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Research Paper

Effects of carrier agents on powder properties, stability of carotenoids, and encapsulation efficiency of goldenberry (*Physalis peruviana* L.) powder produced by co-current spray drying

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ABSTRACT

Maltodextrin, modified starch, inulin, alginate, gum arabic, and combinations thereof were used as carrier agents for spray drying of carotenoid-rich goldenberry (*Physalis peruviana* L.) juice and compared to cellobiose as an alternative carrier. Powders were analyzed with respect to particle size and morphology, yield, moisture content, cold water solubility, suspension stability, hygroscopicity, carotenoid encapsulation efficiency, and carotenoid retention during storage. A high initial carotenoid concentration after spray drying, a high encapsulation efficiency of 77.2%, and a slow carotenoid degradation kinetics favored the high carotenoid content of the cellobiose powder at the end of the storage. Cellobiose might protect the carotenoids from degradation processes by light exposure, high temperature, and oxygen due to a tighter particle crust and larger particle sizes. Therefore, cellobiose may be considered a potential carrier agent for the encapsulation of carotenoid-rich fruit juices.

1. Introduction

The goldenberry (*Physalis peruviana* L.) is a tropical fruit that is native to the Andean highlands (İzli et al., 2014). Benefits associated with goldenberries are their nutritional composition, bioactive components such as carotenoids, and the pleasant flavor making them interesting as a functional food (İzli et al., 2014; Cortés et al., 2017; Etzbach et al., 2018).

Since juices are perishable and valuable compounds are readily lost during storage, drying of fruit juices is commonly applied to increase shelf life and also to develop new products. Due to the short processing time and the use of comparatively low temperatures, spray drying is suitable for heat-sensitive food ingredients such as carotenoids (Ferrari et al., 2012). Fruit juice powders have many benefits over fruit juices, e.g., reduced volume and weight, reduced packaging, easier handling and transportation, and, in particular, a longer shelf life (Goula and Adamopoulos, 2010). The conversion of sugar-rich juices into powders is often accompanied by problems of stickiness because of the hygroscopicity and thermoplasticity of the products promoted by the high humidity and temperature of the drying air (Cortés et al., 2017). Stickiness is mainly caused by the low glass transition temperature (T_g) of the sugars present in fruits such as glucose, fructose, and sucrose (T_g of 31 °C, 26 °C, and 62 °C, respectively) (Bhandari et al., 1997; Thorat et al., 2018). Therefore, spray drying of fruit juices needs the addition of carrier agents to increase the glass transition temperature of the feed. The carrier agents may additionally protect sensitive food components like carotenoids, preserve flavors, and reduce volatility and reactivity (Phisut, 2012).

The most commonly used carrier agents are maltodextrin and gum arabic (Ferrari et al., 2012). Inulin, alginate, and modified starch have also been widely used for microencapsulation purposes (Gharsallaoui et al., 2007; Saenz et al., 2009). An interesting potential carrier agent is cellobiose. Cellobiose is a disaccharide with two (1,4)-β-D-glucopyranose units obtained as the main product from the enzymatic hydrolysis of cellulose (Liu et al., 2015) and as a by-product during the refining process of beet sugar production. Since cellobiose is readily available at an

Abbreviations: DAD, diode array detection; HPLC, high-performance liquid chromatography; BHT, butylated hydroxytoluene; DE, dextrose equivalents; dw, dry weight; FE-SEM, field emission scanning electron microscopy; TLD, through lens detector; ETD, everhart thornley detector; T_g, glass transition temperature.

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industrial scale, not hydrolyzed by human intestinal enzymes rendering it as a potential prebiotic (Nakamura et al., 2004), water soluble, and shows a low sweetness, cellobiose might be a promising food additive. In addition, the high glass transition temperature (102 °C) and melting temperature (248 °C) of cellobiose compared to other disaccharides like sucrose (62 °C and 192 °C, respectively) or maltose (87 °C and 129 °C, respectively) (Thorat et al., 2018), render cellobiose interesting for spray drying as an alternative to widely utilized oligo- and polysaccharides. There are no studies on the potential use of cellobiose as a carrier agent for encapsulation purposes. To evaluate the potential application of cellobiose as a carrier agent for spray drying, properties of spray dried cellobiose powders should be compared to characteristics of powders produced with carrier agents known for their good spray drying features.

The objective of the present work was to evaluate the use of well-established types of carrier agents and cellobiose as a potential new one for spray drying of goldenberry juice regarding particle size and morphology, yield, moisture content, cold water solubility, suspension stability, hygroscopicity, carotenoid encapsulation efficiency, and carotenoid retention during storage.

2. Material and methods

2.1. Sample material and juice production

Ripe goldenberries with calyx imported from Colombia were acquired from a wholesale market. Fruits were homogenized in a Blendtec® Designer 725 Blender and juice was extracted using a fruit press (Para Press Juice Press, Paul Arauner GmbH & Co KG, Kitzingen-Main, Germany). The juice was filtered with the help of tea filters to prevent clogging of the nozzle during spray drying, portioned, and stored at −20 °C until feed mixture preparation.

2.2. Chemicals and standards

Ultrapure water was obtained from a PURELAB flex 2 water purification system (ELGA LabWater, Paris, France). GRINDSTED® sodium alginate LFD 1205 (Danisco, Copenhagen, Denmark), cellobiose ≥99% (Pfeifer & Langen GmbH & Co KG, Cologne, Germany), gum arabic (Symrise GmbH & Co. KG, Holzminden, Germany), inulin (BENEO-Orafti S.A., Tienen, Belgium), lactose (VWR, Mannheim, Germany), maltodextrin DE12 from corn starch (Berco-Arzneimittel Gottfried Herzberg GmbH, Kleve, Germany), and starch sodium octenyl succinate (modified starch, National Starch & Chemical GmbH, Hamburg, Germany) were used for spray drying. Carotenoid analysis was conducted using acetone 99.8% (VWR, Mannheim, Germany), methanol (HPLC grade) from Th. Geyer (Renningen, Germany), and ethanol ≥99.8%, methyl *tert*-butyl ether (HPLC grade), hexane (HPLC grade), and butylated hydroxytoluene (BHT, ≥99.8%), all from Carl Roth GmbH & Co. KG (Karlsruhe, Germany). The β-carotene standard (≥97%) was from Sigma Aldrich (Munich, Germany). Sodium sulfate (Carl Roth GmbH & Co. KG, Karlsruhe, Germany) was used for the determination of the hygroscopicity.

