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Extraction, Microencapsulation, Color Properties, and Experimental Design of Natural Pigments Obtained by Spray Drying

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Abstract
Carotenoids, chlorophylls, betalains and anthocyanins from natural sources have gained popularity due to the benefits to the health of consumers and their multiple uses in the food and other industries. Specifically in the food industry, these pigments are used or could be used as food colorants; however, their use could be affected by environmental factors endangering their stability. Microencapsulation by spray drying is a technique that helps to preserve pigments after incorporating a coating or carrier agent that protects and eases their integration to foods. This review describes the different steps (extraction, pretreatments of the extract, homogenization of the encapsulated agent, spray drying, and stability of the powder obtained) by which microencapsulated pigments can be obtained from different natural sources. In addition, mathematical methods are analyzed to explore how the different parameters affect the drying associated responses. The use of some common and uncommon encapsulating agents is also discussed. It is also mentioned the obtention of some pigments with the spray drying technic. Finally, a section about the uses of microencapsulated pigments in recent years is included.

Keywords Spray drying · Microencapsulation · Natural pigments · Drying steps · Stability of pigments · Color

Introduction

Color in foods is related to consumer expectations (appearance, flavor and aroma) and intensity of flavors or any other attribute such as sweetness [1–4], acidity, and saltiness, among many others sensory attributes. Color is a physical characteristic of acceptability and is involved in the sensation of pleasure and in the evocation of emotions or people's feelings [5, 6]. The intensity or saturation of the color acts as a quality factor [7]. However, most processes affect the food color in some part of the process [7]. Therefore, in many cases, the addition of food colorings is necessary to standardize, recover, improve, or increase the color of foods and beverages [8, 9]. Currently, the two recognized classifications of coloring materials are synthetic colorants, which need a certification given by a "safety for humans" association, and natural pigments, exempt from certification, according to the restrictions of each country [10].

This review summarizes different works performed in recent years on the microencapsulation of natural pigments by spray drying. It shows the procedures currently used for the extraction and stabilization of pigments, as well as the use of different encapsulating agents and the applications of pigments in food systems.

Natural Pigments

The most common natural pigments are chlorophylls, carotenoids, flavonoids, and betalains [11]. In general, pigments like anthocyanins and betalains are soluble in water. In contrast, carotenoids and chlorophylls are fat soluble. Depending on the characteristics of the pigment, when it is used as a concentrated extract from fruits or vegetables for coloring other foods, they are dispersible in water; however, the appearance in the matrix when used in foods could have a cloudy appearance. Basic structure of common natural pigments is show in Fig. 1. Natural pigments, in addition to coloring, can have other properties due to their nutraceutical characteristics, important in the prevention and treatment of some ailments [12–17]. A disadvantage of using natural pigments is their great instability under different

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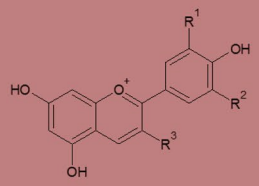
Anthocyanin basic structure

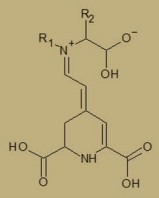


Anthocyanin	R1	R2	R3
Pelargonidin	H	H	H
Cyanidin	H	OH	H
Delphinidin	OH	OH	H
Peonidin	H	OCH_3	H
Petunidin	OH	OCH_3	H
Malvidin	OCH_3	OCH_3	H
Pelargonidin -glucoside	Glc	H	H
Cyanidin -glucoside	Glc	OH	H
Delphinidin -glucoside	Glc	OH	H
Peonidin-glucoside	Glc	OCH_3	H

Instability factors

↑↑↑ instability to light
↑↑ - ↑↑↑ instability to heat
↑↑↑ instability to oxygen
↑ instability to pH
Less stable in anthocyanidin form
Enzyme sensitive
Anthocyanin source
Acylation

Betaxanthin basic structure



R1 = H
R2 = amino acid, amine or derivatives

Indicaxantina (*Opuntia ficus-indica*)
Portulacaxantina-I (*Portulaca grandiflora*)
Vulgaxantina-I (*Beta vulgaris*)
Vulgaxantina-II (*Beta vulgaris*),
Miraxantina-II (*Mirabilis jalapa*),
Humuluxantina (*Rivinia humilis*)

Instability factors

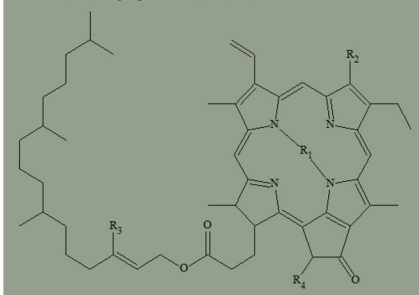
↑↑↑ instability to light
↑ - ↑↑ instability to heat
↑↑↑ instability to oxygen
↑↑ instability to pH low than 3 and above 7
Sensitive to degrading enzymes
Low degree of glycosylation and acylation
Metal cation

Betacyanin basic structure

R1 = glucosyl or derivatives
R2 = glucosyl, glucoronyl, derivatives or H

Betanina (*Beta vulgaris*)
Amarantina (*Amaranthus tricolor*)
Hilocerenina (*Hylocereus polyrhizus*)
Filocactina (*Phyllocactus Hybridus*)
Celosianina-I (*Celosia cristata*)
Iresina-I (*Iresine herbstii*)
Gomferina-I (*Gompherena globoso*)
Rivinianina (*Rivinia humilis*)
Bougainvillein r-I (*Bougainvillea*)

Chlorophyll basic structure



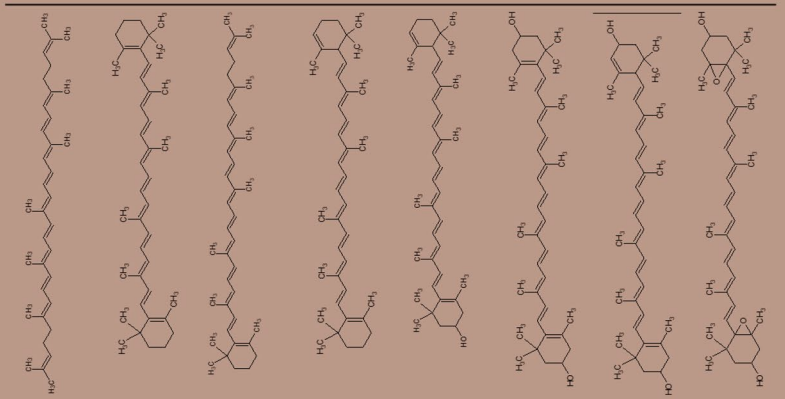
Chlorophyll	R1	R2	R3	R4
Chlorophyll a	Mg	CH_3	phytyl	$COOCH_3$
Chlorophyll b	Mg	CHO	phytyl	$COOCH_3$
Chlorophyllide a	Mg	CH_3	H	$COOCH_3$
Pheophytin a	H	CH_3	phytyl	$COOCH_3$
Pheophytin b	H	CHO	H	$COOCH_3$
Pheophorbide a	H	CH_3	H	$COOCH_3$
Pyropheophytin a	H	CH_3	phytyl	H

Instability factors

↑↑↑ instability to light
↑↑ instability to heat
↑↑↑ instability to oxygen
↑ instability to pH
Enzyme sensitive
Fermentation increases phytol breakdown
Color change due to Mg loss

Carotenoids basic structures

Lycopene β-carotene γ-carotene α-carotene β-cryptoxanthin Zeaxanthin Lutein Violaxanthin



Instability factors

↑ stability to light
↑↑ stability to high heat
↑ stability to oxygen
↑↑↑ stability to pH
Enzyme sensitive
Carotenoid nature (carotene or xanthophyll)
E- or *Z*- configuration
Grade of esterification
Causing color loss due to double bond oxidation

◂**Fig. 1** Characteristics and instability factors of some pigments (anthocyanins, betalains, chlorophylls, and carotenoids). One upward arrow to three upward arrows indicate low to high instability

environmental conditions. The use of methods that allow the preservation of natural pigments has been widely studied; one of the most used is spray drying [18–20].

