type, or the concentration on R_P. Goula and Adamopoulos [2005], when spray-drying tomato pulp with the use of dehumidified air, achieved an R_P between 36.62 and 65.86% [13]. The authors noted that, thanks to the use of dehumidified air, lowering the outlet temperature allowed them to obtain a higher R_P than drying at a higher temperature. The reduced humidity of the drying medium and its temperature resulted in the formation of particles with a durable, solid surface and a fewer particles sticking to the dryer walls. However, the R_P obtained in our work was higher (from 55.2 $\pm$ 3.6 to 90.0 $\pm$ 11.4%), which could be explained by the characteristics of the raw material and the cellulose content, which probably acted as a carrier.

3.2. Morphologies of Particles

The particle morphologies of the variants of the powders with the addition of different carriers (JN130, JMD130, and JK130) did not show significant differences (Figure 1). The particles had the shape typical for powders with carrier additions: they were spherical and smooth and some of them had shrunk. Shrinkage is typically a result of a large loss of water followed by the cooling of the surface of the particles [14]. On the other hand, particles of powders dried at the reduced temperature without carriers (J90 and J90c) were significantly different in appearance. They were spherical and without shrinkage compared to the other powders (Figure 2). Moreover, when comparing the J130 and J90 variants, a significant effect of temperature on the particle morphology was observed—agglomerates were obtained at a higher temperature and single particles at a lower temperature.

3.3. Particles Size Distribution

The particle size distribution and the cumulative particle size distribution are presented in Figures 2 and 3, while the mean particle diameter values are presented in Table 2. Particle size distributions with diameters ranging from 3 to 100 µm were observed (Figure 2). The largest particles were observed for the variants JM130, J130, J90, and J90c. There was a statistically significant influence resulting from the presence of the carrier on the particle size. The powders with the addition of nutriose, maltodextrin, and kleptose (JN130, JMD130, and JK130) had similar and the smallest particle sizes, which was also confirmed by the mean diameter values presented in Table 2. According to the data presented in Table 2, there was no statistically significant influence from the concentration of the feed solutions (comparing the J90 and J90c powders) and the temperature (comparing the J130 and J90 powders) on the particle size.

The powders obtained by Janiszewska (2014), who spray-dried the juice of beetroot with maltodextrin and acacia gum, had an average particle diameter ranging from 7.6 $\pm$ 0.8 µm to 12.8 $\pm$ 0.1 µm [2]. However, the analyzed powders were characterized by larger particle sizes, which likely mainly resulted from the fact that cloudy juice was used instead of concentrate, which resulted in an increase of substances insoluble in water, including cellulose. Other drying conditions (dehumidified air, lower inlet temperatures) and other types of carriers at different concentrations were also used.

Table 2. Powder recovery (R_P) and physical properties of obtained powders: glass transition temperature (T_g), water content (WC), water activity (a_w), median diameter (D_{50}), bulk loose density (D_L), bulk tapped density (D_T), Hausner ratio (HR), apparent density (D_{ap}), and loose bed porosity (ε_L) (abbreviations of variants explained in Table 1).

Variant	Rp (%)	T_g (°C)	WC (%)	a_w	D_{50} (µm)	D_L (g/cm^3)	D_T (g/cm^3)	HR	D_{ap} (g/cm^3)	ε_L (%)
J130	90.0 ± 9.4^{b}	58.4 ± 0.1^{a}	4.0 ± 0.6^{a}	0.169 ± 0.004^{c}	13.2 ± 0.3^{b}	$0.44 \pm 0.05^{a,b}$	0.61 ± 0.06^{b}	1.37 ± 0.03^{cd}	$1.57 \pm 0.04^{b,c}$	$71.7 \pm 3.1^{b,c}$
JM130	55.2 ± 3.6^{a}	$65.6 \pm 1.6^{a,b}$	6.9 ± 1.0^{b}	0.142 ± 0.013^{b}	17.1 ± 0.7^{b}	0.41 ± 0.04^{a}	0.50 ± 0.05^{a}	1.21 ± 0.05^{a}	1.49 ± 0.02^{a}	$72.1 \pm 2.4^{b,c}$
JN130	$71.6 \pm 3.0^{a,b}$	$68.5 \pm 2.7^{b,c}$	4.1 ± 0.6^{a}	0.107 ± 0.004^{a}	9.3 ± 0.3^{a}	0.61 ± 0.03^{c}	0.78 ± 0.01^{c}	$1.27 \pm 0.05^{a,b}$	1.55 ± 0.01^{b}	60.3 ± 1.6^{a}
JMD130	$76.1 \pm 1.7^{a,b}$	70.0 ± 0.3^{c}	3.7 ± 0.6^{a}	0.113 ± 0.005^{a}	9.1 ± 0.1^{a}	0.58 ± 0.02^{c}	0.73 ± 0.01^{c}	$1.26 \pm 0.04^{a,b}$	1.54 ± 0.00^{b}	62.5 ± 1.1^{a}
JK130	$65.3 \pm 11.9^{a,b}$	$69.2 \pm 1.4^{b,c}$	3.6 ± 0.3^{a}	0.148 ± 0.005^{b}	9.2 ± 0.1^{a}	0.57 ± 0.03^{c}	0.74 ± 0.01^{c}	$1.31 \pm 0.06^{b,c}$	$1.56 \pm 0.01^{b,c}$	63.8 ± 1.7^{a}
J90	89.8 ± 2.3^{b}	$65.3 \pm 2.0^{b,c}$	3.5 ± 0.6^{a}	0.179 ± 0.008^{c}	14.1 ± 1.3^{b}	0.42 ± 0.01^{a}	0.60 ± 0.01^{b}	1.44 ± 0.03^{d}	1.60 ± 0.04^{c}	73.9 ± 1.1^{c}
J90c	$63.5 \pm 13.5^{a,b}$	$63.7 \pm 0.6^{a,b}$	3.9 ± 0.6^{a}	0.202 ± 0.010^{d}	15.6 ± 1.0^{b}	0.49 ± 0.03^{b}	0.64 ± 0.05^{b}	$1.29 \pm 0.05^{a,b,c}$	1.65 ± 0.01^{d}	70.1 ± 2.0^{b}