2.3. Preparation of the feed mixture and spray drying conditions

The goldenberry juice was mixed under stirring with water in a ratio of 1:3 (*w/w*) and 20% of carrier agent referred to the juice weight. Feed mixtures were produced by adding the carrier agents to the diluted goldenberry juice according to Table 1 unless otherwise stated. The feed mixtures of the powders P3, P6, and P7 were prepared by dissolving the carrier agents in water using an ultra turrax (IKA, Staufen, Germany) prior to goldenberry juice addition. The feed mixtures were spray dried immediately after preparation. Before and during spray drying, the feed mixtures were kept homogeneously using a magnetic stirrer and protected from light using aluminum foil.

A laboratory-scale mini spray dryer B-290 (Büchi, Flawil, Switzerland) with a nozzle diameter of 1.5 mm was used. Spray drying was carried out at a pump rate of 30% (approx. 10 mL/min), an aspirator rate of 100% (approx. 35 m^2/h), an air flow of approximately 473 L/h, and an inlet temperature of 140 °C. During spray drying, an outlet temperature of 70 ± 4.2 °C was observed since the outlet temperature is not adjustable and depends on the carrier agent and the ambient temperature. The spray dried powders were weighed and stored at −80 °C until analysis. Preparation of feed mixtures and spray drying was performed in duplicate.

Table 1
Composition of feed mixtures used for spray drying. The composition of the carrier agents and additives is based on the juice weight.

Powder	Carrier agents	Composition (%, *w/w*)
P1	Maltodextrin DE 12	20
P2	Modified starch	20
P3	Gum arabic	20
P4	Cellobiose	20
P5	Inulin	20
P6	Alginate	20
P7	Maltodextrin DE12	10
	Gum arabic	10
P8	Modified starch	10
	Gum arabic	10
P9	Maltodextrin DE12	3.325
	Modified Starch	3.325
	Gum arabic	13.35
P10	Maltodextrin DE12	5.9
	Lactose	14.1

2.4. Analysis of spray dried powders

2.4.1. Yield

The powder yield based on the juice dry weight (juice dw) in the powder and in the feed mixture was calculated according to the following equation.

$$yield(\%) = \frac{\text{juice dw}_{powder}}{\text{juice dw}_{feed}} \cdot 100 \quad (1)$$

2.4.2. Particle size distribution and particle morphology

The particle size distribution was analyzed by dynamic image analysis using a Camsizer X2 (Retsch Technology, Haan, Germany) operated in X-Jet mode for air pressure dispersion. The samples were run with a sample size of 1 g, a dispersion pressure of 70 kPa, a nominal covered area of 0.1%, and a slit width of 4 mm. The analyses were performed blind with randomly coded samples. Powder 4 and 10 needed to be sieved (>1 mm) before particle size analysis because of agglomerates formed.

For the determination of the particle morphology, the samples were fixed on stubs using colloidal silver or double-sided carbon tape and coated with platinum at 30 mA for 20 s using a Q150TS vacuum-sputtering coater (Quorum Technologies, Lewes, United Kingdom). A Helios G4 CX field emission scanning electron microscope (FE-SEM, Thermo Fisher Scientific, Braunschweig, Germany) at an accelerating voltage of 1 kV was used. The powders were imaged using a through lens detector (TLD) with a stage bias of 500 V or an everhart thornley detector (ETD) in case of powder 4. A working distance of 4 mm, a dwell time of 200 ns with 16 line integration, and a current of 11 pA was applied.

2.4.3. Moisture content

The moisture content (%dw) was determined thermogravimetrically using a Sartorius Moisture Analyzer MA100/MA50 (Göttingen, Germany) by drying 1 g of the goldenberry juice and 0.5 g of the spray dried powders.

2.4.4. Hygroscopicity

Hygroscopicity was calculated as the moisture absorption observed after exposing the powders to humid air with 81% relative humidity. For the determination of the hygroscopicity, 1 g of powder was weighed in an

aluminum dish and placed in a desiccator containing 200 mL of a saturated solution of Na_2SO_4 at 25 °C. The weight gain was determined in triplicate after 1, 3, 7, and 24 h and the moisture absorption was calculated with the following equation.

$$\textit{moisture absorption}\ (\%)\ \text{after}\ 1,3,7,24\,\text{h} = \frac{\text{weight gain after}\ 1,3,7,24\,\text{h}}{\text{weight of sample}} \cdot 100 \quad (2)$$

2.4.5. Cold water solubility

The cold water solubility of the spray-dried powders was determined in triplicate according to Comunian et al. (2011) with slight modifications. A 50 mL (1%) suspension of the powder was stirred for 30 min at 310 rpm using a magnetic stirrer. The suspension was centrifuged with a Heraeus Megafuge 40R Centrifuge (Thermo Fisher Scientific, Braunschweig, Germany) at 11,000*g* and 20 °C for 10 min. A 25 mL aliquot of the supernatant was weighed in an aluminum dish and dried in an oven at 110 °C for 24 h. For the calculation of the cold water solubility, the following equation was used.

$$\textit{cold water solubility}\ (\%) = \frac{\text{weight of solid in supernatant} \cdot 2}{\text{weight of sample}} \cdot 100 \quad (3)$$

2.4.6. Suspension stability

The suspension stability of feed mixtures and dissolved powders was determined in triplicate as described by Carneiro et al. (2013) with slight modifications. The spray dried powders were dissolved in water to the same dry weight as the respective feed mixtures. A 10 mL aliquot of the suspensions was transferred into a test tube, sealed, and stored at ambient temperature for 24 h and 8 days. The separation (%) was calculated using the following equation.

$$\textit{separation}\ (\%) = \frac{H_S}{H_{100\%}} \cdot 100 \quad (4)$$

where H_S represents the height of the sedimented phase after 24 h and $H_{100\%}$ the maximum sedimentation height after 8 days.

2.4.7. Carotenoid analysis

2.4.7.1. Extraction of carotenoids. All powders were analyzed for total and surface carotenoids. Total carotenoids of the goldenberry juice and the powders were extracted in triplicate by weighing 3 g and 0.5 g in centrifuge tubes, respectively (Etzbach et al., 2018). The powders were dissolved in water before extraction of total carotenoids. The extract was evaporated to dryness with nitrogen and stored at −80 °C until analysis.