Spray Drying

Briefly, spray drying (Fig. 2) consists of the preparation of an emulsion using a stabilizing agent, which in turn will act as the encapsulating. Its incorporation into the extract, rich in pigments, must be perfectly homogenized for its correct passage throughout the atomizer of the drying equipment. Therefore, small droplets should be formed to facilitate the evaporation of water. Finally, the powder is recovered in the collector of the system (Fig. 2) [18–22]. The spray drying technique allows the obtaining of powders of which characteristics may help in the incorporation into different food products, which will depend on factors such as the type of solution, the drying conditions, and the encapsulating material, among others [23–25].

During the passage through the dryer occurs the atomization; it corresponds to the feeding of the active compound-encapsulating agent mixture, usually carried out using a peristaltic pump. The liquid is passed into a spray nozzle. The most common atomizing nozzle is the two-fluid or pneumatic nozzle, but it can find different types such as centrifugal or rotary atomizer, hydraulic or pressure nozzle, and ultrasonic nozzle. The selection of the type of nozzle will depend on the characteristics of the fluid and the powder to be obtained. The two-fluid nozzle is used for a liquid feed and a compressed gas feed, usually air. As the liquid collides with air at high speed, the liquid disintegrates into tiny droplets. One of the disadvantages of this type of nozzle is the low uniformity of the size of the drops. In drop drying, atomized particles come into contact with hot air (concurrent, countercurrent, and mixed flow), where drying begins. During drying, two phenomena occur, the transfer of mass and heat, usually by convection of the hot air to the atomized drop. The drops adopt a spherical shape quickly reaching the equilibrium determined by the evaporation rate. The evaporation of the solvent (usually water) occurs by the migration of moisture from the inside of the drop at constant temperature and constant partial vapor pressure at the surface of the particle. Once critical moisture values have been reached, a shell formation over the surface occurs; therefore, evaporation now depends on the rate of diffusion of moisture through the shell. Drying ends when the temperature of the particle and the air are equal.

Methods of Extraction for Natural Pigments

The first step in the natural pigment encapsulation process is the extraction. Different pigment extraction methods have been reported; the use of each of them will impact on their quantity and stability (Table 1). They can be classified into physical, chemical, and biological methods that in turn are divided into conventional and unconventional methods (or green technologies) [27, 28].

Extraction by Physical Methods

The simplest method to obtain extracts rich in pigments consists of applying a force: pressure ("screw"), centrifugation ("fast spinning metal blade or shredder blade"), or shearing ("blender, high speed homogenization, high speed shearing"), causing cellular disruption of the material and releasing compounds (Fig. 3). This method has been used mainly to obtain betalains (Table 1); however, it has also been used to obtain anthocyanins, carotenoids, and chlorophylls. In addition to the simplicity of the method, it has the advantage of being carried out in short times, avoiding the degradation or isomerization of the pigments [74]. However, the aforementioned forces (pressure, centrifugation or shear) can increase the temperature, affecting negatively the extract [37]; this problem could be solved using a "cold pressing extraction" [39]. Examples of pressing extractions for obtaining different pigments are (a) anthocianins of pomegranate arils (*Punica granatum*), bluberries (*Vaccinium corymbosum*) [43], sour cherry (*Prunus cerasus*) [46], juçara (*Euterpe edulis*) [47] and tamarillo (*Solanum betaceum*) [48]; (b) betalains from beetroot (*Beta vulgaris* subsp. *vulgaris*) [49–56], cacti such as pitaya (*Stenocereus griseus*, *Stenocereus queretaroensis*) [56, 57], prickly pear (*Opuntia ficus-indica*) [58, 59], garambullo (*Myrtillocactus geometrizans*) [60] and jiotilla (*Escontria chiotilla*) [61]; (c) carotenoids from pears (*Pyrus pyrifolia*) [61], papaya (*Carica papaya*) [62], golden kiwifruit (*Actinidia chinensis*) [63], mango (*Mangifera indica*) [66], jackfruit (*Artocarpus heterophyllus*) [70], and tomato (*Solanum lycopersicum*) [75]; and (d) chlorophylls of moringa leaves (*Moringa oleifera*) [75] and green kiwifruit (*Actinidia deliciosa*) [76].

Extraction by Chemical Methods

Conventional methods for obtaining pigments are Soxhlet extraction, maceration or hydrodistillation [78]. The methods are easy to perform, cheap, and relatively quick. The maceration procedure is the most used for the extraction of water-soluble pigments such as betalains and anthocyanins

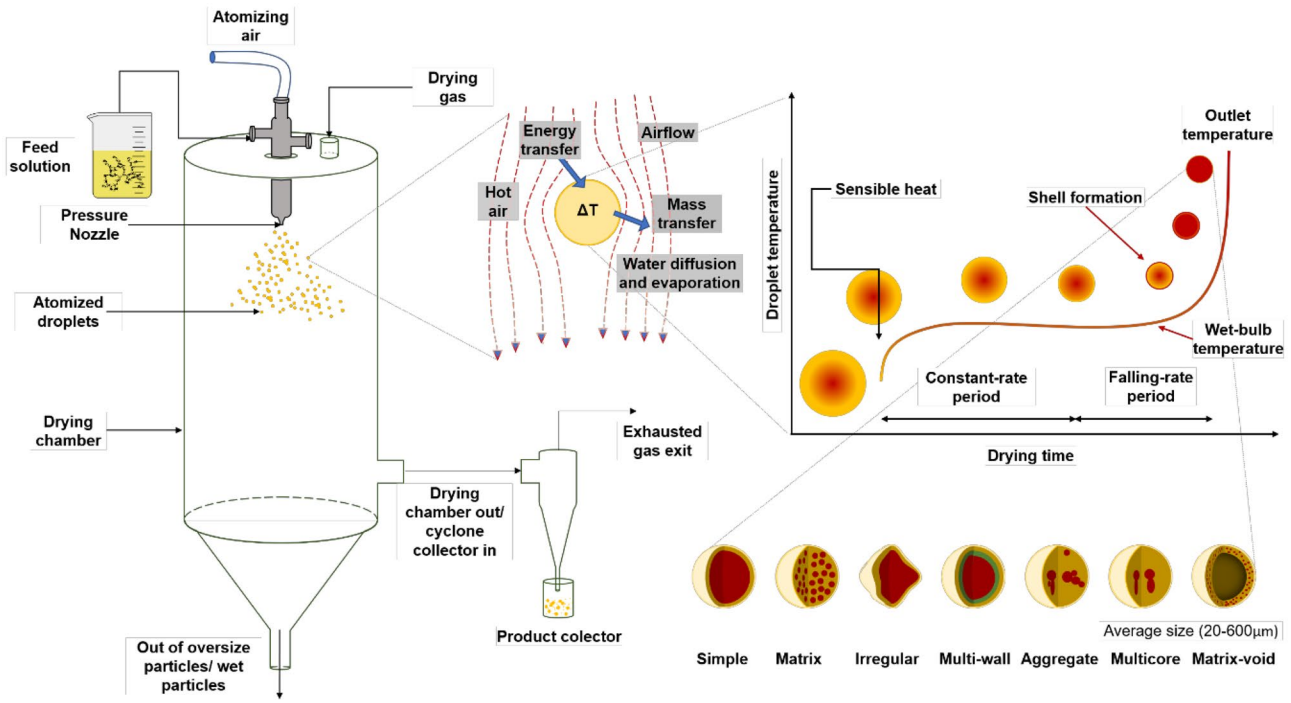


Fig. 2 Summary of spray drying process (adapted from [18, 26 and 20])

or fat-soluble pigments such as carotenoids and chlorophylls (Table 1). Briefly, the method consists of macerating the plant material in a solvent, with or without agitation (agitation increases diffusion and removes the saturated solvent from the surface of the sample), at room temperature or elevated temperature (high temperature can speed up the extraction, reducing the extraction time, but there is a risk of destruction of the pigment) [79, 27, 28]. The method is overseen by the premise of the formation of bonds between the solvent and the compound of interest, which will be extracted from the organic material. The solvent will increase the solubility of the solute; however, sometimes, it is necessary to repeat the macerating process to increase the yield (Fig. 4).