[a–d] the differences between mean values with the same letters in columns were not statistically significant ($p < 0.05$).

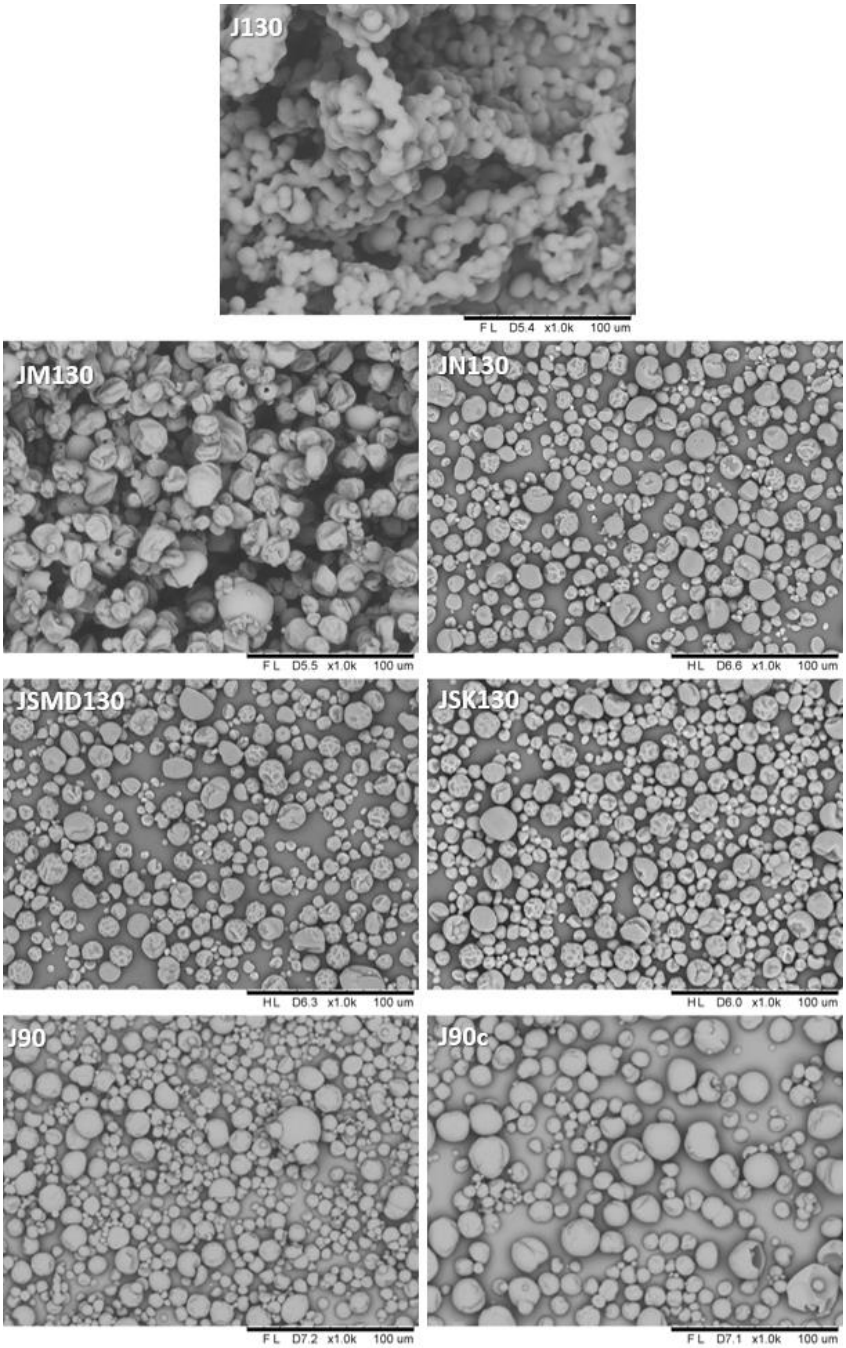

Figure 1. SEM microphotographs of cloudy beetroot powders (abbreviations of variants explained in Table 1).