Surface carotenoids were determined in triplicate according to Wagner and Warthesen (1995) with slight modifications. Powders (0.5 g) were weighed in centrifuge tubes and extracted with 10 mL hexane containing 0.1% BHT. The mixture was shaken and centrifuged with a Heraeus Megafuge 40R Centrifuge (Thermo Fisher Scientific, Braunschweig, Germany) at 11,000*g* and 5 °C for 5 min. The liquid phase was transferred to a brown pear shaped flask and the extraction step was repeated. The combined extracts were evaporated using a rotary evaporator (Büchi, Essen, Germany) at 35 °C and 145 mbar. The residue was transferred to a 4-mL-vial by dissolving it in methanol/methyl *tert*-butyl ether (50:50, *v/v*) containing 0.1% BHT. Samples were evaporated to dryness with nitrogen and stored at −80 °C until analysis.

For HPLC analysis, the extracts were made up to 1 mL with methanol/methyl *tert*-butyl ether (50:50, *v/v*) containing 0.1% BHT and filtered through 0.2 µm Chromafil RC-20/15 MS filters (Macherey-Nagel, Düren, Germany).

2.4.7.2. Identification and quantification of carotenoids. Carotenoids in *Physalis peruviana* L. were already identified by HPLC-DAD-APCI-MS^n (Etzbach et al., 2018). They were quantified as described previously (Etzbach et al., 2019) using a Prominence UFLC system (Shimadzu, Kyoto, Japan) equipped with two Nexera X2 LC-30AD high-pressure gradient pumps, a Prominence DGU-20A5R degasser, a Nexera SIL-30AC Prominence autosampler (15 °C, injection volume 5 µL and 15 µL for total carotenoids and surface carotenoids, respectively), a CTO-20AC Prominence column oven at 25 °C, and a SPD-M20A Prominence diode array detector. The chromatograms were processed at 450 nm and carotenoids were quantified as β-carotene equivalents using an external calibration curve.

2.4.7.3. Encapsulation efficiency. The encapsulation efficiency was calculated using the following equation.

$$\textit{encapsulation efficiency}\ (\%) = \frac{\text{total carotenoids} - \text{surface carotenoids}}{\text{total carotenoids}} \cdot 100 \quad (5)$$

2.4.7.4. Storage stability of the carotenoids in the spray dried powders. The powders of drying duplicates were pooled for the determination of the storage stability. The powders were stored for 6 weeks at 30 °C in glassware under exclusion of light in a heating cabinet. Aliquots of the samples were taken before storage (day 0) and after 2, 4, 8, 14, 21, 28, and 42 days of storage. The aliquots were stored frozen at −80 °C until carotenoid analysis.

To characterize the kinetics of carotenoid degradation in the spray dried powders, the experimental data was fitted to a first-order kinetic model as described in previous studies (Wagner and Warthesen, 1995; Desobry et al., 1997; Weber et al., 2017) (equation (6))

$$c_t = c_0 \cdot e^{-kt} \quad (6)$$

where c_0 is the initial carotenoid concentration, c_t the carotenoid concentration at the time t, and k the reaction rate constant. Half-life times ($t_{\frac{1}{2}}$) at which the carotenoid content was reduced by 50% were calculated according to equation (7).

$$t_{\frac{1}{2}} = \frac{\ln 2}{k} \quad (7)$$

2.5. Statistical analysis

Statistical analysis was conducted using the XLSTAT software version 2014.4.06 (Addinsoft, Paris, France). An ANOVA with Bonferroni post-hoc test was performed to determine significant differences ($p \leq 0.05$). Experimental data was fitted to a first-order kinetic model using the *OriginPro 8 G* software (OriginLab Corporation, Northampton, MA, USA).

3. Results and discussion

3.1. Spray drying yield

The yield of goldenberry juice powders varied between 49.7% (P6) and 67.2% (P2) (Table 2), which is similar to data reported for pomegranate juice (Horuz et al., 2012), açai pulp (Tonon et al., 2008), and black mulberry juice (Fazaeli et al., 2012). The type of carrier agent showed no significant effect on the process yield. However, the process yield of the powders containing cellobiose (P4, 52.0%) and alginate (P6, 49.7%) was slightly lower than that of the maltodextrin and modified starch powders. The lower yield of the cellobiose powder compared to the maltodextrin powders can be explained by a lower glass transition temperature of 102 °C (Thorat et al., 2018) of this disaccharide resulting in particle agglomeration and stickiness within the spray drier. For maltodextrin DE12 with a moisture content <5%, a glass transition temperature of about 160 °C was observed (Goula and Adamopoulos, 2008).

Table 2
Physical properties of the spray dried powders. Different letters indicate significant differences ($p \leq 0.05$). Values are mean ± standard deviation (n = 3).

Powder	Yield (%)	Total solids (%)	Moisture absorption after 24 h (%)	Cold water solubility (%)
P1	61.96 ± 14.14^{a}	96.27 ± 0.70^{abc}	16.37 ± 1.24^{ab}	92.11 ± 0.58^{ab}
P2	67.16 ± 5.53^{a}	96.19 ± 0.76^{abc}	15.86 ± 0.99^{ab}	92.34 ± 0.92^{ab}
P3	60.57 ± 8.16^{a}	95.27 ± 0.48^{bc}	15.24 ± 0.58^{ab}	87.92 ± 0.95^{c}
P4	51.99 ± 1.52^{a}	97.57 ± 0.50^{a}	14.10 ± 0.78^{b}	90.31 ± 1.08^{abc}
P5	57.69 ± 0.46^{a}	96.61 ± 0.36^{abc}	14.60 ± 0.44^{b}	82.73 ± 2.08^{d}
P6	49.68 ± 6.15^{a}	95.97 ± 0.36^{abc}	17.11 ± 0.66^{a}	91.09 ± 1.28^{ab}
P7	60.57 ± 10.51^{a}	95.19 ± 1.30^{bc}	15.14 ± 1.45^{ab}	90.38 ± 2.08^{abc}
P8	61.83 ± 12.38^{a}	94.75 ± 1.34^{c}	15.05 ± 1.99^{ab}	93.23 ± 1.85^{a}
P9	64.67 ± 3.97^{a}	95.64 ± 1.46^{abc}	17.20 ± 0.47^{a}	93.25 ± 2.29^{a}
P10	58.18 ± 7.46^{a}	97.23 ± 0.23^{ab}	15.71 ± 1.98^{ab}	90.16 ± 0.85^{bc}

3.2. Particle size distribution and morphology

Figure 1 shows the particle size distribution and the FE-SEM microphotographs of the powders produced with different carrier agents. Spray drying with maltodextrin DE12 (P1), modified starch (P2), gum arabic (P3), alginate (P6), and the combination thereof (P7) resulted in fine powders with small rounded particles with wrinkled surfaces that had mean diameters of 11.5–21.5 μm. This is within the range of values expected for spray dried powders (Dias et al., 2018; Pereira et al., 2019; Tonon et al., 2009). For the alginate powder (P6), the microphotographs showed cracks on the surface of the particles.

