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Effect of Microencapsulation by Spray-Drying and Freeze-Drying Technique on the Antioxidant Properties of Blueberry (*Vaccinium myrtillus)* Juice Polyphenolic Compounds

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Blueberry juice with high polyphenol concentration was spray- or freeze-dried using different coating materials: HP-β-cyclodextrin and β-cyclodextrin. The quality of the obtained powders was characterised by their anthocyanin content, total polyphenols and antioxidant capacity. SEM was used for monitoring structures and size (2–20 μm) of the microparticles. The losses of total phenolic compounds during spray-drying reached 76–78% on average, while these of anthocyanins about 57%. Freeze-dried powders showed better retention values of anthocyanins, which was about 1.5-fold higher than for the spray-dried counterparts. All blueberry preparations studied were characterised by very high radical scavenging activity.

INTRODUCTION

Blueberries belong to the family Vacciniaceae, and can be classified as highbush, lowbush, and rabbiteye. Blueberries are one of the best recognized fruits for their potential health benefits. They have been considered one of the fruits with the highest antioxidant potentials, and a few studies have evaluated their anticancer activities. Many beneficial properties of blueberries are attributed to their bioactive polyphenolic compounds: proanthocyanidins and anthocyanins [Yi *et al*., 2005; Seeram *et al*., 2006; Srivastava *et al*., 2007; Matchett *et al*., 2005]. Anthocyanins are also known as non-toxic food pigments, when compared to the synthetic colorants. However, anthocyanins have not been extensively used in food because of their tendency to become unstable under a variety of chemical environments [Castañeda-Owado *et al*., 2009].

In the food industry, the use of microencapsulation to protect, isolate or control the release of given substances is of growing interest. There are many different methods of encapsulation depending on the type of core and wall materials, size of desired capsules, and other factors like resistance to high temperature or physical state. Among several encapsulation techniques, spray-drying and freeze-drying are the most common ones [Gouin, 2004; Gharsallaoui *et al*., 2007]. Freeze-drying is based on the dehydration by sublimation of a frozen product. Substances are not exposed to high temperatures, therefore the freeze-dried products preserve their initial nutritious characteristics. On the other hand, spray-drying is an encapsulation technique based on conversion of feed from fluid to powder state by continuous processing of feed in hot drying medium. Spray-drying technique maximizes heat transfer, and can be used for any product with a liquid-like behaviour. Also a consistent particle size distribution and fast water removal is a reason for spray-drying some heat-sensitive industrial products [Gouin, 2004; Patel *et al*., 2009; do Espírito Santo *et al.,* 2013].

The choice of coating material is very important, since it may influence efficiency of encapsulation and stability of capsules. Coating material may be selected from a wide range of natural and synthetic compounds, for instance: gelatin, gum arabic, starch, ethylcellulose, paraffin, dextrins [Leon, *et al*., 1990; Fang & Bhandari, 2010]. Maltodextrin (MD) is the most commonly used encapsulating agent. It has low bulk density and viscosity, forms film easily and creates a barrier from oxygen [Che Man, *et al*., 1999]. Another interesting possible encapsulating agent may be cyclodextrins (CDs). The most popular are α–, β– and γ–cyclodextrin. The central cavity of these molecules is relatively hydrophobic, while the external part of cyclodextrins is hydrophilic. This structure characteristic makes CDs able to form host-guest inclusion complexes with a wide range of organic and inorganic molecules. Inclusion complex changes physicochemical properties of guest molecules (like solubility, stabilization of labile guests, possibility of controlling volatility, changing taste and smell by masking odors, and controlled release of drugs

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and flavours). For these reasons, cyclodextrins are widely used in food and pharmaceutical industry, but also cosmetics, packing and textile industry [Astray *et al*., 2009; Del Valle, 2004].

The limitation in β-CD application as a wall material in microencapsulation by spray-drying is caused by its low water solubility (1.8%). Hydroxypropyl-β-cyclodextrins (HP-β-CDs) are modified β-CDs having a higher aqueous solubility (60%), which makes the complex formation very simple and enables the use of an appropriate drying technique such as freeze-drying or spray-drying, if required [Gould & Scott, 2005].

