Microencapsulation of Anthocyanins by Spray Drying; a Review

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***Abstract*— there has been an increased interest in the development of food colorants from natural sources as alternatives to synthetic dyes because of both legislative actions and consumer concerns. Anthocyanins emerge as a great interest for the food industry since they give *a wide range of colors* as well as nutraceutical activities. Nevertheless, due to a low stability to environmental conditions during processing and storage, introducing those compounds into foods is challenging. Microencapsulation may be an efficient way to introduce such compounds into those products. Hence, this review will focus on microencapsulation of anthocyanins using spray drying to develop natural colorant pigments that possess high stability, solubility and dispersibility. This review refers to updated information regarding microencapsulation of anthocyanins by spray drying, as well as its effectiveness, developments and optimized conditions are discussed.**

Key words: Anthocyanin; stability; microencapsulation; natural color; spray drying

I.INTRODUCTION

Color is one of the most important quality attributes affecting the consumer's acceptance of food since it gives the first impression of food quality. There is a worldwide trend towards the use of natural additives in general, and food colorants in particular, in food applications. The interest of the food industry in natural colorants replacing synthetic dyes has increased significantly over the last years, mainly due to safety issues. The use of natural pigments requires knowledge of a chemical knowledge structure of their molecules and stability in order to adapt them to the conditions of use during processing, packaging and distribution [1].The industry requires technologies which protect the natural pigments in the environment f, due to their instability in the presence of light, air, humidity and high temperatures. Currently, in order to provide this protection, one alternative is microencapsulation technology [2]

Microencapsulation is a rapidly expanding technology which is a unique way to package materials in the form of micro- and nano-particles and defined as a process to entrap one substance (active agent) within another substance (wall material) [3]In food industry, it involves the incorporation of natural ingredients, polyphenols, volatile additives, colors, enzymes, and bacteria in small capsules, giving them the chance to be stabstabilize themle, protected and preserved against nutritional and health losses. In addition, microencapsulation can simplify the food manufacturing processes by converting liquids to solid powder, decreasing production costs by allowing batch processing using low cost, powder handling equipment.Most microcapsules are small spheres with diameters comprised between a few micrometers and a few millimeters. In the simplest form, a microcapsule is a small sphere with a uniform wall around it. The material inside the microcapsule is referred to as the core, encapsulants, internal phase, payload phase or fill, whereas the wall is sometimes called shell, coating, wall material, encapsulating agents or membrane shell, carrier material, external phase, or matrix[4]In this review, only ‘‘core’’ and ‘‘wall’’ will be used to refer to the encapsulated ingredient and encapsulating agent, respectively.

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II.ANTHOCYANIN

Anthocyanins (Greek anthos, flower and Greek kyanose, blue) are the largest group in the plant kingdom and generally accepted as the largest and most important group of water-soluble pigments in nature. They are responsible for the blue, purple, red and orange colors of many fruits, flowers, and other parts of plants. Anthocyanins are glycosides of anthocyanidins (also called aglycones) and sugars. There are about 17 anthocyanidins found in nature, but More than 90% of all anthocyanins isolated in nature are based only on the following six anthocyanidins: pelargonidin (plg), cyanidin (cyd), peonidin (pnd), delphinidin (dpd), petunidin (ptd), and malvidin (mvd), as shown in Table 1 [5, 6]Natural anthocyanins have powerful coloring properties as only small doses of anthocyanins are required to display the color desired in several food matrixes (e.g.30–40 ppm for soft drinks and 20–60 ppm for fruit preserves). Generally, natural colorants have higher coloring capacities than synthetic ones [7]Commercial applications of anthocyanins as food colorants include soft drinks, fruit preserves (jams, canned fruits), sugar confectionary (jellies), dairy products (essentially yogurts), dry mixes (acid dessert mixes and drink powders) and more rarely frozen products (ice cream) and few alcoholic drinks. Among these applications, soft drinks have been the main and ideal target for the use of anthocyanins as colorant. The interest in anthocyanin pigments and scientific research have increased in recent years due not only to give color to products that contain them but to their probable role in nutraceutical and health benefits mainly as natural antioxidants[2, 8]Despite the advantages of anthocyanins as potential substitutes of synthetic colors, due to their particular chemical structure, anthocyanins are unstable and susceptible to degradation. the stability of anthocyanins is affected by factors include structure and concentration, pH, temperature, light, presence of copigments, self association, metallic ions, enzymes, oxygen, ascorbic acid, sugar and their degradation products, proteins and sulphur dioxide. More particularly, the bioavailability of anthocyanins is low due to their sensitivity to changes in pH. Anthocyanins are generally stable at pH values of 3.5 and below, and are therefore stable under stomach conditions. However, they degrade at higher pH values, such as those more typical for the intestinal tract (pH of 7) and thus nutritional value is greatly reduced. Therefore, This high instability of anthocyanins has a direct consequence on possible color stabilization actions and need to be stabilized to preserve the product's original color and its potential health benefits. For this reason many studies have been conducted with the aim to increase the stability of these substances. among them encapsulation is one of the main techniques to increase stability.