Particles of cellobiose (P4), inulin (P5), and lactose with maltodextrin (P10) exhibited irregular spherical shapes, smoothed surfaces, and were agglomerated. The largest particles were found in the cellobiose powder with a mean diameter of 524 μm. The occurrence of agglomeration and the formation of link bridges between the particles might be related to the low glass transition temperature of cellobiose (T_g of 102 °C), inulin (T_g of 102–154 °C, depending on the inulin used) (Dias et al., 2018; Hinrichs et al., 2001), and lactose (T_g of 101 °C) (Thorat et al., 2018). A smooth surface was also found for inulin jussara pulp microparticles, which was attributed to the higher molecular flexibility of this carbohydrate (Lacerda et al., 2016; Mensink et al., 2015).

The particle size and the morphology affect various technological properties like bulk density, flowability, compaction, rehydration, and solubility, which have an impact on the possible applications of the powders. Very fine powders tend to have low wettabilities (Dias et al., 2018). In the study of Kyaw Hla and Hogekamp (Kyaw Hla and Hogekamp, 1999) cocoa beverage powders with particle sizes smaller than 200 μm showed long wetting times of several minutes. The authors defined a critical particle size of 180 μm with a product-depending margin of ±20 μm below which as few particles as possible should appear in the final product. Therefore, in case of fine powders, agglomeration processes are suggested (Pereira et al., 2019; Schubert et al., 2003; Lacerda et al., 2016).

3.3. Moisture content and hygroscopicity

All spray dried powders had moisture contents lower than 5.25% (Table 2), ensuring microbiological stability (Koç et al., 2011; Da Silva et al., 2013). Goldenberry juice powder produced with cellobiose (P4) showed significantly lower moisture contents than powders produced with gum arabic (P3, P7, P8). This can be attributed to the branched structure of gum arabic, which has a high number of ramifications with hydrophilic groups that can adsorb water from the ambient air (Da Silva et al., 2013).

The hygroscopicity of the powders after 24 h varied between 14.1% and 17.2% (Table 2). Despite the low moisture content, the cellobiose powder showed the lowest moisture absorption after 24 h among all powders (Table 2). A higher hygroscopicity of cellobiose (P4) was expected compared to high molecular weight compounds such as maltodextrin (P1) and gum arabic (P3) (Kurozawa et al., 2009). Probably, a dense molecular packaging of the cellobiose particles enhanced the barrier properties toward water and reduced the moisture absorption from the ambient air inside the particle. Moreover, particle agglomeration positively affected the moisture absorption.

Changes in the morphology of the stored powders were observed after 24 h. Some powders showed particle clumps and compaction (P3, P5, P6, P7, P8, and P9), whereas others had the appearance of a highly sticky liquid as a result of an advanced state of caking (P1, P2, P4, and P10). The increasing moisture content of the powders decreased both the glass transition and collapse temperatures of the powders (Le Meste et al., 2002). A high initial glass transition temperature of the carrier agent might compensate for high moisture absorption because of particle porosity, whereas in case of a comparatively low glass transition temperature of the carrier agent, caking might be impeded by a tight molecular packaging of the particle crust.

3.4. Cold water solubility

The spray dried powders showed a good cold water solubility of at least 82% (Table 2). Among the carrier agents tested, inulin (P5) exhibited the lowest cold water solubility with 82.7%. The low water solubility of inulin at ambient temperature was already described (Kim et al., 2001) and can be explained by its gel forming properties leading to a white creamy appearance due to sol-gel transition. The comparable low water solubility of the gum arabic powder (P3) was increased significantly by about 6.0% due to partial replacement with modified starch (P8) (Table 2). In accordance with several studies (Loksuwan, 2007; Tontul and Topuz, 2017), the present study revealed high cold water solubilities (>90%) of powders produced with maltodextrin or modified starch. The good water solubility of maltodextrin can be attributed to its hydrophilic character. The amphiphilic character of the modified starch favored the simultaneous interaction with polar and non-polar constituents and their dispersion (Da Silva et al., 2013).

3.5. Suspension stability

A stable suspension is essential to produce a homogenous powder (Gharsallaoui et al., 2007). During the spray drying process, suspension stability was ensured by stirring. After 24 h without stirring, all feed mixtures showed a separation of 100% and a sedimented layer (7 mm) of fruit pulp. Similarly, the reconstituted powder suspensions exhibited a sedimentation height of 5 mm at 100% separation after 24 h except the alginate powder (P6), which was stable over this period showing a separation of 0%. The improved solubility of the particles may be explained by a reduction in the particle diameter, increased physical particle interaction, and polymorphic changes from a crystalline to an amorphous form or a higher energy crystalline form through spray drying (Broadhead et al., 1992). Due to the apparent higher viscosity of the alginate powder suspension and its fine powder structure, the sedimentation speed of the particles was reduced. The suspension of both the feed and the reconstituted inulin powder (P5) showed the separation of inulin from the water phase, confirming its low cold water solubility (Kim et al., 2001).