3.4. Water Content and Activity

The water content (WC) of most powders ranged from 3.5 to 4.1% (Table 2). Powder based on milk (JM130) was characterized by significantly higher water content 6.9%. This phenomenon could have been due to the ability of milk proteins to form a film on the surface of liquid particles, which prevented effective evaporation of water. Bhusari et al. (2014) found similar conclusions after drying tamarind pulp with whey protein concentrate as a carrier—with the increase in the share of WPC, an increasing WC of the powders was observed [15]. Muzaffar and Kumar (2016) also noticed a similar effect, obtaining tamarind pulp powder with soy protein isolate; WC also increased with the increased protein content [16]. The typical WC for powder powders obtained by spray-drying is not more than 5% [17], which indicates that, for most variants, satisfactory results were obtained. Therefore, it can be concluded that the powders without the addition of carriers and dried at a lower temperature had appropriate quality.

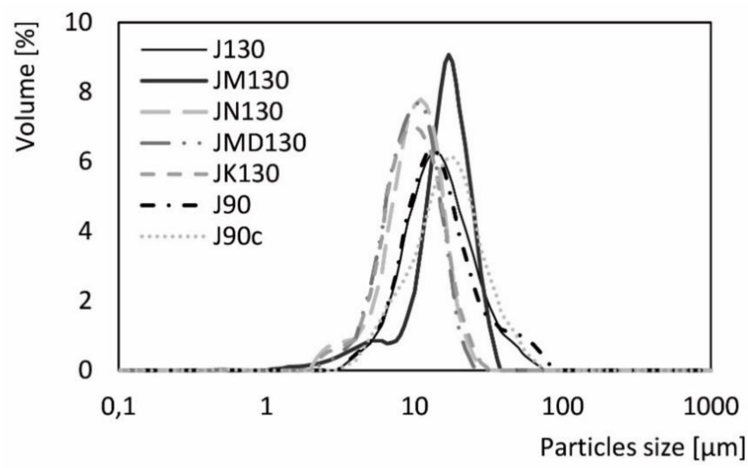

Figure 2. Particle size distribution of cloudy beetroot juice powders (abbreviations of variants explained in Table 1).

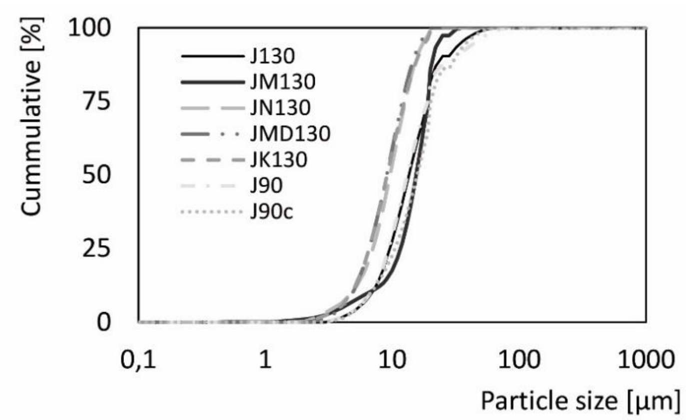

Figure 3. Cumulative particle size distribution of cloudy beetroot juice powders (abbreviations of variants explained in Table 1).

The obtained powders were characterized by water activity (a_w) in a range from 0.107 ± 0.004 to 0.202 ± 0.010. The lowest a_w was observed in the case of powders obtained from juices with added carriers. Powders based on nutriose and maltodextrin (JN130 and JMD130) were characterized by significantly lower a_w compared to powders with skim milk or kleptose (JM130 and JK130). Despite the high water content (6.9 ± 1.0%) in the milk powder variant (JM130), the water activity was low (0.142 ± 0.013). It can be concluded that the availability of this water was small and water was structurally bound. Powders with a_w lower than 0.3 are considered to be microbiologically and chemically safe, so the powders had a proper a_w [7]. Powders without the addition of carriers were characterized by a statistically significantly higher a_w value than powders with the addition of carriers. A significantly higher value of a_w was obtained for powder from concentrated juice (J90c). The likely reason was the higher concentration of the drying solution. With regard to the other powders without carriers (J90 and J130), no differences were found; the drying temperature (90 or 130 °C) did not significantly affect the value of a_w.

3.5. Glass Transition Temperature

The glass transition temperatures (T_g) of the beetroot powders were presented in Table 2; the temperatures ranged from 58.46 to 70.06 °C. The value of T_g is an indicator of the quality and storage stability of food powders. Flores-Mancha et al. (2020) investigated the influence of carriers (maltodextrin and inulin) on the encapsulation process of beetroot powders [18]. T_g values of 18.34 °C, 61.63 °C, and 27.59 °C were presented for spray-dried beetroot powder without a carrier, with maltodextrin, and with inulin, respectively.

The powder produced with maltodextrin (JMD 130) showed a higher T_g of 70.06 °C. The T_g of maltodextrin is depended on the dextrose equivalent (DE), which is related to the decrease in the molecular weight that decreases the T_g [19]. According to Sobulska and Zbiciński (2020), maltodextrin with a DE from 36 to 5 is characterized by a T_g in a range from 100 to 188 °C [20]. The T_g of skimmed milk powder at 58 °C (water activity: 0.1) and 34 °C (water activity: 0.2) was obtained by Jouppila and Roos (1994) [21]. Higher values for the T_g of skimmed milk powder at 64.3 and 53.0 °C at the same a_w were presented by Shrestha et al. (2007) [22]. The differences could have been caused by other parameters involved the drying process and the chemical composition of skimmed milk powder. The addition of nutriose (T_g: 15 °C) and kleptose to beetroot powder as a carrier resulted in an increase of the T_g of beetroot powder to 68.52 °C and 69.22 °C, respectively [20]. Higher values for T_g were found in for powders with the addition of carriers.