III.MICROENCAPSULATION OF ANTHOCYANINS

As previously mentioned, the major problem associated with the storage of anthocyanins is their instability. So, the stabilization of anthocyanins is the source of many works. Among the existing stabilization methods of anthocyanins, encapsulation is an interesting means. The utilization of encapsulated anthocyanins instead of free compounds can overcome the drawbacks of their instability as well as improve the bioavailability of anthocyanins. Encapsulation facilitates light- and heat-labile molecules like many pigments, such as anthocyanins, to maintain their stability and improve their shelf lives and effects. It is a rapidly expanding technology, highly specialized, with affordable costs. According to Cavalcanti et al., 2011, encapsulation is one of the main techniques to increase stability of anthocyanins.[9] An important step in developing microcapsules is the selection of a wall material that meets the required criteria. In fact, microencapsulation efficiency and microcapsules stability are largely dependent on wall material composition. This wall could act as a barrier and it may protect against oxygen, water, light or could avoid

contact with other ingredients or control diffusion [10]Wall material properties include :Wall material properties include

1. Stabilization of core material.

2. Inert toward active ingredients.

3. Controlled release under specific conditions.

4. Film-forming, pliable, tasteless, stable

5. Non-hygroscopic, no high viscosity, economical.

6. Soluble in an aqueous media or solvent, or melting.

7. The coating can be flexible, brittle, hard, thin etc.

A number of commercially approved wall materials are available to produce microencapsulated anthocyanins like gum Arabic, maltodextrin, inulin, tapioca starch , citrus fibre, glucose syrup , soy protein isolate, whey proteins and other wall materials like cold-set glucan gel or a heat-set curdlan gel etc. Not all wall materials meet all the properties needed, so they are often used in combination with other wall materials and other modifiers such as oxygen scavengers, antioxidants, chelating agents and surfactants .According to the Zhang, et al, 2005, it is better that for encapsulation of an oil-soluble core materials, water-soluble wall materials been used. In contrast, the water-soluble core materials require wall materials with oil-solubility therefore lipids can be good wall materials to embed the anthocyanins. [11] For this reason (Ge, Wan, Song, Fan, & Wu, 2009) used molten mixtures of beeswax and stearic acid as wall materials for microencapsulating hybridrose red pigments.

There are many encapsulation techniques; among which some have been successfully applied to anthocyanins (see Table 2). The selection of a microencapsulation method depends upon specific applications and parameters such as required particle size, physicochemical properties of the core and coating materials, release mechanisms, process cost, etc. [12-14]In general, encapsulation has increased the stability of anthocyanins independently of the encapsulation technique used; in contrast the degree of stabilization seems to be directly related to the employed encapsulation technique conditions.

A. Spray-drying

Spray-drying is a unit operation which is the continuous transformation of feed from a fluid state into dried particulate form by spraying the feed into a hot drying medium. There are three fundamental steps involved in spray drying [15]:

1) Atomization of a liquid feed into fine droplets,

2) Mixing of these spray droplets with a heated gas stream, allowing the liquid to evaporate and leave dried solids,

3) Separation of dried powdpowderer from the gas stream and collection.