The selection of the solvent for extraction is very important as it will influence the stability of the pigments and the toxicity of the final product. Based on the bibliography reviewed in this work, the most widely used solvents are water, ethanol [81], acetone, and phosphate buffer (for the extraction of betalains) [49]. The plant material is subjected to grinding for reducing the size of particles (from fresh, heat dried, or lyophilized material) in order to increase the surface area for extraction, increasing the permeability of the material and helping to extract the solvent mixture-material [28].

Some advantages of this extraction method are simplicity, little investment, and the possibility of doing it at low temperatures. However, it has some disadvantages: long extraction time (in some cases), large amount of solvent, requires filtering the extract, multiple extractions, and techniques to remove the solvent such as evaporation under atmospheric or reduced pressure. Some researchers have reported the obtaining of anthocyanins through maceration with ethanol: calyces Roselle (*Hibiscus sabdariffa*) [31], garden ginger pericarp (*Renealmia alpinia*) [32], Cabernet Sauvignon (*Vitis vinifera*), and *Vitis labrusca* grape bagasse [36]. Ethanol has been also used for obtaining betalains from pitaya pulp (*Stenocereus pruinosus*) [52] and chlorophylls from spinach (*Spinacia oleracea*) [71]. It is common to use acidified solvents for anthocyanins extraction to aid the pigment stability. Acidified ethanol has been used to obtain saffron petals (*Crocus sativus* L.) extracts [29] or barberry (*Berberis vulgaris*) extracts [30]. Also, aqueous solutions of citric acid have been used for extracting anthocyanin from purple cabbage (*Brassica oleracea* L. var. Capitata L. f. Rubra) [34]. Phosphate buffer, with pH adjusted, has been used for extracting betalains of prickly pear (*Opuntia megacantha*) [49]. Hexane, a non-polar solvent, has been used commonly for making extractions of carotenoids from annatto seeds (*Bixa orellana* L.) [64].

Non-conventional Methods

Among the non-conventional methods used for extracting compounds is the supercritical fluid technique. This method avoids the use of solvents; the extracts rich in pigments are

Table 1 Encapsulation of pigments (source, pigment extraction, homogenization, and drying condition) with different wall materials and color parameters

Material	Extraction method	Homogenization method	Drying conditions	Encapsulation agents	*L**	*a**	*b**	Reference
Anthocyanins								
Dried saffron petals (*Crocus sativus* L.)	Maceration (acidified ethanol, 50% v/v, pH = 2)/stirring (24 h, three times)/concentration (RotEva 30 min/40 °C, 10°Bx)	Predissolved EA (1:10, w/v) in water/mixed extract-EA (1:5, w/w, ratio) at pH = 2/homogenizer (10 min)	130 °C/80 °C	Barley β-glucan	40.33	27.32	35.21	Ahmad et al. [29]
				β cyclodextrin	61.95	13.45	81.47	
Barberry (*Berberis vulgaris*)	Puree maceration (acidified ethanol 75% v/v)/2 h at 50 °C/2 h at room temperature/filtration/concentration (RotEva 40 °C, 15°Bx)	Predissolved EA (70 °C/1 h stirring, overnight rehydration/cold temperature, gelatin in hot water)/mixed extract-EA (20% solids)	150 °C/100 °C	MDX	66.18	41.74	2.75	Akhavan Mahdavi et al. [30]
				MDX-GA (3:1)	73.27	34.74	−0.14	
				MDX-gelatin (3:1)	69.81	30.83	0.62	
Roselle calyces (*Hibiscus sabdariffa*)	Calyces (600 g) maceration (600 L ethanol 50%)/stirring (2 h)/centrifugation (4500 rpm)/filtration/concentration (RotEva 35 °C)	Direct addition of EA		Mesquite gum 1% (w/v)	23.96	36.02	14.84	Ochoa-Velasco et al. [31]
				Mesquite gum 2% (w/v)	26.6	38.5	16.41	
				Mesquite gum 3% (w/v)	30.54	42.84	18.66	
				Mesquite gum 4% (w/v)	29.19	40.37	17.68	
				Mesquite gum 5% (w/v)	24.05	32.83	14.56	
Xkijit (*Renealmia alpinia*)	Peel maceration (1:5 w/v, ethanol 95%/90 min/multiple times)/filtration/concentration (RotEva 55 °C)	Direct addition of EA (20°Bx)/magnetic stirring homogenization	150/98 °C	MDX	22.97	21.85	−7.06	Jimenez-Gonzalez et al. [32]
				MDX-GA	28.3	23.12	−6.58	
				GA	25.3	18.24	−7.48	
Pomegranate (*Punica granatum* L.)	Mechanical extraction stored (4 °C/overnight)/clarification (spiral ultrafiltration, 40 kD)/adjusted (12°Bx)	Direct addition of EA	124/48 °C; rotatory atomizer 11,300rpm	MDX 25%	46.83	30.02	−2.7	Jafari et al. [33]
				MDX 35%	47.5	31.53	−3.44	
				MDX 45%	46.88	27.36	−3.87	
Red cabbage (*Brassica oleracea* L. var *capitata* L. *f. rubra*)	Blanched cabbage maceration (1:2 w/v, citric acid solution (2%)/16 h/darkness)/filtration	Direct addition of EA/ultraturrax homogenization (4000×*g*/5 min)	140 °C	GA 10%	72.37	36.54	−4.26	Bernstein and Noreña [34]
			140 °C	GA 15%	75.79	30.13	−4.07	
			160 °C	GA 10%	71.85	34.96	−2.43	
			160 °C	GA 15%	74.06	31.47	−4.6	
			140 °C	Polydextrose 10%	71.46	39.33	−0.52	
			140 °C	Polydextrose 15%	73.32	35.64	−0.68	
			160 °C	Polydextrose 10%	64.58	43.19	3.85	
			160 °C	Polydextrose 15%	71.78	37.48	0.08	

Table 1 (continued)