3.6. Carotenoids

3.6.1. Retention during spray drying

In comparison to the goldenberry juice (4134 μg/100 g juice dw), spray dried powders showed a significant reduction in the total carotenoid content of 38.9%–69.7% depending on the carrier agent used (Fig. 2). Carotenoids are susceptible to isomerization, oxidation, conversion, and cleavage reactions induced by heat, oxygen, light, free

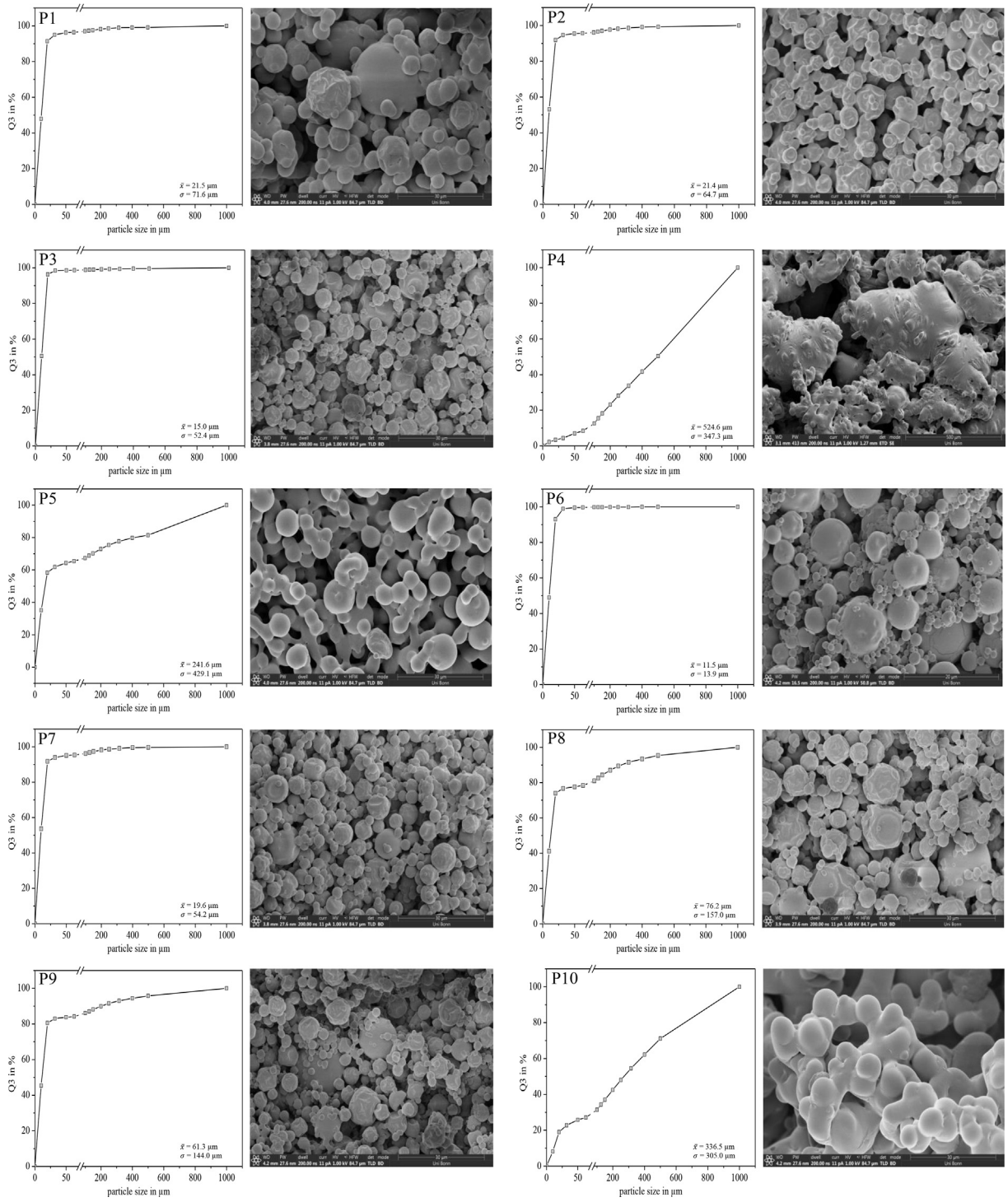

Fig. 1. Particle size distribution plotted as the cumulative distribution Q3 in % versus particle size in µm and FE-SEM micrographs of goldenberry juice powders produced with different carrier agents according to Table 1.

radicals, and acids (Schieber et al., 2016). The highest total carotenoid contents after spray drying were found in the maltodextrin (P1) and cellobiose powder (P4) with 2524 and 2482 µg/100 g juice dw, respectively (Fig. 2). Spray drying with alginate (P6) resulted in the lowest total carotenoid content of 1251 µg/100 g juice dw. The combination of gum arabic with maltodextrin or/and modified starch (P7, P8, and P9) significantly reduced the total carotenoid content compared to the maltodextrin (P1) and modified starch (P2) powder.

Despite similar particle size distributions of the maltodextrin DE12 (P1), modified starch (P2), gum arabic (P3), and alginate (P6) powders, there are significant differences in their carotenoid concentration after spray drying (Fig. 2). These differences in carotenoid retention during spray drying may be explained by different particle morphologies and drying rates during spray drying caused by the viscosity of the feed suspension. The duration of the first and second drying phase in the spray drying process depends on the viscosity of the feed suspension. Due to the evaporation chill, the first drying phase is characterized by milder temperatures compared to the second drying phase of crust formation. Highly viscous feed suspensions like alginate (P6) and gum arabic (P3) impair circulation currents within the droplet, promote a fast crust

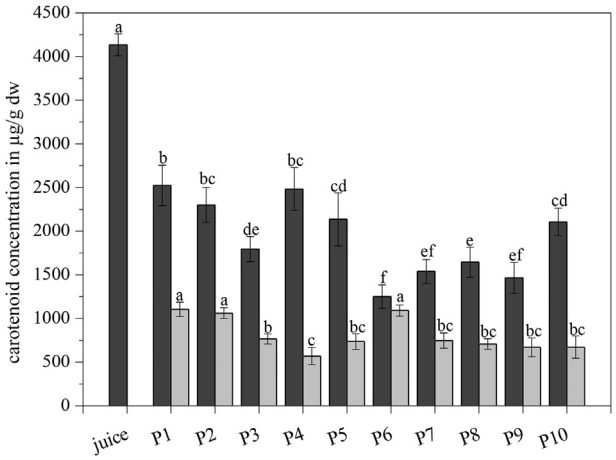

Fig. 2. Total carotenoid (■) and surface carotenoid (▒) concentrations of the encapsulated goldenberry juice powders (P1–P10). Different letters indicate significant differences ($p \leq 0.05$). Values are mean ± standard deviation ($n = 3$ for juice; $n = 6$ for powders).