The use of lower inlet/outlet air temperatures makes it possible to obtain pure beetroot powders with higher T_g. Bhandari and Howes (1999), Balasubramanian et al. (2016), and Fan and Roos (2017) have conducted extensive research on T_g phenomena [23–25]. The great problem during spray-drying is obtaining food powders from a material characterized by a low T_g. The structural changes in food powders, like increased stickiness and caking susceptibility, depend on the differences between the T_g of the product and the particle surface temperature during spray-drying [23–25]. The outlet air temperature is even 10–20 °C higher than the temperature of the particle surface during spray-drying. At temperatures 10–20 °C higher than the T_g, the material state changes from amorphous to rubbery. It can be concluded that the temperature of the particle surface during drying should not reach 10–20 °C above the T_g [26,27]. Research on various carriers used in spray-drying has been undertaken, including maltodextrin, gum arabic, and inulin. Sobulska and Zbiciński (2020) investigated honey and fruit juice powders, Flores-Mancha (2020) studied beetroot powder, and Goula and Adamopoulos (2008) examined tomato pulp [18,20,27]. The T_g of beetroot powder produced with different carriers explains the good quality of the powders obtained at 55 and 80 °C outlet temperatures.

3.6. Hygroscopicity

The ability of powders to absorb water during their storage causes the deterioration of their properties because water acts as a plasticizer, which in turn leads to clumping or sticking [28]. The changes in water content during the measurement are presented in Figure 4. Most carrier-based powders were characterized by higher hygroscopicity than that obtained without carriers. Powders with carriers—nutriose, maltodextrin, and milk powder dried at 130 °C (JN130, JMD130, and JM130)—were characterized by significantly higher hygroscopicity. The powders obtained without the use of carriers were characterized by the lowest hygroscopicity: powder obtained at 90 °C from concentrated juice (J90c) and powder obtained from juice at 130 °C (J130). At the same time, analysis of powders without carrier additions showed that there was no significant effect from the drying temperature (90 or 130 °C) or the form of the drying solution (juice, concentrated juice) on the hygroscopicity. The results obtained were satisfactory, as powders without carriers and dried at a reduced temperature (90 °C) had lower hygroscopicity than variants dried with carriers at a higher temperature (130 °C). The exception was the powder based on kleptose (JK130), which had hygroscopicity similar to powders without the addition of carriers. Bazaria and Kumar (2016) found that the water content affects the hygroscopicity of a material [29]. The lower the water content is, the higher the hygroscopicity, as the difference in concentration between the product and the surrounding environment is greater, which gives a greater driving force to the process. However, the results obtained did not confirm this relationship, as powders with low water content (J90c, J130) were characterized by the lowest hygroscopicity. Tonon et al. (2009) concluded that particle size may affect the hygroscopicity of the obtained powders [30]. Larger particles have a smaller range of exposed surface on which water from the air can be adsorbed. Therefore, powders with large particle sizes are characterized by lower hygroscopicity. The obtained results confirmed this relationship, as the variants J90c and J130, which were the least hygroscopic, were characterized by large average particle diameters (Table 2).

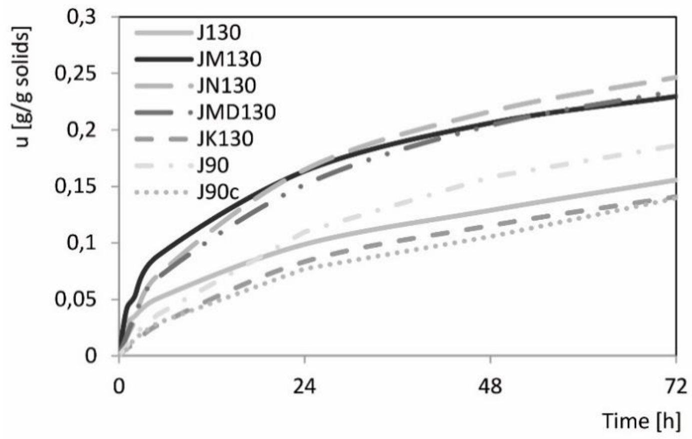

Figure 4. Water vapor adsorption kinetics of obtained cloudy beetroot juice powders (abbreviations of variants explained in Table 1).

3.7. Apparent Density

The apparent density (D_{ap}) of the obtained powders ranged from 1.49 ± 0.02 g/cm^3 to 1.65 ± 0.01 g/cm^3 (Table 2). The highest D_{ap} was found for powders without carriers which were obtained from concentrated juice at the reduced temperature (J90c). The lowest D_{ap} was recorded for powders with the addition of powdered milk (JM130). Janiszewska and Witrowa-Rajchert (2007), with regard to the microencapsulation of rosemary aroma, concluded that the concentration of the feed solution affects the D_{ap} value of the obtained powders [31]. The higher the concentration, the greater D_{ap} is, because the intramolecular porosity is lower. A similar statement can be extended to the cases of the obtained beetroot powders. It can also be seen that increasing the temperature to 130 °C caused a decrease in the D_{ap} of the obtained powders compared to the variants dried at lower temperatures (J90 and J90c). Kwapińska and Zbiciński (2006) concluded that the carrier particles often contain gas bubbles, and their presence could often be a result of desorption from the feed solution or absorption during atomization [32]. This phenomenon causes an increase in the volume of trapped gases, which translates into a decrease of D_{ap}. The obtained results confirmed this dependence, as the powders with the addition of carriers were characterized by the lowest D_{ap}. Moreover, the variant dried at 130 °C without the addition of a carrier (J130) did not differ significantly from powders with carriers (kleptose, maltodextrin, and nutriose), which may have indicated a sufficient amount of cellulose and its ability to behave as a carrier. The presence of the carrier and its type, the concentration of the raw material, and the temperature all had a statistically significant influence on the parameter value.