Spray drying involves complex interactions of process, apparatus and feed parameters which all have an influence on the final product quality. The spray-dried products have important properties like uniform particle size, nearly spherical regular particle shape, excellent flowability, improved compressibility, low bulk density, better solubility, reduced moisture content, and increased thermal stability, and suitability for further applications. Such product characteristics majorly depend on the physical properties of feed, equipment components and processing parameters. By modifying the spray drying process, it is possible to alter and control the mentioned properties of spray-dried powders.{Jafari, 2007 #97;Kandansamy, 2012 #105;Reineccius, 2004 #183}.Spray drying is the most commonly used encapsulation technique in the food industry It is also one of the oldest encapsulation methods and has been used in the food industry since the late 1950s because there are several advantages for this technique such as low operating cost, high quality of capsules in good yield,

rapid solubility of the capsules, small size, high stability capsules, and continuous operation [16, 17]After selecting the suitable wall material, it is hydrated in water. For water soluble materials like anthocyanins, the ingredient to be encapsulated is added to the wall material and homogenized. The ratio of core to wall is usually 1:4 [18], but this can be optimized for each individual ingredient. This mixture is then fed into the spray dryer and atomized with a nozzle or spinning wheel. Water is evaporated by the hot air (100-160oC) and the small particles are deposited to the bottom of the spray dryer where they are collected. The resulting microcapsules are then transported to a cyclone separator for recovery. [17, 19]During the drying process, a film is formed at the droplet surface and the concentration of ingredients in the drying droplet increases. Finally, a porous, dry particle is formed. Critical parameters of spray drying are inlet and outlet temperature of air, viscosity of feed, solid content, surface tension, feed temperature, volatility of solvent, and nozzle material.

The choice of a wall material for microencapsulation by spray drying is very important for encapsulation efficiency and microcapsule stability. Typical wall materials generally available and suitable for spray drying microencapsulation include natural gums (gum arabic, alginates, carrageenans, etc.), proteins (dairy proteins, soy proteins, gelatin, etc.), carbohydrates (maltodextrins and cellulose derivatives) and/or lipids (waxes, emulsifiers). According to Zuidam and shimoni., (2010), maltodextrin is extensively used as wall in spray drying as to satisfy demand and low cost. [20]Maltodextrins turned out to be the best thermal defenders, essential to preserve the integrity of the anthocyanins during their encapsulation [21, 22]Nowadays, maltodextrin is commonly mixed with gum arabic. A mixture of maltodextrin (60%) and gum arabic (40%) has been used for encapsulation of procyanidins from grape seeds [23]No change of procyanidins was observed during the critical drying stage, the rate of encapsulation was around 85 %, and their stability was improved. Another wall material successfully used for encapsulation of anthocyanins was protein-lipid (sodium caseinate-soy lecithin) emulsion, which has been used in spray drying of grape seed extract [24]Chiou and Langrish ,2007 introduced citrus fruit fiber as a wall material for spray drying of bioactives extracted from *Hibiscus sabdariffa* L.[25] The main bioactive compounds in H sabdariffa L. extract are polyphenols, more specifically, the anthocyanin complexes. The presence of the bioactive material in the fibers did not appear to significantly affect the product size or shape. The results demonstrated that natural fruit fibers might be a potential replacement carrier for spray drying sticky materials[25]Encapsulation efficiency could be maximized by the right choice of spray drying parameters including inlet and outlet drying air temperatures, infeed temperature, atomization type and conditions, drying air flow rate and humidity, and powder particle size [26, 27]According to Liu et al. (2004), the optimum inlet air temperature for anthocyanin microencapsulation was 120℃ and outlet air temperature was 80℃ [28]Table 3 summarizes experimental conditions that have been recently optimized for the encapsulation of different anthocyanins by spray-drying. The best spray-drying conditions are a compromise between high air temperature, high solid concentration of the solution, and easy pulverization and drying without expansion and cracks of final particles .