Compared to freeze-drying, the cost of the spray-drying method is 30–50 times lower [Desobry *et al*., 1997]. Moreover, the main drawback of co-precipitation method that has to be performed to form β-CD complexes before freeze--drying is the scale-up. The limited solubility of β-CD causes that the large volume of water needs to be used. Important cost factors may also occur taking into account tank capacity, time, and energy for heating and cooling. In the case of HP--β-CD, the complex formation is easier and may be achieved at ambient temperature.

The objective of this study was to investigate the antioxidant activity of polyphenols from lowbush *Vaccinium myrtillus* blueberry fruit juices microencapsulated by spray--drying and freeze-drying with the use of cyclodextrin as a coating agent. Attention was focused on the application of hydroxypropyl-β-cyclodextrin and β-cyclodextrin as polyphenol carriers. Maltodextrin was used as a control sample for spray-dried polyphenols.

MATERIALS AND METHODS

Plant material

Blueberries were purchased in the local market in Lodz (Poland) and kept at -20°C until use. Thermal and pectinolytic processing of pulp was performed. After short heating at high temperature (80°C) the pulp was cooled to 40°C in order to enable pectinolytic enzyme action (Pectinex Smash XXL, Novozyme). Fruit pulp was pressed with the use of a horizontal screw. Juice was frozen until further analysis.

Chemicals

Most of the chemicals used in this research, being 2,2'-Azinobis 3-ethylbenzothiazoline-6-sulfonic acid (ABTS) and N,N--dimethyl-p-phenylenediamine dihydrochloride (DMPD), 2,4,6-Tris (2-pyridyl-S-triazine) (TPTZ), 6-hidroxyl-2,5,7,8--tetramethyl-2-carboxylic acid (Trolox), $FeCl_3$, Folin-Ciocalteu reagent, HP-β-cyclodextrin and β-cyclodextrin were obtained from Sigma. Maltodextrin Maldex 150/Maltosweet 150 – DE 14–17 was produced by Brenntag. Anthocyanin standards were from Extrasynthese (France) and PhytoLab (Germany).

HP-β-CD inclusion complex and maltodextrin microcapsules by spray-drying

Two different types of coating material were tested: HP-β--cyclodextrin and maltodextrin as a control sample. For each type, 15% (w/w) ratios were added and homogenised with blueberry juice (10°Bx). Tween 80 was used as an emulsifier.

The mixtures were spray-dried in a BUCHI Mini Spray Dryer B – 290. The inlet/outlet temperatures, the drying air flow rate and dispersing air flow were constant at 140°C/70°C, 75% and 50 mm of rotameter, respectively. The 0.7 mm diameter dispersing nozzle was used.

β-CD inclusion complex by freeze-drying

β-CD in 15% (w/w) ratio was added to hot (75°C) blueberry juice in order to increase its solubility. The mixture was cooled to 4°C under continuous stirring. The precipitated product was separated by filtration and freeze-dried. The Christ Delta 1–24LSC freeze dryer was used. The samples were slowly frozen at −50°C. Pre-drying was performed by keeping the product at a pressure of 0.42 mbar at 30°C. Secondary drying was carried out by reducing the pressure to 0.05 mbar and increasing the shelf temperature to 40°C.

Microparticles characterisation

Morphology of the microcapsules was examined using a scanning electron microscope (HITACHI). Microcapsules were coated with Au/Pd and observed at different extensions.

HPLC analysis

The analysis was carried out on the basis of the method performed by Gavrilova *et al*. [2011] with few modifications. All extracts were analysed by HPLC (Spectra System SN4000) on a 150 x 4.6 mm C18 column (Acclaim). The solvents were (A) $HCOOH/H_2O$ (1:20) and (B) CH_3CN/H_2O (20:1). Spectral data from all peaks were accumulated in the range of 200—700 nm, and chromatograms were recorded at 280 nm, at 320 for conjugated forms of hydroxycinnamic acids, and at 520 nm for anthocyanins.

Analysis of total polyphenols

Total phenols were measured using a UV spectrophotometer (Celil, CE 2041), based on a colorimetric oxidation/reduction reaction as described by Škerget *et al.* [2005]. The results were expressed as mg of gallic acid per g of powder (mg GA/g powder).

Analysis of antioxidant capacity

ABTS radical-scavenging system

Radical scavenging activity against $ABTS^{+\bullet}$ was determined spectrophotometrically according to Rivero-Perez *et al.* [2008].