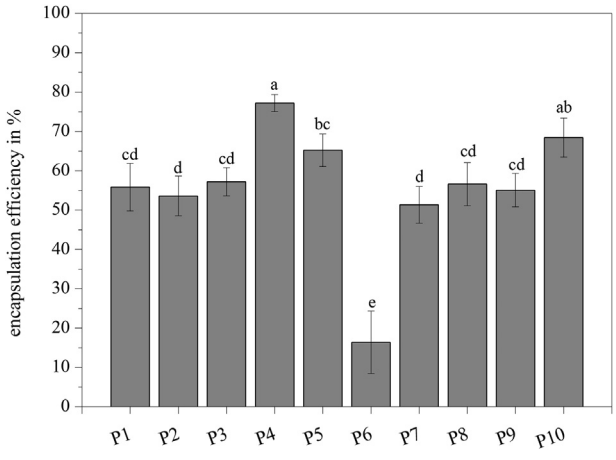

Fig. 3. Carotenoid encapsulation efficiency of the different carrier agents used. Different letters indicate significant differences ($p \leq 0.05$). Values are mean ± standard deviation ($n = 6$).

formation and, therefore, shorten the first drying phase at moderate drying temperatures (Teixeira et al., 2004). Cracked particles were observed in the FE-SEM microphotographs of the alginate powder (Fig. 1) owing to a fast crust formation because of the increasing vapor pressure inside the particle. The evolving dented surfaces and porous particles might enhance oxidation and facilitate further degradation processes of carotenoids. Additionally, the extractability of the carotenoids might be affected by the gelling properties of some carrier agents, e.g. in case of the alginate powder (Lee and Mooney, 2012). Low viscous feed suspensions such as maltodextrin (P1) and cellobiose (P4) reduced the time of high temperature exposure due to an extended first drying phase of crust formation (Teixeira et al., 2004). Mild drying conditions and a reduced contact surface of carotenoids with air might be responsible for the high carotenoid contents of the maltodextrin (P1), modified starch (P2), cellobiose (P4), and inulin (P5) powders. Low molecular weight sugars such as cellobiose might act as a plasticizer and prevent shrinkage of the particles (Loksuwan, 2007) (Fig. 1).

A high encapsulation efficiency is needed to reduce oxidative degradation of compounds on the particle surface and to increase product stability. Surface carotenoid contents varied between 569 and 1105 µg/100 g juice dw (Fig. 2). Depending on the carrier agent used, an encapsulation efficiency of 16.4–77.2% was obtained. The cellobiose powder (P4) showed the highest encapsulation efficiency of about 77.2% (Fig. 3). The alginate powder (P6) had the lowest encapsulation efficiency of about 16.4% because of cracked particles (Fig. 1). The particle size strongly affects the encapsulation efficiency since fine powders (P1, P2, P3, and P6) due to their larger surface area showed significant higher surface carotenoid contents and significant lower encapsulation efficiencies compared to the agglomerated powders (P4, P5, and P10) (Figs. 2 and 3).

Besides particle size, differences in the encapsulation efficiency might be attributed to differences in the polymer matrices formed by the carrier agents, which may impair the retention properties and crust forming capacity. The replacement of 70.5% of maltodextrin with lactose (P10) significantly increased the encapsulation efficiency by 22.6% in comparison to the maltodextrin powder (P1) (Fig. 3). Rosenberg and Sheu (1996) suggested that the addition of lactose may limit the diffusion of small organic molecules through the particle crust. In its amorphous state, lactose may act as a hydrophilic sealing material and reduce the diffusion of carotenoids from the particle core to the surface during spray drying due to fast and dense crust formation. This may also apply to cellobiose, which improved the molecular packaging of the particles and stabilized the system regarding diffusion, resulting in good encapsulation properties.

The carotenoid profile of goldenberry comprises of all-*trans*-β-carotene (>50%) and its *cis*-isomers, all-*trans*-lutein, and a considerable amount of carotenoid fatty acid esters (Etzbach et al., 2018). Owing to the thermal effect during the spray drying process, isomerization reactions took place and increased the proportion of *cis*-β-carotene in the resulting powders (6.5–8.4%) compared to the juice (4.9%) (Table 1S in the Supporting Information). Since the alginate powder was possibly exposed to higher temperatures during spray drying because of the longer second drying phase, *cis*-isomerization of all-*trans*-β-carotene (8.4%) was favored. The proportion of all-*trans*-lutein was slightly higher in the spray dried powders (0.9–4.2%) compared to the juice (1.2%), whereas the proportion of the carotenoid fatty acid esters was significantly higher in the juice (24.4%) than in the powders (11.6–22.2%). The carotenoid fatty acid esters of goldenberry are mainly esterified with lutein (Etzbach et al., 2018), hence deesterification of carotenoid fatty acid esters during spray drying led to the release of free all-*trans*-lutein.

3.6.2. *Storage stability of carotenoids*

The degradation of surface carotenoids during storage was fitted to a first-order kinetic model ($k = 0.053–0.22$), whereas for the total carotenoids two first-order kinetic models were required to obtain a suitable fit (Fig. 4). The degradation of total carotenoids was characterized by a rapid first-order kinetics ($k = 0.080–0.171$) of carotenoids, due to the enhanced degradation of non-encapsulated carotenoids by oxidation and isomerization, followed by a slower first-order kinetics ($k = 0.015–0.029$) owing to the protective effects of the carrier agents toward the degradation of the encapsulated carotenoids. Surface carotenoids were reduced to one half in the first 2–4 days of storage, indicating the sensitivity of carotenoids toward oxidation, isomerization, and degradation despite moderate storage temperature and the exclusion of light. Nearly all surface carotenoids were degraded at the end of the storage (Table 3).