Degradation kinetics and color stability of spray-dried encapsulated anthocyanins from roselle *(Hibiscus sabdariffa*) was studied by Idham et al. (2010). In this study, four different matrices, i.e., maltodextrin, gum Arabic, combination of maltodextrin and gum Arabic, and soluble starch were used for wall materials. The stability of encapsulated pigments was investigated during storage under three different storage temperatures (4, 25 and 37°C) until 105 days. The four type of matrices largely increased the half-life of the pigments during storage especially at

37°C ($P < 0.05$) compared with the non-encapsulated roselle extract[29]Degradation studies in many works indicated that encapsulated extract by spray drying were more stable to light and temperature than the free extract, thus stability of encapsulated anthocyanins was improved . Burin, et al.,(2011) used Maltodextrin, maltodextrin／c-cyclodextrin and maltodextrin／arabic gum and the combination of maltodextrin／arabic gum as wall materials for Cabernet Sauvignon Grapes and the combination of maltodextrin／arabic gum presented the longest half-life time and lowest degradation constant for all the conditions evaluated. The stability of the anthocyanins added to the isotonic soft drink system was studied under different temperature and light conditions. The degradation of the anthocyanins fitted a firstorder reaction model under all conditions evaluatedused [30]. Maltodextrin, maltodextrin／c-cyclodextrin and maltodextrin／arabic gum and the combination of maltodextrin／arabic gum as wall materials for Cabernet Sauvignon Grapes and the combination of maltodextrin／arabic gum presented the longest half-life time and lowest degradation constant for all the conditions evaluated. The stability of the anthocyanins added to the isotonic soft drink system was studied under different temperature and light conditions. The degradation of the anthocyanins fitted a first order reaction model under all conditions evaluated. Table 4 summarizes some examples of anthocyanin encapsulation from diverse sources utilizing spray drying. Radical scavenging activity studies demonstrated a significant retention of antioxidant activity after encapsulation by the spray-drying process [31-33]Optical microscopy and particle size distribution analysis indicated that the encapsulated particles all had spherical morphology and uniform size distribution [31, 32, 34, 35]Although spray dryers are widespread in the food industry, there are several disadvantages of this technique such as:

1- Production of no uniform microcapsules
2- Limitation in the choice of wall materials (low viscosity at relatively high concentrations)
3- Production of very fine powders which needs further processing
4- Not good for heat-sensitive materials [17, 36]

IV. Conclusion and Juture Trends

The use of anthocyanins may show benefits over that of synthetic colors. However, the use of these colorants in food products may face some problems due to their stability during storage caused by temperature, oxygen and light. In order to overcome the instability problem, which results in restricted commercial applications, from the literature, it is clear that the utilization of encapsulated anthocyanins, can help to increase shelf life and protecting the biological properties of the anthocyanins. In this review, the results of recent studies implementing spray drying techniques applied to anthocyanins extracts confirmed that encapsulation is an interesting means to potentialize their activity. Spray-drying is the most common technique used to encapsulate anthocyanins and about 80–90% of encapsulates are spray-dried ones. Other techniques include freeze-drying, Emulsification, gelation etc. There are still some technologies not being applied for these special anthocyanins, including Spray Cooling/Chilling, Inclusion complexation, Liposome Entrapment, Melt injection, Rotational suspension separation, and nanoencapsulation.

Table1. Chemical structures and characteristics of six naturally occurring common anthocyanidins

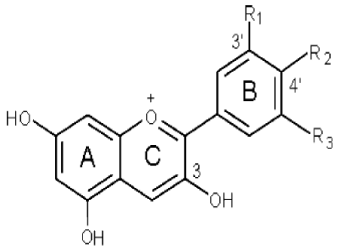
Anthocyanidin	Basic structure	R_1	R_2	R_3	Main color	Examples of sources
Cyanidin		−OH	−OH	−H	reddish-orange	Apple, elderberry, blackberry, nectarine, plum, peach, red cabbage
Delphinidin		−OH	−OH	−OH	purple, blue	Grape, beans, eggplants
Pelargonidin		−H	−OH	−H	orange,	Strawberry, red radishes, some beans
Malvidin		$-OCH_3$	−OH	$-OCH_3$	purple	Grape
Peonidin		$-OCH_3$	−OH	−H	purplish-red	cranberries, blueberries, plums, grapes**,** cherries purple corn
Petunidin		−OH	−OH	$-OCH_3$	dark-red or purple	Grape, red berries