DMPD radical-scavenging system

One milliliter of 100 mmol/L DMPD solution in distilled water was mixed with 100 mL of 0.1 mol/L acetate buffer (pH 5.25). Adding 0.2 mL of a 0.05 mol/L ferric chloride solution resulted in purple radical cation formation. Two mL of DMPD radical solution were added to 0.1 mL of polyphenol preparation and incubated for 10 min in darkness. Absorbance of this assay was measured at 505 nm [Fogliano *et al*., 1999].

FRAP "ferric reducing/antioxidant power"

The method described by Rivero-Perez *et al.* [2008] with few modifications was used. The reaction mixture was prepared by adding of 2.5 mL of TPTZ (10 mmol/L), 2.5 mL of $FeCl_3$, 25 mL of acetate buffer (0.3 mol/L, pH 3.6)

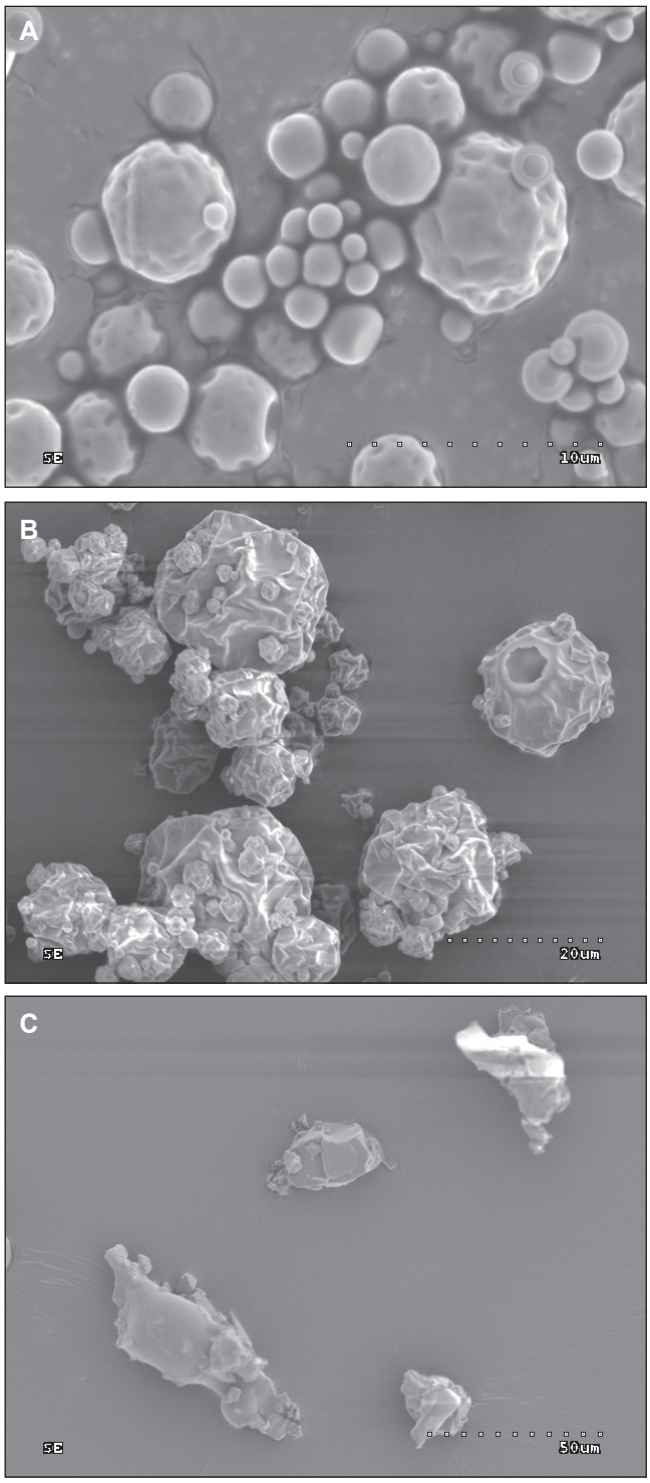

FIGURE. 1. Scanning electron microscopic photographs of microcapsules for: MD – magnification x 5000 (A), HP-CD – magnification x 1500 (B), CD – magnification x 700 (C).

and 3 mL of water. The sample (300 μL)was incubated with 900 mL of the latter mixture at 37°C for 30 min. Absorbance of this assay was measured at 593 nm.