Material	Extraction method	Homogenization method	Drying conditions	Encapsulation agents	L^*	a^*	b^*	Reference
Blueberry pomace (*Vaccinium* cv *Elliot*)	Enzymatic extraction (Pectimex Ultra SP-L and Cellubrix, 1:1, 0.1 mL/100 g pomace, incubation (1 h), 20–50 °C, shaking 200 rpm, darkness) Enzymatic treated and untreated maceration (1:3 w/v, citric acid solution, pH = 2/50 °C/1 h)/stirring (200 rpm)/filtration	Predissolved EA (3%, 60 °C)/mixed extract (2%)/shear homogenizer (3000 rpm/5 min)/filtered	150/80 °C	Inulin	27.3	1.6	0.1	Waterhouse et al. [35]
				Enzymatic extract-inulin	28.9	1.4	0.4	
				Alginate	28	1.5	−2.1	
				Enzymatic extract- alginate	29.8	1.2	−1.7	
Grape Bordeaux (*Vitis labrusca*) Grape Cabernet Sauvignon (*Vitis vinifera*)	Pomace maceration (1:20 w/v, ethanol 40%, shaking 125 rpm/180 min/25 °C)/centrifugation (6000 rpm/10 min)/concentration (RotEva 40 °C)	Magnetic stirring homogenization/room temperature	130/80 °C	Yeast biomass (*Saccharomyces cerevisiae*) 5%	35.49 49.44	17.57 7.71	−3.17 12.44	Rubio et al. [36]
Blueberry (*Vaccinium corymbosum* L.)	Mechanically crushed/centrifugation (21,000×*g*/5 min)/filtration	Direct addition of EA extract ultraturrax homogenization (21,000×*g*/5 min)	125 °C/ultrasonic nozzle 50–60 kHz	MDX 1%	37.1	30.8	−3.6	Tatar Turan et al. [37]
					56.2	36.4	−0.8	
				MDX-GA 4:1 w/w, 1%	44.2	32.2	−6.9	
					56	38.9	−3.4	
Elderberry (*Sambucus nigra*)	Grinded sieved lyophilized elderberry/acetone maceration (3:10 w/v, 80%)/sonication assisted extraction (ultrasonic bath 23 °C/15 min)/filtration/petroleum ether addition (shake 5 min)/aqueous phase concentration (RotEva, 15°Bx)	Direct addition of EA extract heating (60 °C)/magnetic stirring (hydration)/ultraturrax homogenization (8000 rpm/3 min)	140 °C	β-glucan 0.5%	77.38	10.07	2.48	Sobieralska and Kurek [38]
				β-glucan 1%	76.49	9.84	2.56	
				β-glucan 2%	77.88	11.25	2.62	
				β-glucan 3%	79.49	9.97	2.26	
				MDX-GA (92.5:7.5)	79.65	9.63	2.35	
Sour cherry juice (*Prunus cerasus* L.)	Mechanical pressing/filtration/clarification/concentration (RotEva 35 °C, 61°Bx)	Predissolved EA (magnetic stirred)/extract mixed (30% final total solids)	170/83 ± 2 °C; rotatory atomizer 18,000 rpm	MDX	37.39	33.53	12.98	Sarabandi et al. [39]
				MDX-GA (4:1)	34.65	31.04	14.27	
				MDX-GA (1:1)	34.83	28.09	16.33	
				MDX-GA (1:4)	33.88	26.49	19.05	
				GA	35.31	24.05	20.57	

Table 1 (continued)

Material	Extraction method	Homogenization method	Drying conditions	Encapsulation agents	*L**	*a**	*b**	Reference
				MDX-WPC (9:1)	40.7	26.94	12.46	
				MDX-WPC (4:1)	43.32	25.53	13.61	
				MDX-WPC (7:3)	37.86	21.1	13.04	
				MDX-WPC (3:2)	40.19	19.44	15.57	
				GA-WPC (9:1)	40.67	18.87	20.79	
				GA-WPC (4:1)	34.98	14.72	17.46	
				GA-WPC (7:3)	39.47	15.21	18.97	
				GA-WPC (3:2)	35.53	15.06	17.81	
Blue maize grains (*Zea mays* L.)	Maize flour maceration (ethanol 80%/30 min/stirring)/sonication-assisted extraction (30 min)/entrifugation (6700×*g*/15 min/4 °C)/concentration (RotEva 40 °C)	Direct addition of EA (120 g/L)/magnetic stirring homogenization	150/80 °C	MDX	45.57	*C* = 4.4	*H* = 340.3	Ruiz Canizales et al. [40]
				MDX-pectin (84:16)	40.19	*C* = 11	*H* = 357.5	
Hibiscus sabdariffa flowers	Maceration (1:1 w/v, citric acid solution (2%), under turbolization (60 Hz/5 min); sonication assisted extraction (ultrasonic bath (37 kHz)/20 min)/filtration	Direct addition of EA/ultraturrax homogenization (18,000 rpm/25 °C)	180 °C/nozzle size (0.7, 1.0, and 1.2 mm)	GA 10%	45.2	32.8	4.9	Martins et al. [41]
					45.1	34	5.2	
					46.2	36.3	6.2	
				Chitosan 10%	43	19.9	−7.7	
					41.7	27.7	−2	
					43.4	5.5	−0.5	
				MDX 10%	46.1	32.8	4.9	
					45	36.1	5.2	
					50.4	27.8	2.9	

Table 1 (continued)

Material	Extraction method	Homogenization method	Drying conditions	Encapsulation agents	*L**	*a**	*b**	Reference
				GA-chitosan (1:1, 10%)	38.4	18.7	18.7	
					40.4	21.9	−5.4	
					38.4	18.7	−5.4	
				GA-MDX (1:1, 10%)	46.7	32.7	4.6	
					42.5	33.6	5.2	
					48.3	35.6	4.4	
				Chitosan-MDX (1:1, 10%)	42.8	25.2	−3	
					39.9	26.7	−2.9	
					41.4	25.2	−5	
				GA-chitosan-MDX (0.3:0.3:0.3, 10%)	40.2	23.1	−3	
					40.6	29.3	−0.9	
					41.7	27.7	−2	
Hibiscus sabdariffa L.	Maceration (1.5:10 w/v, 100 °C water/10 min)/filtration	Magnetic stirring homogenization	180/80 °C	MDX-GA 95:5	37.79	29.09	2.58	Archaina et al. [42]
				MDX-GA 85:15	36.32	30.87	2.61	
				MDX-GA 70:30	36.87	29.28	2.68	
Jucara pulp (*Euterpe edulis* Martius)	Homogenization (3 min/in a blender)/filtration		160/86 °C	None	14.76	8	−4.04	Pereira et al. [43]
Rose residues (*Rosa rugosa*)	Oven-dried ground rose residue maceration (1:20 w/v, acidified ethanol 55%, pH = 3)/sonication assisted extraction (ultrasound/60 min/55 °C)/filtration/concentration (RotEva 45 °C)	Stirring homogenization (35 °C/30 min)	170/80 °C	MDX-GA (1:1, 10%)	77.57	11.7	5.86	Yu and Lv [44]
Black chokeberry (*Aronia melanocarpa*)	Ground lyophilized fruit maceration (3:1 w/v, acetone 80%)/sonication assisted extraction (ultrasonication 15 min)/filtration/petroleum ether addition (shake 5 min)/concentration (RotEva until no acetone smell detected)	Predissolved MDX and dietary fibers (74:6 ratio)/heating (60 °C)/stirring/extract mixed/ultra-turrax homogenization (8000 rpm/3 min)	140 °C	MDX-GA	74.01	11.2	4.92	Pieczykolan and Kurek [45]
				MDX-inulin	73.75	10.91	4.99	
				MDX-β-glucan	71.64	12.03	5.39	
				MDX-pectin	71.05	11.75	5.23	
				MDX-guar gum	74.4	9.04	4.43	

Table 1 (continued)