Table2. Overview of common microencapsulation processes for anthocyanins and their characteristics

Technology	Process steps	Particle size (μm)	Advantages	Disadvantages	Ref.
Spray-drying	1. dissolve core material in aqueous wall material solution 2. Atomize 3. Dehydrate	10–400	1. Low process cost 2. Wide choice of wall material 3. good encapsulation efficiency 4. good stability of the finished product 5. possibility of large-scale production in continuous mode	1. Can degradate highly temperature-sensitive compounds 2. control of the particle size is difficult 3. yields for small batches are moderate	(Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007; Gouin, 2004; Reineccius, 2001)
Freeze Drying	1. Dissolve core material and wall material in water 2. Freeze the sample 3. Drying under low pressure 4. Grinding (option)	20–5,000	Thermosensitive substances that are unstable in aqueous solutions may be efficiently encapsulated by this technique	1. Long processing time 2.expensive process costs 3.Expensive storage and transport of the capsules	(Madene,Jacquot, Scher, & Desobry, 2006)
Fluid bed coating	1. Fluidize active powder 2. Spray coating 3. Dehydrate or cool	20–200	1.Low cost process 2. it allows specific capsule size distribution and low porosities into the product	Degradation of highly temperature- sensitive compounds	(Dewettinck & Huyghebaert, 1999)
Emulsification	1. Dissolve active and emulfiers in water or oil phase 2. Mix oil and water phases under Shear	0.2–5,000	Polar, non-polar (apolar), and amphiphilic can be incorporated	1.limited number of emulsifiers that can be used 2. Difficult control of the capsule formation	(Betz and Kulozik)
Melt extrusion	1. Melt the wall material 2. dissolve active in the coating 3. Extrude with extruder 4. Cool	300–5,000	1. The material is totally surrounded by the wall material 2. Any residual core is washed from the outside 3. It is a relatively low-temperature entrapping method	1. The capsule must be separated from the liquid bath and dried 2. is difficult to obtain capsules in extremely viscous carrier material melts	(Nedovic, et al., 2011)

Encapsulation by rapid expansion of supercritical fluid (RESS)	1. Create a dispersion of core material and dissolved or swollen wall material in supercritical fluid 2. Release the fluid to precipitate the shell onto the active	10–400	1.non-toxicity and easy removal of the solvent 2. operation at low temperatures and in an inert atmosphere that allows avoiding degradation of the product	1.both the core and the wall material must be very soluble in supercritical fluids 2.low or no solubility of high molecular weight, polar compounds CO_2 3.poor control over the precipitated crystal morphology, size distribution	(Cocero, Martín, Mattea, & Varona, 2009)
Ionic Gelation	1.wall material with dissolved core material is extruded as drops within an ionic solution 2. The capsules are formed by ionic interaction		Organic solvents and extreme condions of temperature and pH are avoided	1.Mainly used on a laboratory scale the capsules 2. in general, have high porosity which promotes intensive burst	(Santos & Meireles, 2010)
Thermal Gelation	The principle is almost the same of ionic gelation' principle, nonetheless there is no necessity of an ionic solution to form a gelled drop, the gelation is only due to thermal parameters		The same of ionic gelation	The same of ionic gelation	(Ferreira, Faria, Grosso, & Mercadante, 2009)