Statistical analysis

Data was reported as mean ± standard deviation for determinations by triplicate. Analysis of variance (one-way ANOVA) was conducted to determine the differences between the microencapsule samples with respect to polyphenolic compound quantity and antioxidant activity. Tukey's Honestly Significant Differences test was used to calculate the mean separations.

TABLE 1. Product yield of spray-drying and freeze-drying process for different carriers.

	Spray-drying		Freeze-drying
	HP-β-CD	MD	β-CD
Total solids in the drying slurry (g)	105.1	105.1	105.1
Product (g)	46.2	45.9	82.0
Yield (%)	43.9	43.7	78.1
Moisture (%)	3.2	3.3	3.9

RESULTS AND DISCUSSION

Microparticles characterisation

The analysis of micrographs taken by scanning electron microscopy revealed the formation of 2–20 μm in size, well-formed microcapsules in samples obtained by spray-drying, and the formation of amorphous material in the process of freeze-drying (Figure 1). Spray-dried polyphenols were encapsulated by the MD within a typical morphology for microcapsules, with a little dented, rounded outer surface, without cracks or pores. In the case of HP-CD microcapsules, the concave surfaces were probably produced as a result of the liquid drop shrinkage due to rapid moisture loss during the early stages of spray-drying. Similar morphology was observed in polyphenol preparation of different spray-dried cactus pear cultivars using maltodextrin (DE=10) as an encapsulating agent [Saenz *et al*., 2009; Diaz *et al.,* 2006]. Nevertheless, to the best of our knowledge, there is no previously reported analytical data concerning the morphology of spray-dried polyphenols on hydroxypropyl– β–cyclodextrin.

Product yield of drying process

The selected spray-drying process conditions resulted in satisfying product yield (Table 1). Spray-drying yield was very similar for both carriers. For HP-β-CD it was equal to 43.9%, while for MD it was 43.7%. In both cases, water content after drying was low – below 2%. The freeze-drying process resulted in 78.1% product yield. The decrease of product yield for spray-dried samples is connected with the fact that only some part of powder was sent to the receiver; while the rest remained on the walls of the drier and cyclone. Some amount of powder was lost due to the fact that it was not separated in cyclone and was blown with air and remained on the filter. According to literature, the efficiency of spray-drying in the case of MD is similar. In studies performed by Tonon *et al*. [2008], where fruits of açai were microencapsulated on MD by spray-drying, the yield did not exceed 48.4%. In the case of drying *Morinda citrifolia* L. with different concentrations of MD, the yield was between 5.1 and 48.1% [Krishanaiah *et al*., 2009].

TABLE 2. Polyphenolic compounds composition of blueberry microcapsules.

Compound	Microcapsules			Juice (mg/L)
	HP-β-CD (mg/100 g)	MD (mg/100 g)	β-CD (mg/100 g)	
Anthocyanins				
Delphinidin 3-galactoside	178.12^{a}±0.13	159.23^{ab}±15.88	137.83^{b} ± 2.31	502.79±5.21
Delphinidin 3-glucoside	218.58^{a}±16.06	196.69^{ab}±26.15	179.24^{b} ± 3.20	638.97±4.12
Cyanidin 3-galactoside	91.88^{b}±2.28	88.96^{b}±10.14	93.04^{b} ± 5.84	299.51±1.66
Delphinidin 3-arabinoside	119.23^{b}±6.46	120.67^{b}±17.81	93.10^{a}±0.11	380.03±2.38
Cyanidin 3-glucoside	124.10^{b}±0.53	120.68^{b}±10.17	102.23^{a}± 2.34	358.28±1.98
Petunidin 3-galactoside	70.30^{a}±2.69	72.12^{a}±6.42	52.51^{b}±1.10	206.07±1.54
Petunidin 3-glucoside	101.11^{a}±10.31	99.54^{a}±8.63	87.59^{a}±2.69	351.30±1.04
Peonidin 3-galactoside	98.30^{a}±11.69	97.66^{a}±4.37	67.66^{b}±3.58	238.19±2.57
Petunidin 3-arabinoside	17.37^{a}±0.28	18.54^{a}±0.52	15.40^{a}±0.20	57.08±0.95
Peonidin 3-glucoside	29.61^{a}±0.69	30.91^{a}±1.63	22.14^{b}±2.75	93.37±2.15
Malvidin 3-galactoside	98.65^{a}±2.32	108.04^{a}±5.39	77.69^{b}±0.90	116.65±0.56
Malvidin 3-glucoside	129.09^{a}±2.12	131.99^{a}±9.72	97.24^{b}±3.44	450.67±1.44
Peonidin 3-arabinoside	9.04^{a}±0.62	11.22^{a}±1.39	6.24^{b}±0.95	16.11±0.00
Malvidin 3-arabinoside	26.31^{a}±0.61	27.93^{a}±1.23	18.43^{b}±2.15	94.23±0.75
Hydroxycinnamic acids				
p-Coumaroylguinic acid	20.99^{a}±0.98	22.16^{a}±0.51	16.80^{b}±0.19	63.82±1.41
Chlorogenic acid	50.90^{a}±2.30	52.38^{a}±0.61	42.01^{b}±1.70	151.09±3.77
Ferulic acid derivative	60.90^{a}±6.42	63.39^{a}±5.40	51.24^{b}±4.09	191.58±6.65