Material	Extraction method	Homogenization method	Drying conditions	Encapsulation agents	*L**	*a**	*b**	Reference
Tamarillo (*Solanum betaceum*) fruit	Mechanical extraction/dark cold storing/concentration adjust (12°Bx)	Direct addition of EA (20% w/w)/homogenization (9500 rpm/10 min); filtration	150/70 °C; rotary atomizer 15,000 rpm	MDX 10DE	67.45	17.43	5.01	Ramakrishnan et al. [46]
				GA	64.66	20.23	6.23	
				n-octenyl succinic Anhydride modified Starch	64.53	20.23	6.42	
				n-octenyl succinic Anhydride modified Starch	66.32	18.85	5.32	
				Resistant MDX	67.48	17.43	5.16	
Betalains								
Red beetroot (*Beta vulgaris* L.)	Mechanical extraction	Direct addition of EA (30% w/w)	150/90 °C	MDX	13.76	18.6	7.65	Antigo et al. [47]
				MDX-xanthan gum (99.5:0.5)	13.15	19.24	9.35	
Red beetroot (Beta *vulgaris* L.) juice	Mechanical extraction/concentration (RotEva 55 °C/200 mbar, 22°Bx)	Direct addition of EA (20% total solids; single and binary mixture)/blender homogenization	170 °C	MDX 10DE	23.54	26.17	3.12	Bazaria and Kumar [48]
				MDX 20DE	22.87	36.93	1.98	
				GA	21.34	35.34	2.26	
				MDX10DE-MDX20DE	22.07	34.67	2.06	
				MDX10DE-GA	24.89	39.03	2.34	
				MDX20DE-GA	21.93	31.76	2.65	
Cactus fruit (*Opuntia megacantha*) orange pulp	Mechanically crushed fruit/filtration (seed removement)/lyophilization (1.9 and 2.3% wb moisture content)/maceration (1:10 w/w, phosphate buffer pH = 5.5)	Direct addition of EA/magnetic stirring homogenization	170/77 °C	Extract-MDX-cladode mucilage, 1:1:0.225 w/w/w	50.42	1.18	20.69	Otálora et al. [49]
Cactus fruit (*Opuntia ficus-indica*) purple pulp	Mechanically crushed fruit/filtration (seed removement)/lyophilization (1.9 and 2.3% wb moisture content)/maceration (1:5 w/w, phosphate buffer pH = 5)	Direct addition of EA/magnetic stirring homogenization	170/98 °C	Extract-MDX-cladode mucilage (1:1:0.225)	66.46	29.99	−4.84	Otálora et al. [50]
				MDX	66.04	30.14	−5.28	

Table 1 (continued)

Material	Extraction method	Homogenization method	Drying conditions	Encapsulation agents	*L**	*a**	*b**	Reference
Pitaya (*Stenocereus griseus*) red pulp	Manually crushed pulp/filtration (seed removement)/juice (10.2°Bx; total solids 87.55 g/L)	Direct addition of EA (MDX and pectin material mixed at 40:20 and 60:30 ratios, added to the pitaya juice 60% and 90% total solids, respectively), homogenization (5 min)/magnetic stirring homogenization (400 rpm/1 h)	150 °C; 1.5 L/h	MDX-pectin 40:20	39.52	44.19	11.31	García-Lucas et al. [51]
			180 °C; 1.5 L/h	MDX-pectin 40:20	42.35	41.7	20.61	
			150 °C; 2 L/h	MDX-pectin 40:20	39.24	44.56	10.24	
			180 °C; 2 L/h	MDX-pectin 40:20	40.79	43.5	15.24	
			150 °C; 1.5 L/h	MDX-pectin 60:30	41.05	42.68	11.49	
			180 °C; 1.5 L/h	MDX-pectin 60:30	41.35	42.17	14.94	
			150 °C; 2 L/h	MDX-pectin 60:30	39.38	43.26	11.28	
			180 °C; 2 L/h	MDX-pectin 60:30	39.7	43.05	12.1	
Pitaya (*Stenocereus prunosus*)	Pulp maceration (1:2, ethanol 50%/15 h)/filtration/concentration (RotEva)/sugar and mucilage removal	Direct addition of EA/storage (4 °C)/filtration	140/80 °C	Phosphorylated potato native starch 10%	63.59	$C=28.71$	$H=8.08$	Vargas-Campos et al. [52]
				Succinylated potato native starch 10%	60.68	$C=26.25$	$H=9.3$	
				N-Lok 10%	67	$C=23.21$	$H=4.66$	
Stenocereus queretaroensis pulp	Manually mature pulp obtention	Mixed (pulp/cladode)/agitation (200 rpm/30 min)/filtration	140/70 °C	Cladode (90:6.1)	56.28	31.37	27.13	Delia et al. [53]
				Cladode (45:6.1)	58.58	25.16	27.89	
	Manually unmatured pulp obtention			Cladode (90:6.1)	67.63	12.75	50.59	
				Cladode (45:6.1)	66.24	10.73	47.38	
Stenocereus queretaroensis skin	Mature fruit peel	Mixed (skin/acidified cladode, 1% acetic acid)/agitation (200 rpm/30 min)/filtration		Cladode (90:6.1)	52.58	29.28	21.94	
				Cladode (45:6.1)	57.89	18.68	22.95	
	Unmatured fruit peel			Cladode (90:6.1)	56.43	23.43	48.47	
				Cladode (45:6.1)	61.25	7.6	51.39	
Escontria chiotilla pulp	Manually mature pulp obtention	Mixed (pulp/cladode)/agitation (200 rpm/30 min)/filtration		Cladode (90:6.1)	61.42	8.06	29.26	
				Cladode (45:6.1)	56.7	6.59	24.61	
Escontria chiotilla skin	Mature fruit peel	Mixed (skin/acidified cladode, 1% acetic acid)/agitation (200 rpm/30 min)/filtration		Cladode (90:6.1)	58.45	12.35	31.78	
				Cladode (45:6.1)	54.33	17.91	35.66	

Table 1 (continued)

Material	Extraction method	Homogenization method	Drying conditions	Encapsulation agents	*L**	*a**	*b**	Reference
Beetroot (*Beta vulgaris* L.)	Mechanically extracted juice/filtration	Direct addition of EA (10%)	150/90 °C	MDX	34.45	37.08	10.35	Antigo et al. [54]
				MDX-freeze dried chia (99.5:0.5)	32.8	35.57	11.11	
				MDX-oven dried chia (99.5:0.5)	36.64	32.23	9.91	
				MDX-GA (1:1)	34.99	35.24	8.91	
		Direct addition of EA (15%)		MDX	39.79	33.53	9.07	
				MDX-freeze dried chia (99.5:0.5)	36.88	33.41	9.41	
				MDX-oven dried chia (99.5:0.5)	35.47	32.71	10.72	
				MDX-GA (1:1)	34.41	36.31	10.03	
Bougainvillea glabra bracts	Ground dried bracts maceration (water 1:20 w/v)/magnetic stirring (5 min)/cooling (0 °C)/microwave-assisted extraction (2450 MHz, 600 W, 13 min)/filtration	Direct addition of EA/ultraturrax homogenization (6500 rpm/5 min)	160 °C	Polydextrose 15%	40.76	26.15	−12.72	Kuhn et al. [55]
				Inulin 15%	42.17	21.99	−11.36	
				Polydextrose-inulin 7.5–7.50%	41.34	23.93	−12.12	
				Inulin-egg albumin (pH 5) 7.5–7.5%	37.77	18.73	−9.33	
Beetroot (*Beta vulgaris* L.)	Boiled (98 °C) fruit/cutting (small pieces)/mechanically extracted juice/filtration/centrifugation (13,000 rpm)	Direct addition of EA/magnetic stirring homogenization (1000 rpm/45 min)	120/60 °C	Tetraethyl orthosilicate	75.18	39.1	2.45	Hernández-Martínez et al. [56]
Garambullo (*Myrtillocactus geometrizans*)	Mechanically grinded (water 20% v/w)/filtration/centrifugation (13,000 rpm)				74.8	37.12	1.12	
Beetroot (*Beta vulgaris* L.)	Mechanically extracted juice/filtration	Direct addition of EA/ultraturrax homogenization (5000 rpm/5 min)	130/85 °C	Inulin-WPI 1:2	52.8	$C=30.5$	$H=6.6$	Carmo et al. [57]
				Inulin-WPI 1:1	47.6	$C=31.9$	$H=6.5$	
				Inulin-WPI 2:1	52.1	$C=29.9$	$H=5.1$	
			150/95 °C	Inulin-WPI 1:2	54.3	$C=29.6$	$H=9.2$	
				Inulin-WPI 1:1	51.9	$C=30.5$	$H=8.4$	
				Inulin-WPI 2:1	51	$C=29.8$	$H=10.7$	
			170/108 °C	Inulin-WPI 1:2	52.2	$C=31.4$	$H=5.7$	
				Inulin-WPI 1:1	52.6	$C=31.3$	$H=6.1$	
				Inulin-WPI 2:1	51.8	$C=31.1$	$H=9.4$	