Anthocyanin's source	Wall material	Operation Conditions			Encapsulation efficiency (%)	Referenc
		Inlet air T (°C)	Outlet air T (°C)	feed flow rate		
black carrot	A range of maltodextrins [Stardri 10 (10DE), Glucodry 210 (20–23DE) and MDX 29 (28–31 DE)]	160,180, 200	107±2, 118±2, 131±2	5 ml/min	NR[a]	(Ersus & Yurdagel, 2007)
Garcinia indica	maltodextrin of various dextrose equivalents (DE 06, 19, 21, and 33) and gum acacia and tricalcium phosphate	150	80	NP	NR	(Nayak & Rastogi, 2010)
Berberis	Maltodextrin,β-cyclodextrin, gum acacia	160	80	NP	90-95	(Huang & Yang, 2011)
Opuntia stricta	Glucose syrup (DE 29)	160	variable : 50-68	0.72 l/h	NR	(Obón, Castellar, Alacid, & Fernández-López, 2009)
blueberry	mixture of soybean protein isolation(SPI)-maltodextrin and β-cyclodextrin-maltodextrinn-arabic gum	120	80	400r / h	92.48	(Ying-chang, Chun-mao, Xian-jun, Yong, & Na21, 2010)
Andes berry	maltodextrin DE 20	230	150	10 mL/min	NR	(Olaya, Castano, & Garzon, 2009)
butterfly pea	Hydroxyl propyl methyl cellulose (HPMC) and gelatin	130	80	10 ml/min	NR	(Tantituvanont, Werawatganone, Jiamchaisri, & Manopakdee, 2008)
Pomegranate	maltodextrin (MD) or soybean protein isolates (SPI)	140 to 160 ± 5	100 to 140 ± 5	10 mL min)1	SPI = 35.8–100% MD= 89.4–100	(Robert et al., 2010)
Proanthcyanidins	arabic gum and maltodextrin	180	88	NP	88.84	(Lianfu, Yanshan, & Dehua, 2006)
urucum	chitosan	180 ± 5	100 ± 5	NP	NR	(Parize et al., 2010)
Roselle-Pineapple Juice	Three different maltodextrin DE 10 concentrations (3%, 5% and 10% w/w)	140, 160, 180 and 200	80	NP		(Osman & Endut, 2009)
Tamarillo	maltodextrin DE 20	230	150	10 mL/min	NR	(Olaya et al., 2009)
red prickly	glucose	120-160	120	0.72 l/h	NR	(Obón et al., 2009)

pear	syrup					
Parthenocissus tricuspidata	soluble starch	175 −180	60−70	3 mL/min	NR	(DONG, BU, & LI, 2010)
açai juice	maltodextrin 10DE, maltodextrin 20DE and gum Arabic	140, 170 and 200C	82 ± 2 97 ± 2 112 ±2	15 g/min	NR	(Tonon, Brabet, & Hubinger, 2008)

[a]NR: not reported

Table 4. Examples of stability improvement by spray drying encapsulation.

Anthocyanin source	Stabilization improvement	Reference
Apple pomace	The stability of anthocyanins increased. The improvement in the shelf life was attributed to reduction of water activity	(Delgado-Vargas *et al*. 2000)
Black currant	high storage stability at 8 ◦C and 25 ◦C for 12 months	([37]
curcumin	The stability against light, heat, pH was effectively improved and its solubility was increased greatly.	[38]
Opuntia stricta fruits	The stability of anthocyanins increased.	[39]
Berberis kaschgarica	Athocyanins from the fruits of *Berberis kaschgarica* Rupr showed an increase in the stability towards light, temperature, carbohydrates, reducing agents, oxidants and metal ions after microencapsulation.	[40]
black carrot	Storage at 4° C increased half life of spray dried anthocyanin pigments 3 times according to 25° C storage temperature.	[21]
hybrid rose	stability of the pigments under different pH, light and heat was significantly enhanced.	[41]
grape	An increase in the storage temperature from 4 to 25 °C led to a change in the characteristic color index (bright red) of anthocyanins, and a darkening to brick red was noted.	[42]
açai (Euterpe oleracea Mart.) juice	stability at different temperatures (25 and 35°C) and water activities (0.328 and 0.529) was improved. Anthocyanin degradation exhibited two first-order kinetics: the first one, with higher reaction rate constant, up to 45–60 days of storage, and the second one, after this period, with lower degradation rate	[43]

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