3.8. Bulk Density

The highest D_L was achieved in powders with the addition of nutriose, maltodextrin, and kleptose carriers and was, respectively, 0.61 ± 0.03 g/cm^3, 0.58 ± 0.02 g/cm^3, and 0.57 ± 0.03 g/cm^3 (JMD130, JN130, and JK130). The lowest D_L was obtained in the powder with milk powder (JM130—0.41 ± 0.04 g/cm^3) and the powders dried at a lower temperature without carriers (J90—0.42 ± 0.01 g/cm^3). Only the variant with milk powder differed significantly from other powders with the carrier addition. The low D_L of the powders with the milk addition could have been the result of the creation of a liquid film on the surface of the feed solution molecules, which prevented them from sticking together. A similar phenomenon was observed by Suhag and Nanda (2015), who dried honey using whey protein concentrate (WPC) as a carrier [33]. With the increase in the protein addition in the feed solution, the D_L of the obtained powders decreased. A higher value of D_L translates into less air in the spaces between powder particles, which results in a lower risk of oxidation (loss) of the valuable ingredients contained in the beetroot powder. Thus, it can be stated that the possibility of degradation of labile ingredients during storage was most limited for the powders with the addition of nutriose, maltodextrin, and kleptose. The particle size affects the D_L. The larger the particle size, the lower D_L is [34]. The powders with large particle diameters were also distinguished by a low D_L: J130, JM130, J90, and J90c. There was a significant correlation (Pearson correlation coefficient, r = −0.884 and r = −0.926; Table 3) between the particle size and the values of D_L and D_T. It can be concluded that the presence of the carrier and its type, the drying temperature, and the concentration of the feed solution all had a statistically significant influence on the D_L.

Table 3. Pearson correlation coefficients (r) between the powder recovery (R_P), glass transition temperature (T_g), water content (WC), water activity (a_w), median diameter (D_{50}), bulk loose density (D_L), bulk tapped density (D_T), Hausner ratio (HR), apparent density (D_{ap}), and loose bed porosity (ε_L) of the beetroot powders; asterisks indicate statistically significant correlations ($p < 0.05$).

	T_g	WC	a_w	D_{50}	D_L	D_T	HR	D_{ap}	ε_L
R_P	−0.400	−0.604	0.152	−0.201	−0.168	0.078	0.838 *	0.298	0.220
T^g		−0.089	−0.649	−0.594	0.697	0.579	−0.415	−0.313	−0.718
WC			−0.152	0.597	−0.446	−0.667	−0.617	−0.662	0.303
a_w				0.657	−0.643	−0.507	0.522	0.743	0.765 *
D_{50}					−0.884 *	−0.926 *	0.017	0.127	0.874 *
D_L						0.956 *	−0.344	−0.048	−0.982 *
D_T							−0.061	0.136	−0.905 *
HR								0.547	0.447
D_{ap}									0.226

3.9. Bulk Porosity

The bulk porosity (ε_L) of the obtained powders ranged from 60.33 ± 1.66% to 73.96 ± 1.13% (Table 2). The highest ε_L values were obtained for the powders without the addition of a carrier (J90 and J130) and for the powder with the addition of powdered milk (JM130). No statistically significant influence of the drying temperature on the ε_L of the powders was found. The lowest ε_L was found for powders dried at a higher temperature with the addition of the carriers nutriose, maltodextrin, and kleptose (JN130, JMD130, and JK130). Similar values (63.3 ± 0.7 to 72.5 ± 0.92%) were obtained by Jedlińska et al. (2019), who dried rapeseed and honeydew honey using a traditional spray-drying method and with dehumidified air [9]. It can be concluded that the powders with the largest mean particle diameters had at the same time the highest ε_L, which was confirmed by the statistically significant Pearson coefficient (r = 0.874, Table 3). The higher the ε_L of the material, the more air there is in the intermolecular spaces, which influences the degradation of valuable ingredients and reduces the storage stability of the powders [35]. Therefore, the powders without carriers (J90 and J130) and those with the addition of powdered milk (JM130) were the most durable and the best from the point of view of storage. The larger particle sizes resulted in lower bulk density and higher porosity. The powders with low loose bulk densities were characterized by high ε_L (J130, JM130, J90, and J90c), which was confirmed by the statistically significant Pearson coefficient (r = −0.982, Table 3). Similar conclusions were reached by Caparino et al. (2012), who studied mango powders—the powders with large particle sizes had both a low bulk density and a high porosity [36]. A correlation between ε_L and a_w was also observed and confirmed by a Pearson coefficient of r = 0.765 (Table 3). It can be concluded that the presence of the carrier and its type had a significant influence on the ε_L of the obtained powders.