Table 1 (continued)

Material	Extraction method	Homogenization method	Drying conditions	Encapsulation agents	L^*	a^*	b^*	Reference
Beetroot (*Beta vulgaris* L.)	Mechanically extracted juice/filtration		130/70 °C	Pumpkin protein isolate	72.4	1.47	23.91	Čakarević et al. [58]
Carotenoids								
Asian pears (*Pyrus pyrifolia Nakai* cv. *Niitaka*)	Mechanically extracted juice (1325 kgf/cm^2/3 min)/filtration	Direct addition of EA/blender homogenization	130/82 °C, rotatory disc atomizer	MDX 15%	93.3	−3.16	13.39	Lee et al. [59]
				MDX 20%	94.17	−3.43	11.01	
				MDX 25%	94.48	−3.94	10.29	
			150/85 °C, rotatory disc atomizer	MDX 15%	92.54	−3.17	14.46	
				MDX 20%	93.68	−3.5	13.02	
				MDX 25%	94.19	−3.48	10.29	
			170/88 °C, rotatory disc atomizer	MDX 15%	88.18	−1.33	20.86	
				MDX 20%	91.51	−1.89	14.14	
				MDX 25%	92.27	−2.59	13.64	
Papaya (*Carica papaya* cv *Sunrise solo*)	Manually pulp obtention	Direct addition of EA	150 °C	MDX 14%	14.07	3.99	14.49	Gomes et al. [60]
Golden kiwi (*Actinidia* sp.)	Mechanically pureed peeled fruit/centrifugation (5000×*g*/25 min/4 °C)/filtration	Direct addition of EA shear homogenization (500 rpm/1 min/3 bursts)	150 °C	Skim milk-MDX	839	−1.05	25.7	Sun-Waterhouse and Waterhouse [61]
				Skim milk-MDX (warmed feed)	84.1	−1.03	26.5	
		Direct addition of EA to extract and residue (skim milk, 40:60 ratio, MDX 3%)/shear homogenization (1000 rpm/1 min/3 bursts)		Skim milk-MDX	74.8	−1.18	33.9	
Mango (*Mangifera indica)*	Mechanically puree obtention/filtration	Direct addition of EA	150 °C	None	80.33	5.99	50.34	Zotarelli et al. [62]
				MDX	87.71	1.51	32.24	
Jackfruit (*Artocarpus heterophyllus*)	Blended pulp (22,000 rpm/30 s)/enzymatic liquefaction (Pectinex®, Celluclast®, 50 °C)/enzyme deactivation (90 °C/5 min)	Direct addition of EA	140 °C	MDX 20% at 1:1 ratio	87.33	0.87	37.95	Wong and Tan [63]
			150 °C	MDX 20% at 1:1 ratio	85.98	0.86	41.26	
			160 °C	MDX 20% at 1:1 ratio	86.49	0.9	38.89	
			170 °C	MDX 20% at 1:1 ratio	85.59	0.83	40.81	
			180 °C	MDX 20% at 1:1 ratio	86.02	0.87	40.56	
			160 °C	MDX 10%	85.11	0.87	37.95	
			160 °C	MDX 15%	85.98	0.88	41.26	
			160 °C	MDX 20%	86.49	0.82	38.88	
			160 °C	MDX 25%	85.59	0.83	40.81	
			160 °C	MDX 30%	86.02	0.87	40.56	

Table 1 (continued)

Material	Extraction method	Homogenization method	Drying conditions	Encapsulation agents	*L**	*a**	*b**	Reference
Annatto (*Bixa orellana*) seeds	Hexane extraction/methanol extraction (twice)/ethyl acetate extraction (15 min/magnetic stirring)/concentration (RotEva)/recovering (dichloromethane/ethanol)/crystal formation (−18 °C/12 h)	Dissolved crystals (ethanol)/combination with EA (20°Bx)/magnetic stirring homogenization (50 °C/30 min)	150 °C	MDX:GA (100:0)	48.9	7.88	17.55	Tupuna et al. [64]
				MDX:GA (85:15)	49.45	5.58	17.05	
				MDX:GA (65:35)	47.7	7	18.9	
				MDX:GA (50:50)	45.77	9.74	19.11	
				MDX:GA (35:65)	46.24	6.3	17.84	
				MDX:GA (15:85)	48	4.12	16	
				MDX:GA (0:100)	46.83	6.43	14.9	
Mandarin (*Citrus unshiu*) beverage		Direct addition of EA	120 °C; rotary atomizer 9860 rpm	MDX 20DE-corn syrup (30%, 1:1)	84.64	5.16	35.77	Lee et al. [65]
			135 °C; rotary atomizer 9860 rpm		82.45	5.27	34.93	
			150 °C; rotary atomizer 9860 rpm		81.51	5.61	34.1	
			120 °C; rotary atomizer 9860 rpm	MDX 20DE-corn syrup (35%, 1:1)	85.33	5.15	35.1	
			135 °C; rotary atomizer 9860 rpm		84.01	5.25	35.81	
			150 °C; rotary atomizer 9860 rpm		82.63	5.57	33.98	
			120 °C; rotary atomizer 9860 rpm	MDX 20DE-corn syrup (40%, 1:1)	86.17	5.11	34.78	
			135 °C; rotary atomizer 9860 rpm		84.59	5.24	34.68	
			150 °C; rotary atomizer 9860 rpm		83.61	5.56	33.77	
Tomato (*Solanum lycopersicum*)	Mechanically extracted juice (pressure-type juicer)/filtration		140 °C	MDX 30:70	53.33	*a**/*b** = 0.84		Sidhu et al. [66]
				MDX 40:60	54.0	*a**/*b** = 1.15		
				MDX 50:50	52.0	*a**/*b** = 1.53		
			150 °C	MDX 30:70	53.78	*a**/*b** = 0.79		
				MDX 40:60	54.33	*a**/*b** = 1.05		
				MDX 50:50	52.23	*a**/*b** = 1.40		
			160 °C	MDX 30:70	54.09	*a**/*b** = 0.76		
				MDX 40:60	57	*a**/*b** = 1.00		
				MDX 50:50	53.32	*a**/*b** = 1.13		

Table 1 (continued)

Material	Extraction method	Homogenization method	Drying conditions	Encapsulation agents	L^*	a^*	b^*	Reference
Orange (*Citrus sinensis*) concentrate juice		Direct addition of EA (33°Bx final concentration	Superheat steam 200 °C; steam 40 °C vacuum 5 kPa; vacuum jacket 50 °C	MDX 12DE 50%	88.8	−2.84	46.52	Islam et al. [67]
				MDX 12DE 60%	91.78	−3.99	37.39	
				MDX 12DE 70%	92.86	−3.53	31.22	
Chlorophylls								
Nannochloropsis oculata	Biomass growth		150/95 °C	None	54.5	−11.01	44.34	Palabiyik et al. [68]
			160/95 °C	None	56.95	−11.22	44.54	
			170/95 °C	None	56.39	−11.32	44.4	
			180/95 °C	None	56.52	−11.67	46.25	
			190/95 °C	None	55.13	−10.95	47.28	
			200/95 °C	None	56.57	−9.42	48.43	
Isochrysis galbana			150/95 °C	None	40.99	−3.74	49.54	
			160/95 °C	None	40.02	−4.08	41.12	
			170/95 °C	None	39.47	−4.58	42.21	
			180/95 °C	None	38.35	−4.97	42.55	
			190/95 °C	None	39.75	−5.4	44.16	
			200/95 °C	None	38.81	−5.41	44.19	
Freeze-dried chlorophylls	Pigment dilution (bergabest MCT oil 60/40)	Predissolved EA (30% w/v/60 °C/3 h; cooled 4 °C/12 h, 1.5% w/v tween 80/blender homogenization)/ ultraturrax homogenization (core-EA 2:1, 20,500 rpm/3 min)	145/95 °C; rotary atomizer 10 × 10 kPa	GA:MD (5:5) 30%	21.55	−12.1	13.39	Kang et al. [69]
				GA:MD (3:7) 30%	25.41	−17.4	20.08	
				GA:MD (0:10) 30%	26.22	−16.72	19.64	