Values are shown as means±SD (n=3).

The lowest moisture, which means better preservation and stability of microcapsules, was found using the spray--drying technique (3.2% for HP-βCD and 3.3% for MD). The moisture content achieved in freeze-dried preparation was 3.9%, which confirmed that this technique was less effective at removing water. The lower moisture level in the spray--dried samples is explained by the intense contact between the hot drying air and microparticles.

Polyphenols content

High quality blueberry juice was used as raw material for microcapsules preparation. According to the analytical results, the blueberry juice contained 7.44 g/L of total polyphenolic compounds, where its anthocyanins content was determined as 3.80 g/L. Similar results were reported by Lee *et. al.* [2004a, b] for different Vaccinium species, where the concentration of total phenolics ranged from 489 to 702 mg/100 g

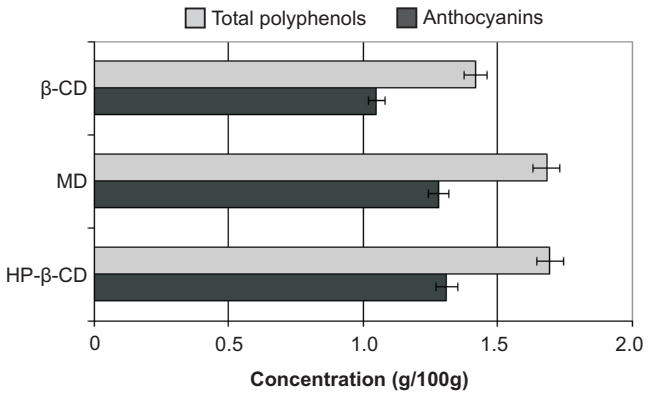

FIGURE 2. Contents of anthocyanins and total polyphenols in microcapsules.

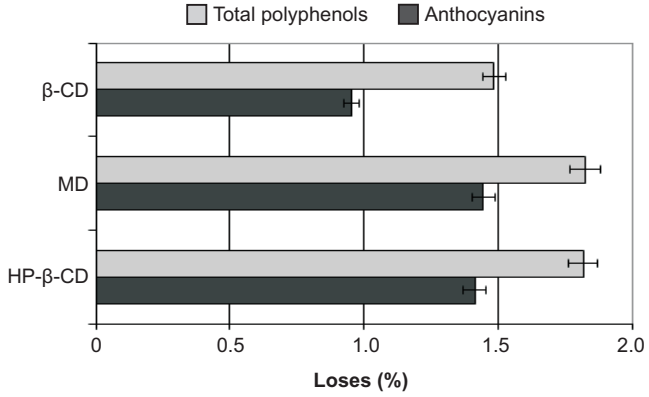

FIGURE 3. Total losses of anthocyanins and total polyphenols during different drying procedures.

and that of total anthocyanins from 176 to 311 mg/100 g, finding a great variability.