3.10. Flowability

The flowability of powders was determined with the Hausner ratio—a value from 1 to 1.10 describes powders of good flowability, 1.11 to 1.25 describes powders of medium flow, 1.26 to 1.4 describes cohesive powders, and values above 1.4 indicate very cohesive powders that form a coherent structure [37]. The obtained powders could be classified as powders of medium flowability (JM130), cohesive powders (J90c, J130, JN130, JMD130, and JK130), and very cohesive powders (S90) (Table 2). Sahni and Shere (2017) obtained beetroot pulp powder with an HR value of 1.30, which is similar to the values obtained in this study [38]. The best flowability was observed for the powder with the addition of milk (SM130), which may have been a result of the ability of proteins to form a film on the surface of the feed solution particles (preventing them from sticking together) and because of the particle size, which was the largest. Tze et al. (2012) found that pitaya fruit powders with smaller particle sizes were characterized by lower flowability [39]. Samborska et al. (2015) noted that the addition of a protein carrier (sodium caseinate) improved the flowability of the honey powder obtained [40]. Landillon et al. (2008) explained that larger particle sizes translate into a smaller number of points at which particles can combine and interact with each other, which results in the lower cohesiveness of the powders and thus better flow properties [41]. Among the powders with carriers, the powders with the addition of kleptose (J130) were characterized by worse flowability. When comparing the powders that were dried at the reduced temperature, a significant influence from the concentration of the juice fed for drying was noticed—the powder obtained from the concentrated juice had better flowability. There was a positive correlation between the flowability and powder recovery—the lower the powder recovery, the better the flowability (r = 838, Table 3). It can be concluded that the temperature, concentration, and the presence of carriers and their type had a statistically significant influence on the flowability of the obtained powders.

3.11. Color

The L* brightness of the obtained powders differed significantly ($p \leq 0.05$), creating four homogeneous groups (Table 4). Powders dried at the reduced temperature without carriers were characterized by the highest brightness (J90 and J90c), and all the variants with the addition of carriers had the darkest colors (JM130, JN130, JMD130, and JK130). Based on the obtained results, it can be concluded that the effects of temperature and the presence of the carrier were significant; however, the type of carrier used did not contribute to the change in brightness. The highest proportions of the red color a* were observed in variants dried without carriers at 130 and 90 °C (J130 and J90) and the lowest in powders that were obtained from concentrated juice at 90 °C (J90c) (Table 3). Powders with the addition of carriers dried at 130 °C did not differ significantly from each other ($p \leq 0.05$). It can be concluded that the temperature, the presence of the carrier, and the concentration had significant impacts on the a* color parameter. The proportion of red color is directly related to the content of red pigments (betanins). The low value of the a* parameter for the J90c variant could have been related to degradation during concentration, but this was not confirmed by the results obtained in the determination of the betanin content, as the betanin content in this case was in fact high (Table 5). However, in the case of the J130 and J90 powders, the betanin content in the final product was confirmed by a high proportion of red color. The value of the b* parameter (Table 4), which characterizes the yellow content, was the highest in the powders dried at a high temperature without the addition of carriers (J130). As in the case of betanins and the red color, the content of vulgaxanthin has a significant influence on the yellow color. The higher the vulgaxanthin content, the greater the value of the b* color parameter. J130 powders, which had the highest proportion of yellow color, were characterized by high vulgaxanthin content (Table 5). On the basis of the obtained results, it can be concluded that the temperature and the presence of the carrier and its type did not significantly affect the yellow color in the obtained powders. However, a significant influence of the juice concentration on the b* color parameter was noted. The powder obtained from the concentrated juice was characterized by a lower proportion of yellow color and lower vulgaxanthin content.

Table 4. Color parameters of cloudy beetroot juice powders (abbreviations of variants explained in Table 1).

Variant	L*	a*	b*	C*	H*	Color*
J130	32.3 ± 0.4 [b]	46.6 ± 0.6 [c]	9.8 ± 0.3 [b]	47.6 ± 0.7 [d]	0.2 ± 0.0 [c]	
JM130	36.4 ± 0.5 [c]	44.7 ± 0.5 [b]	5.4 ± 0.4 [a]	45.0 ± 0.6 [b,c]	0.1 ± 0.0 [a]	
JN130	38.0 ± 0.1 [c]	44.2 ± 0.2 [b]	6.2 ± 0.1 [a]	44.6 ± 0.2 [b]	0.1 ± 0.0 [a,b]	
JMD130	37.1 ± 0.8 [c]	44.2 ± 0.7 [b]	6.3 ± 0.5 [a]	44.7 ± 0.8 [b]	0.1 ± 0.0 [a,b]	
JK130	37.5 ± 0.8 [c]	44.3 ± 0.8 [b]	6.5 ± 0.6 [a]	44.8 ± 0.9 [b,c]	0.1 ± 0.0 [a,b]	
J90	24.4 ± 0.9 [a]	45.6 ± 0.5 [b,c]	9.1 ± 0.4 [b]	46.5 ± 0.5 [c,d]	0.2 ± 0.0 [c]	
J90c	25.7 ± 1.6 [a]	39.4 ± 0.2 [a]	6.9 ± 1.2 [a]	40.0 ± 0.4 [a]	0.2 ± 0.0 [b,c]	

* Derived by an appropriate program (http://hclwizard.org/hclcolorpicker/, accessed on 25 May 2021) and based on H*, L*, and C* parameters. [a–d] The differences between mean values with the same letters in columns were not statistically significant ($p < 0.05$).