Table 1 (continued)

Material	Extraction method	Homogenization method	Drying conditions	Encapsulation agents	L^*	a^*	b^*	Reference
Moringa oleifera Lam leaf juice	Blanched leaves/mechanical extraction (10 g/25 mL water)/filtration	Direct addition of EA	116 °C	MDX 12DE 14%	62	−1	31	Looi et al. [70]
			120 °C	MDX 12DE 8%	64	0	28	
			120 °C	MDX 12DE 20%	70	0	26	
			130 °C	MDX 12DE 5.5%	66	3	26	
			130 °C	MDX 12DE 14%	66	1	29	
			130 °C	MDX 12DE 14%	59	−1	31	
			130 °C	MDX 12DE 14%	73	0	22	
			130 °C	MDX 12DE 14%	74	0	21	
			130 °C	MDX 12DE 14%	57	−1	34	
			130 °C	MDX 12DE 22.5%	70	−1	25	
			140 °C	MDX 12DE 8%	60	−1	29	
			140 °C	MDX 12DE 20%	63	−1	30	
			144 °C	MDX 12DE 14%	67	0	26	
Spinach leaves (*Spinach oleracea*)	Dried ground leaves maceration (ethanol 80%/24 h)/stabilization (115 °C/30 min/zinc 300 ppm)/concentration (RotEva)	Diluted extract (1:1)/ direct addition of EA	180/80 °C; rotatory disc 28,140 rpm	Agave fructans 0.5%	24.12	4.93	9.92	Femat-Castañeda et al. [71]
				Agave fructans 1%	30.44	6.98	17.67	
				Agave fructans 1.5%	32.76	7.99	19.92	
				MDX 0.5%	24.08	3.88	9	
				MDX 1%	28.36	6.37	15.82	
				MDX 1.5%	32.62	7.88	19.55	
			190/80 °C; rotatory disc 28,140 rpm	Agave fructans 0.5%	25.12	4.85	9.12	
				Agave fructans 1%	30.6	7.04	26.3	
				Agave fructans 1.5%	32.36	5.46	19.94	
				MDX 0.5%	23.84	4.26	8.74	
				MDX 1%	30.29	6.99	17.3	
				MDX 1.5%	30.96	6.91	17.74	

Table 1 (continued)

Material	Extraction method	Homogenization method	Drying conditions	Encapsulation agents	L^*	a^*	b^*	Reference
Green kiwi (*Actinidia* sp.)	Mechanically pureed peeled fruit/centrifugation (5000×*g*/25 min/4 °C)/filtration	Direct addition of EA shear homogenization (500 rpm/1 min/3 bursts)	150 °C	Skim milk-MDX	82.3	−3.09	24.3	Sun-Waterhouse and Waterhouse [61]
				Skim milk-MDX (warmed feed)	93.6	−3.86	25.9	
		Direct addition of EA to extract and residue (skim milk, 40:60 ratio, MDX 3%)/shear homogenization (1000 rpm/1 min/3 bursts)		Skim milk-MDX	73.8	−7.82	27.8	
Others								
Chryseobacterium artocarpi	Pregrowth bacteria (nutrient broth, 30 °C/200 rpm/24 h)/centrifugation (10,000 rpm/10 min)/maceration (acetone 5%)/ultrasound assisted extraction (twice/20 s)/centrifugation (8000 rpm/10 min)/concentration (RotEva)	Direct addition of EA (2:1 v/w)/magnetic stirring homogenization	140–220/85 °C	GA	69.92	19.4	26.5	Venil et al. [72]
				κ-carrageenan	69.67	20.2	24.2	
Sargassum muticum	Soaking (1:10 w/v, water)/agitation (97 °C/210 min)/filtration		140 °C	MDX 10DE 4%		$C=16.17$	$H=261$	Tun Norbrillinda et al. [73]

MDX maltodextrin, *GA* gum arabic, *C* chrome, *H* hue, *RotEva* rotary evaporator, *EA* encapsulation agent, *wb* wet basis

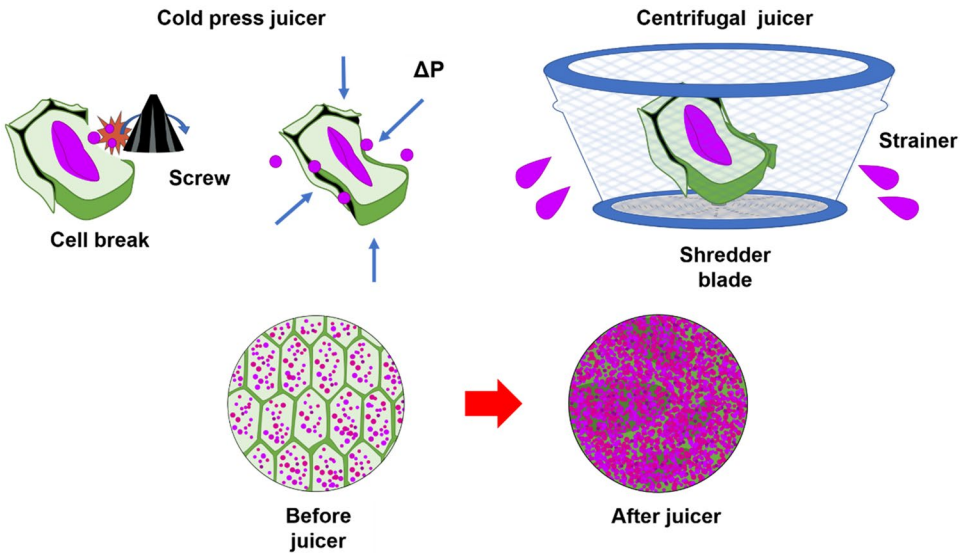


Fig. 3 Compound extraction by physical methods (adapted from [77])

safer. To carry out the extraction with supercritical fluids, CO_2 and propane are used. These gases are brought to their critical conditions of pressure and temperature, acting as fluids with high diffusivity, low viscosity, and high solvation capacity. These properties help to penetrate the food matrix, improving the extraction [82, 27]. The use of supercritical fluids works well for the extraction of low polarity compounds such as carotenoids and chlorophylls but not for the extraction of anthocyanins and betalains, due to their high polarity [27]. For the extraction of anthocyanins and betalains, the addition of modifiers such as ethanol, acetone, and water is necessary [28]. In supercritical extraction, since no solvents are used (which could be toxic), the extract obtained is safe for human consumption and, when working under reduced temperatures, the temperature-sensitive pigments are less affected. However, the cost of operation and equipment as well as gases is high [27].