After 6 weeks of storage, the cellobiose powder provided the significantly best preservation of carotenoids, with a retention of 32.4% and a carotenoid content of 815 µg/100 g juice dw (Table 3). The cellobiose powder exhibited twice the carotenoid content after storage than all other powders. Low reaction rate constants, an initial high carotenoid concentration after spray drying, and a high encapsulation efficiency favored the high carotenoid content at the end of the storage. The use of cellobiose as a carrier agent might have positively affected the particle size and crust formation during the spray drying process, allowing a dense packaging that protects the carotenoids from degradation processes through light, temperature, and oxygen. Probably, a tight crust formation and surface reduction both hindered the diffusion of carotenoids to the particle surface during the spray drying process and

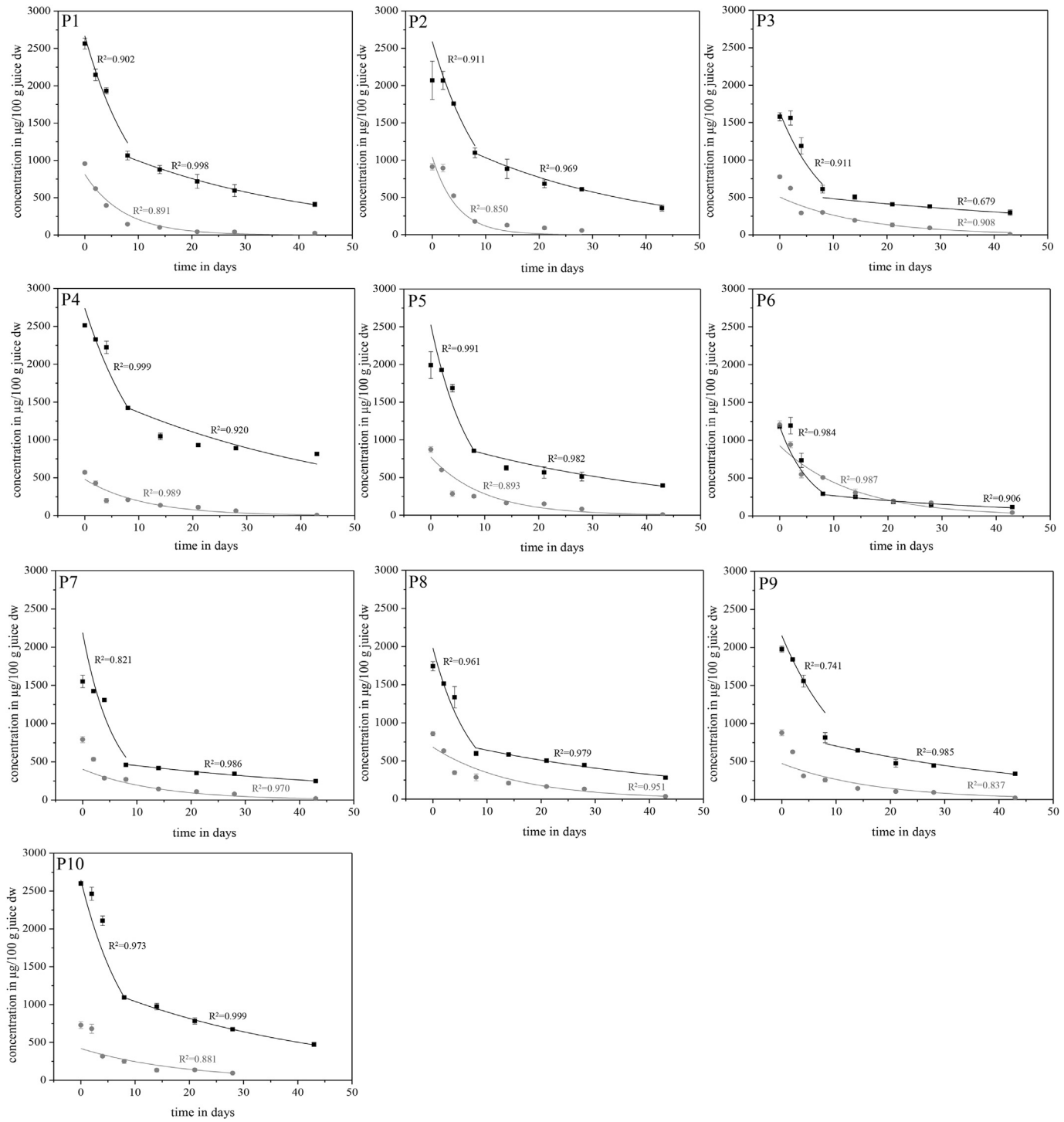

Fig. 4. Development of total carotenoid (■) and surface carotenoid (●) concentrations during the storage of the powders (P1–P10). Different letters indicate significant differences ($p \leq 0.05$). Values are mean ± standard deviation ($n = 3$).

reduced the diffusion of oxygen and water into the particles during storage. Similar results were obtained by Desobry et al. (1999), who observed increased half-lives of β-carotene in different blends of maltodextrin with mono- or disaccharides compared to the reference system without added saccharides. They hypothesized that the mono- and disaccharides presumably reduced the pore size or occupied the pores of the maltodextrin network, thus limiting oxygen diffusion. Low-molecular weight carbohydrates are usually associated with caking, collapsing, and re-crystallization of the amorphous carbohydrate because of the formation of inter-particle bonds (Gharsallaoui et al., 2007). The cellobiose (P4) powder retained its pourability despite its low molecular weight and agglomeration, owing to its dense particle crust, limiting moisture absorption. In contrast, the powders made from modified starch (P2) and maltodextrin with lactose (P10) were not stable upon storage because of caking. During storage, the glass transition temperature of these two powders decreased to a level, which results in particle collapsing. The alginate powder (P6) provided the significantly lowest retention of carotenoids of about 10.1% (Table 3) caused by the low encapsulation efficiency owing to cracked particles (Fig. 1).

Table 3
Half-live times and carotenoid concentrations after 6 weeks of storage of the spray dried powders. Different letters indicate significant differences ($p \leq 0.05$). Values are mean ± standard deviation (n = 3).