Table 5. Betalain (BL), betanin (BN), and vulgaxanthin (V) content and the retention of betalains in the obtained cloudy beetroot juice powders (abbreviations of variants explained in Table 1).

Variant	BL (mg/100 g Solids)	BN (mg/100 g Solids)	V (mg/100 g Solids)	Betalain Retention (%)	Betanin Retention (%)	Vulgaxanthin Retention (%)
Juice	1141.26 ± 3.82	651.56 ± 1.68	489.70 ± 3.82	-	-	-
J130	819.1 ± 13.0 [d]	465.4 ± 6.8 [d]	345.0 ± 13.1 [d]	71.8 ± 0.6 [b]	71.4 ± 1.3 [b]	70.5 ± 2.7 [b]
JM130	527.1 ± 9.4 [a]	311.9 ± 6.5 [a]	215.3 ± 3.1 [a]	77.0 ± 1.4 [c]	79.8 ± 1.2 [c]	73.3 ± 1.4 [b,c]
JN130	576.7 ± 11.7 [b]	329.8 ± 3.6 [a,b]	246.9 ± 7.9 [b]	84.2 ± 0.3 [d]	84.4 ± 0.5 [d]	84.0 ± 0.2 [d]
JMD130	546.1 ± 25.2 [a,b]	314.4 ± 13.7 [a]	231.8 ± 11.7 [a,b]	79.8 ± 1.6 [c]	80.4 ± 0.9 [c]	78.9 ± 2.6 [c]
JK130	581.1 ± 4.9 [b]	342.1 ± 16.0 [b]	238.9 ± 11.2 [a,b]	84.9 ± 0.7 [d]	87.5 ± 3.8 [d]	81.3 ± 2.6 [d]
J90	691.3 ± 11.1 [c]	384.6 ± 8.2 [c]	306.8 ± 2.9 [b]	60.6 ± 0.8 [a]	59.0 ± 2.3 [a]	62.7 ± 1.3 [a]
J90c	824.6 ± 9.4 [d]	416.0 ± 4.5 [c]	408.7 ± 5.6 [e]	72.3 ± 1.2 [b]	63.9 ± 1.8 [a,b]	83.5 ± 0.8 [d]

[a–e] The differences between mean values with the same letters in columns were not statistically significant ($p < 0.05$).

The C* color parameter indicates the saturation (purity) of the color. The highest value for this parameter was obtained in the case of the powder dried without any carriers at the temperature of 130 °C (J130) and the lowest for the concentrated juice without carriers, dried at low temperature (J90c). The temperature, type, and presence of the carrier did not significantly affect the value of this parameter because the powders with the addition of carriers did not differ from each other ($p \leq 0.05$). Juice thickening decreased the color saturation of the obtained powders (Table 4). The powders without the addition of carriers had a significantly higher H* parameter compared to those with carriers.

3.12. Betalains

The betanin and vulgaxanthin contents in the juice were 651.56 ± 1.68 and 489.70 ± 3.82 mg/100 g solids, respectively (Table 5). Other authors have obtained betanin and vulgaxanthin contents in juice at amounts of 429.24 and 525.9 mg/100 g solids (Carmo et. al., 2019).

The powder without the addition of carriers dried at 130 °C (J130) was characterized by the highest betanin content. The lowest was found for the powders with the additions of powdered milk and maltodextrin (JM130 and JMD130) (Table 5). With regard to the yellow pigment vulgaxanthins, the highest content was obtained in the powder without carriers dried at a reduced temperature from concentrated solution (J90c) and the lowest in the powders with powdered milk (JM130).

Janiszewska (2014), who obtained powders from beetroot juice concentrate, attained betanin content in a range from 109 ± 7 to 129 ± 1 mg/100 g solids [2] and vulgaxanthin content from 34 ± 3 to 61 ± 2 mg/100 g solids. However, the results in the present study were higher, which could have been due to the differences in beetroot type, the method of juice preparation, and the drying conditions of the process, especially the air temperature.

It can be concluded that there was no significant influence of temperature on the red pigment content, but there was a significant influence resulting from the presence of a carrier presence on the red pigment content in the obtained powders. In the case of the yellow pigment, the temperature, the presence of the carrier, and the concentration of the juice had significant impacts on the content in the final product.

There was a significant negative correlation (−0.799) between the brightness and the vulgaxanthin content and a positive (0.836) correlation between the proportion of yellow color and the betanin content (Table 6).

Table 6. Pearson correlation coefficients (r) between color parameters and betalain content in the obtained cloudy beetroot juice powders; asterisks indicate statistically significant correlations ($p < 0.05$).

	a*	**b***	**C***	**BL**	**BT**	**V**
L*	0.297	−0.597	0.215	−0.740	−0.634	−0.799 *
a*		0.420	0.994 *	−0.255	−0.005	−0.470
b*			0.510	0.725	0.836 *	0.577
C*				−0.161	0.086	−0.382
BL					0.963 *	0.967 *
BN						0.866 *