The results of HPLC assay of blueberry juice and the obtained powders are shown in Table 2. A total of 14 anthocyanins were detected across all microencapsulated samples as well as in raw blueberry juice. Delphinidin and malvidin derivatives were the most representative forms in all samples**.** Total anthocyanin contents of spray-dried samples were quite homogenous, while in freeze-dried powder the concentration of anthocyanins was lower by 20% (Figure 2). HP-β-CD microcapsules showed the highest total anthocyanins (1.31 g/100 g), among which delphinidin and peonidin derivatives were found in much higher concentrations than in other samples. In overall, the ratio of anthocyanins to total polyphenols per 1 g of the microcapsules did not show high variability, as it ranged from 74.1% for β-CD to 77.3% for HP--β-CD. Data shows that the total phenolics contents were also in a narrow range from 1.41 to 1.69 g/100 g and the spray--dried samples represented the highest values. In the case of studies carried out by Jimenez-Aguilar *et al*. [2011], contents of total phenolics and anthocyanins in ethanolic extract of *Vaccinium ashei* blueberry spray-dried with the use of mosquite gum ranged from 1.82 and 1.97 g/100 g of powder, and from 1.25 to1.35 g/100 g of powder, respectively. This result is very similar to our findings.

Comparing the concentration of polyphenols in juice slurry before encapsulation and in encapsulated preparations, it was possible to estimate the overall losses of those compounds during processes of spray- and freeze-drying. Taking into account product yield levels and total amount of received powder, the individual determination of anthocyanin and total polyphenol losses put in evidence some more differences between spray-dried and freeze-dried microparticles. In particular, the freeze-dried powders showed much more satisfying retention values of anthocyanins when comparing the total amount of the obtained powder, for which it was about 1.5 times higher than for the spray-dried ones (Figure 3). Similar values of total polyphenol losses were counted for both of the spray-dried samples. The total polyphenolics decrease in spray-dried samples was only 15% higher than in the freeze-dried ones. In the case of total phenolics, about 73% of these compounds were lost on average during spray--drying, whereas in the case of anthocyanins the loss was about 57%. These results indicated that high inlet gas temperature and lower drying product yield had a significant effect on the total polyphenols preserved in the overall quantity of the obtained spray-dried product.

TABLE 3. Antioxidant and antiradical activity of microencapsulated blueberry polyphenols.

Antioxidant activity	HP-β-CD	MD	β-CD
ABTS (μmol/L TE/g)	105.21^{a}±1.71	94.93^{b}±1.33	104.55^{a}±1.28
DMPD (mmol/L TE/g)	1.83^{a}±0.02	1.65^{b}±0.01	1.80^{a}±0.01
FRAP (mmol/L TE/g)	79.70^{b}±2.80	77.27^{b}±1.53	67.28^{a}±1.59

Values are shown as means±SD (n=3).

Antioxidant activity

Table 3 presents analytical data of antioxidant and radical scavenging activity of microencapsulated blueberry polyphenols with the use of different coating materials. Due to high polyphenolic content, all dried preparations showed a strong antioxidant and antiradical activity. The spray-dried microcapsules showed the highest total antioxidant activity, which was more correlated to the total phenolic content rather than to anthocyanin concentration. The results were consistent with the lower anthocyanin to total phenolics ratio in the obtained powders. This is in agreement with the fact reported by other studies [Jimenez Aguilar *et al*., 2011], that anthocyanins have lower antioxidant activity with respect to other polyphenolic compounds. Radical scavenging activity studies demonstrated a significant retention of the antioxidant activity after encapsulation by the spray-drying as well as by freeze-drying process. According to the obtained TE values, the highest antiradical activity was determined for both cyclodextrin preparations tested.

CONCLUSIONS

Higher product yield was obtained for freeze-dried microcapsules.

Microencapsulated samples were characterised by high diversity of polyphenolic compounds, especially anthocyanins which are the highest in amount when encapsulated with the use of the spray-drying technique.

Both types of β-CDs provide better protection for polyphenol antiradical activity than maltodextrin.

Polyphenol preparations of high antioxidant potential microencapsulated in β-CD and HP-β-CD with the use of spray-drying as well as freeze-drying technique described in this research could have a promising application in food and pharmaceutical industry and could become an attractive food additive for incorporation into functional foods and provide health benefits due to their high antioxidant potential.

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