Powder	Total carotenoids				Surface carotenoids		
	$t_{\frac{1}{2}}$ (days)		Concentration after 6 weeks (μg/100 g juice dw)	Retention after 6 weeks (%)	$t_{\frac{1}{2}}$ (days)	Concentration after 6 weeks (μg/100 g juice dw)	Retention after 6 weeks (%)
	1st Period	2nd Period					
P1	7.39 ± 1.51^{ab}	25.50 ± 0.66^{b}	409.15 ± 29.45^{bc}	15.95 ± 1.15^{c}	5.18 ± 0.82^{de}	24.27 ± 1.66^{b}	2.54 ± 0.17^{b}
P2	7.34 ± 1.53^{ab}	23.62 ± 1.87^{b}	355.91 ± 38.99^{cde}	17.19 ± 1.88^{bc}	3.17 ± 0.35^{e}	nd	nd
P3	6.45 ± 1.43^{ab}	49.39 ± 17.45^{a}	296.49 ± 36.12^{def}	18.79 ± 2.29^{bc}	11.26 ± 3.41^{ab}	6.10 ± 2.02^{c}	0.79 ± 0.26^{c}
P4	8.48 ± 0.14^{ab}	33.76 ± 6.61^{ab}	815.24 ± 17.53^{a}	32.43 ± 0.70^{a}	7.68 ± 0.69^{bcd}	4.91 ± 0.841^{c}	0.86 ± 0.15^{c}
P5	5.15 ± 0.26^{ab}	30.90 ± 2.83^{ab}	394.10 ± 12.22^{bcd}	19.79 ± 0.61^{b}	6.94 ± 0.48^{cde}	8.52 ± 0.29^{c}	0.98 ± 0.03^{c}
P6	4.15 ± 0.50^{b}	25.57 ± 3.94^{b}	118.60 ± 8.52^{g}	10.07 ± 0.72^{d}	9.40 ± 0.41^{abc}	43.73 ± 3.17^{a}	3.63 ± 0.26^{a}
P7	4.32 ± 1.35^{b}	38.63 ± 1.85^{ab}	248.36 ± 1.69^{f}	16.01 ± 0.11^{c}	9.69 ± 1.27^{abc}	19.51 ± 0.92^{b}	2.47 ± 0.12^{b}
P8	5.16 ± 0.87^{ab}	30.56 ± 3.01^{ab}	281.07 ± 12.24^{ef}	16.12 ± 0.70^{c}	10.32 ± 0.88^{abc}	34.51 ± 2.23^{a}	4.03 ± 0.26^{a}
P9	9.69 ± 4.00^{a}	30.66 ± 2.02^{ab}	339.50 ± 8.88^{cdef}	17.17 ± 0.45^{bc}	11.97 ± 1.26^{a}	20.80 ± 3.16^{b}	2.37 ± 0.36^{b}
P10	6.33 ± 0.46^{ab}	28.50 ± 0.16^{b}	473.72 ± 27.10^{b}	18.22 ± 1.04^{bc}	13.06 ± 1.10^{a}	nd	nd

nd, not detectable.

Differences in the stability of individual carotenoids during storage were observed. The carotenoid profile of the alginate powder consisted only of all-*trans*-β-carotene with a retention of 6.7% owing to the advanced carotenoid degradation (Table 2S in the Supporting Information). A higher retention was found for *cis*-β-carotene (12.0–43.1%) than for its *trans*-isomer (6.7–26.8%). The concentration of *cis*-isomers is affected by the balance of degradation and continuous formation through isomerization from the *trans*-isomers. Thus, the calculated retention does not represent the stability of the *cis*-isomers. A retention of carotenoid fatty acid esters of 3.3–23.9% was observed. A higher retention of all-*trans*-β-carotene than of carotenoid fatty acid esters was shown for almost all powders except for the powders of cellobiose (P4) and maltodextrin with lactose (P10) which showed similar retentions of carotenoid fatty acid esters and all-*trans*-β-carotene. Probably, the enhanced barrier properties toward water and oxygen of P4 and P10 reduced the hydrolysis and oxidation of carotenoid fatty acid esters and further degradation of the liberated xanthophylls to colorless cleavage products. After 6 weeks of storage, the concentration of all-*trans*-lutein was below the limit of detection in all powders, promoted by the low stability of non-esterified xanthophylls (Mertz et al., 2010) and by its low initial concentration in the powders. The cellobiose (P4) powder exhibited the highest retention of all carotenoids detected, whereas the ratio of all-*trans*-β-carotene to *cis*-β-carotenes was shifted toward the *cis*-isomers. It is expected that *cis*-isomers show higher bioavailability than their respective *trans*-isomers, due to their reduced tendency to crystallization and aggregation, resulting in a better incorporation into micelles (Desmarchelier and Borel, 2017). A high retention of provitamin A-active carotenoids like all-*trans*-β-carotene and *cis*-β-carotenes is preferred to maintain the nutritional value of the encapsulated goldenberry juice. Encapsulation of goldenberry juice with suitable carrier agents such as cellobiose may decelerate oxidative degradation of carotenoids.

4. Conclusions

Cellobiose proved to be an effective carrier agent for spray drying of goldenberry juice. In contrast to conventionally used carrier agents like maltodextrin, modified starch, inulin, alginate, gum arabic, and combinations thereof, cellobiose showed similar or even better results regarding moisture content, hygroscopicity, cold water solubility, and carotenoid encapsulation efficiency. The low viscosity of the cellobiose feed mixture and the large particle size of the powder through agglomeration positively affected the retention of carotenoids during spray drying and their encapsulation, respectively. Moreover, the cellobiose powder showed the highest retention of carotenoids during storage because of its tight packaging and high encapsulation efficiency. Since process yield is affected by the molecular weight of the feed composition as a result of increasing glass transition temperature, the combination of cellobiose and maltodextrin may improve the moderate yield of the cellobiose powder and, moreover, impede particle agglomeration. Cellobiose might act as a sealing material in the maltodextrin network, resulting in a tight packaging of the particles and high carotenoid retention. Owing to the lack of data, there is an increased need for further research on the technofunctional properties of cellobiose to fully exploit the potential of this compound as a food additive.

Author declaration

None.

Credit author statement

Lara Etzbach: Conceptualization, Supervision, Methodology, Visualization, Writing - original draft. **Messina Meinert:** Investigation, Methodology, Formal analysis. **Thilo Faber:** Investigation, Resources. **Carolin Klein:** Investigation, Resources. **Andreas Schieber:** Supervision, Writing - Reviewing & Editing, Project administration, Funding acquisition. **Fabian Weber:** Conceptualization, Supervision, Writing - Reviewing & Editing, Project administration, Funding acquisition.

Declaration of Competing Interest

The authors declare no conflict of interest. There was no financial support by Pfeifer & Langen and the anlyses performed by Pfeifer & Langen were randomly coded to prevent any bias.

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Appendix A. Supplementary data

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