Retention

The lowest residual betanin and vulgaxanthin levels in comparison to juice were found in the case of the non-concentrated juice powder obtained at 90 °C (retention of 59.0% and 62.7%, respectively). In the case of the concentrated juice powder, the betanin residue was also low (63.9%), but the vulgaxanthin residue was higher (83.5%). The highest retention of both betanins and vulgaxanthins was found in powders based on nutriose and kleptose: up to 84.0% in the case of vulgaxanthins and 87.5% in the case of betanins. Powders based on milk and maltodextrin were characterized by significantly lower retention: up to 78.9% for vulgaxanthins and 80.4% for betanins. Thus, the powders based on carriers showed significantly higher retention compared to powders without carriers based on juices only. The addition of carriers probably creates a shell that protects the dyes against high temperature. At the same time, higher retention values were found for powders obtained at a higher temperature (130 °C). Do Carmo et al. (2019), while spray-drying beetroot juice with the addition of inulin and whey protein, also found higher betalain contents at a higher temperature [42]. Delia et al. (2019), who microencapsulated the fruit extract of *Escontria chiotilla* and *Stenocereus queretaroensis* with the addition of cactus mucus as a carrier, observed a loss of betalain pigments in the range from 29.7 to 71.4% [43]. Janiszewska and Włodarczyk (2013) obtained losses of betalain at levels of 70.7–73.3% [44].

3.13. HCA

HCA classification of the powders based on powder recovery and the physical properties of powders (Figure 5) distinguished three clusters: (1) the powders without carriers that were not from concentrated juice; (2) the powder without carriers that was from concentrated juice along with the powder with milk; and (3) the powders with nutriose, maltodextrin, and kleptose. HCA classification of the powders based on color and betalain content (Figure 6) indicated a division into two groups: (1) powders with the addition of carriers and (2) powders without carriers.

The addition of carriers noticeably affected the color and the betalain content. When taking into account the physical properties of the powders, a significant effect resulting from the addition of carriers was also observed, but the surprising thing was that the physical properties of the powder with milk and that obtained from concentrated juice were similar.

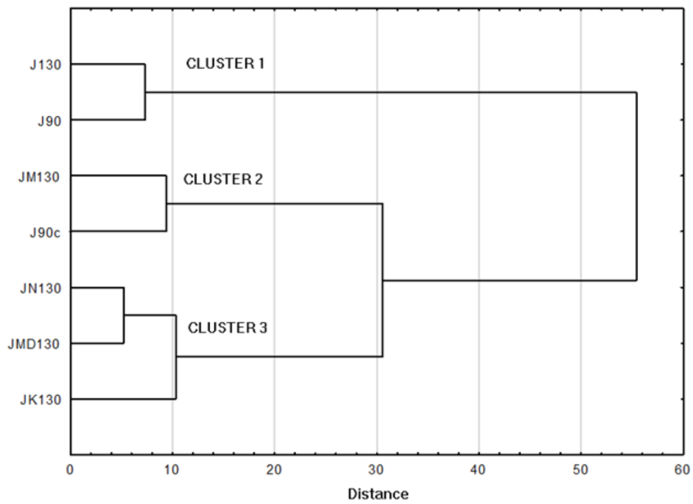

Figure 5. HCA classification of the powders based on powder recovery and the physical properties of the powders.

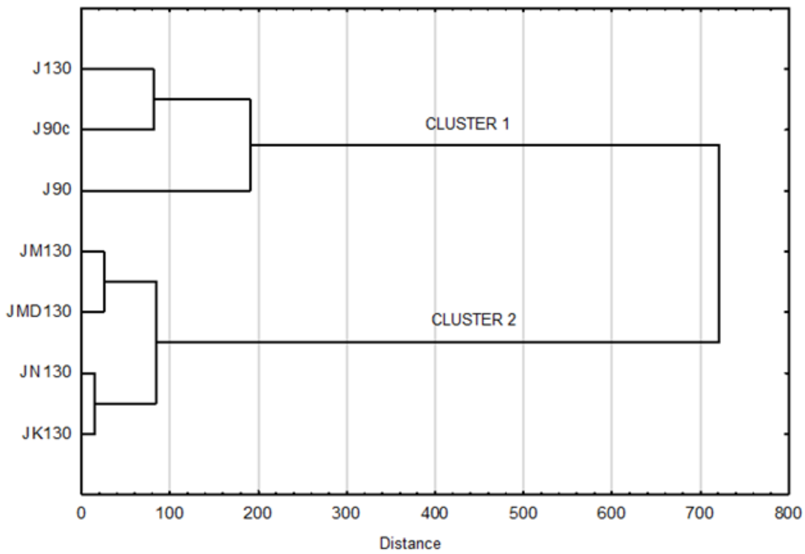

Figure 6. HCA classification of the powders based on color and betalain content.

4. Conclusions

It was possible to obtain 100% beetroot powders (without carriers) with a dehumidified air spray-drying method at temperatures of 90 or 130 °C. The physical and chemical properties of the obtained 100% beetroot powders were satisfactory and often better than those obtained with the addition of carriers. The best procedure for the production of pure red beetroot powder was spray-drying at 130 °C, which made it possible to obtain low hygroscopicity, high color saturation, and a high production yield of up to 90%. On the other hand, the powders obtained at 90 °C were also characterized by satisfactory physical and chemical properties and a high efficiency in the drying process. Higher retention of betalains was achieved with powders with the addition of carriers.

The most interesting variant seems to be the one obtained from the pure juice (without any carriers) at a temperature of 130 °C: it was characterized by high powder recovery (90%), appropriate water content (4%), a betalain retention of 71%, and average flowability.

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