Another homogenization technique is the high-pressure homogenization/microfluidization. It consists of reducing the size of the particles by passing a mixture at high speed, with the help of a pump, through a reduced orifice (disruption valve) for very short times (less than one second) [83–85]. Homogenization can be carried out as a continuous flow [84] and occurs within the valve; it is necessary to increase the pressure between 20 and 70 MPa to overcome the friction

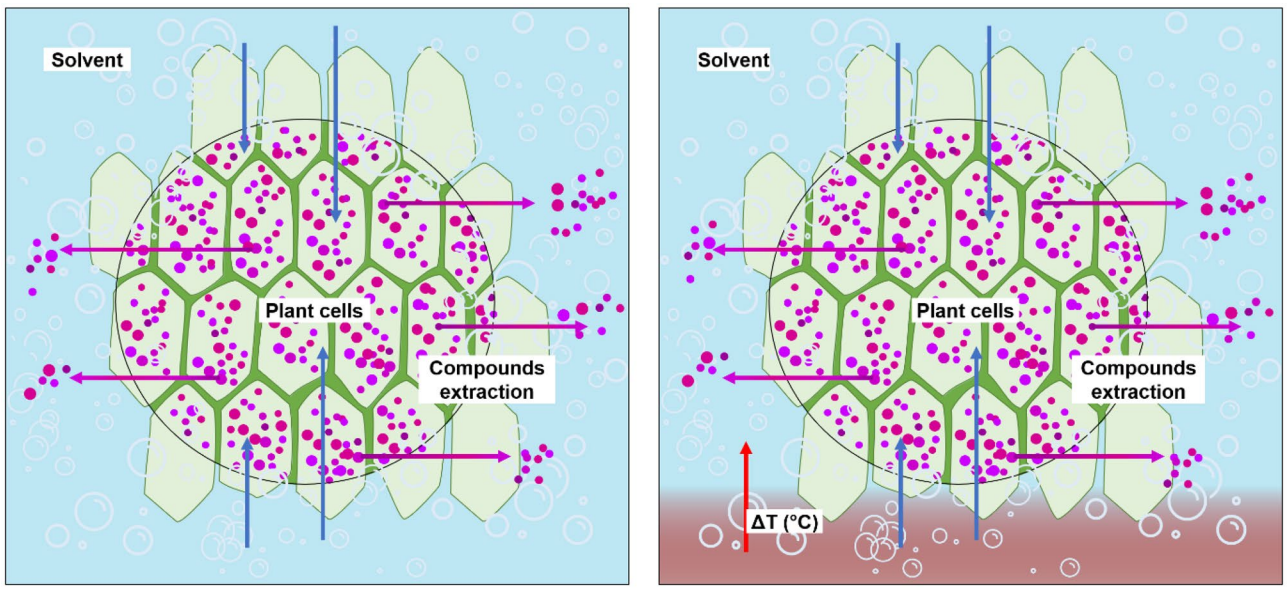


Fig. 4 Compound extraction by chemicals or solvents (adapted from [80])

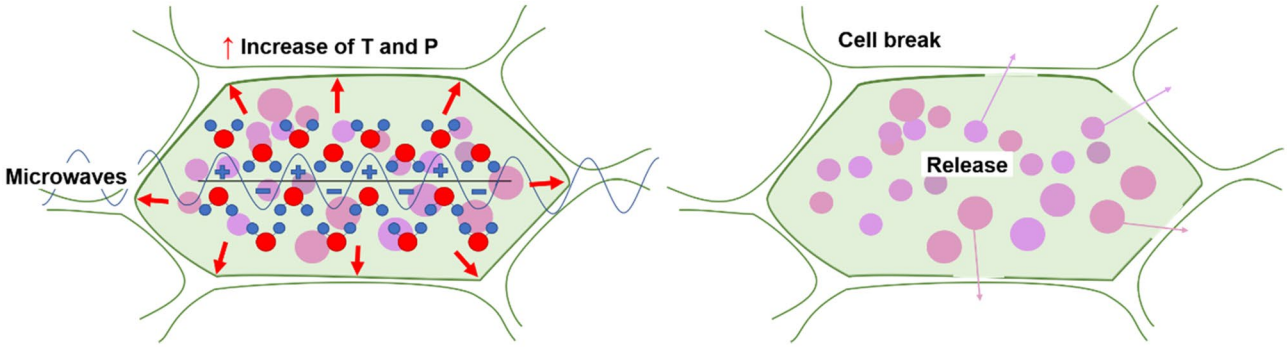


Fig. 5 Compound extraction assisted by microwaves (adapted from [88])

inside the valve [84, 85]. Three mechanisms are those that help the reduction of size: shear, cavitation, and turbulence. Cavitation is generated by the reduction in pressure below the water vapor pressure, generating evaporation and forming small bubbles that, when expanding and collapsing, generate cavitation that helps to reduce the size of the particles [84]. However, the use of this technique can increase the temperature of the fluid and affect its components [85].

Extraction with Assisted Methods

Based on the studies analyzed in this review (Table 1), microwaves and ultrasound are the most used technologies to assist the extraction of natural pigments, prior to encapsulation.

Microwaves improve the extraction of both water- and fat-soluble pigments. It consists of two oscillatory electromagnetic fields that help the penetration of the solvent inside the cells [28]. At the same time, the waves excite the water molecules, acting as a non-direct heating source, accelerating the transfer of energy, and reducing the thermal gradient [28]. Due to the rapid heating, the cell structure can break down and the compounds migrate from the interior of the cell to the solvent (Fig. 5) [86, 87].

Ultrasound is based on cavitation (generation, growth, and collapse of bubbles). When the bubbles implode, the "local" pressure and temperature rise up to 5000 K, causing the transfer of energy and at the same time the transfer of mass. Cavitation causes disruption of the cell wall, releasing the compounds (Fig. 6) [27, 89, 28]. It can be applied for obtaining water-soluble or fat-soluble pigments. The use of ultrasound decreases the extraction times and the amount of solvent and improves yield [90].

There are other methods that can promote mass transfer by rapid depressurization, electroporation of plant material, or changes in chemical bonds: CO_2 applied (pressure) in a supercritical way [92], pulsed electric fields (electroporation) [27, 28], high hydrostatic pressure, and high intensity electric discharges (changes in bonds) [93, 28].

However, the most common method for pigments extraction is maceration assisted by ultrasound. Gagneten et al. [94] analyzed the effect of ultrasound on the extraction of pigments from black currant (*Ribes nigrum*), raspberry (*Rubus idaeus*) and elderberry (*Sambucus nigra*) for their subsequent encapsulation. They observed that only two cycles of ultrasound were necessary for raspberry, three cycles for blackcurrant (10 min at 30 kHz/100% amplitude), and three cycles of 5 min at the same conditions for elderberry to extract the greatest amount of compounds. Other researchers have focused on the extraction of pigments from different parts of plants: blue corn grains (*Zea mays* L.) with ethanol-ultrasound [40], *Rosa rugosa* residues with acidified ethanol-ultrasound [44], chokeberry (*Aronia melanocarpa*) with acetone-ultrasound [45], and elderberry (*Sambucus nigra*) with acetone-ultrasound [38]. It has also been used to obtain flexirubin from *Chryseobacterium artocarpi* (bacterium) with acetone-ultrasound [72]. Other researchers have reported the use of microwaves to obtain betalains from bracts of bougainvillea (*Bougainvillea glabra*), using water as a solvent [55].

Extraction by Biological Non-conventional Methods

It has been observed that the use of enzymes as pretreatment can help the extraction of chemical compounds [95] combined with maceration. Enzymes can break down compounds that form the cell wall (pectin, cellulose, and hemicellulose), helping to release pigments (Fig. 7) [96, 97]. On the other hand, fermentation in liquid or solid state can also help to extract pigments. During fermentation, different enzymes are produced by microorganisms [98]. A combination of enzymes (Pectimex Yltra SP-L and Cellubix) has been used as a pretreatment of blueberry (*Vaccinium corymbosum* var. Elliot) bagasse for obtaining pigments; therefore, the bagasse was macerated with a citric acid solution to obtain pigments [35]. The authors mention that (a) the enzymatic treatment helped to extract anthocyanins, without affecting the physical characteristics of the powder obtained; (b)

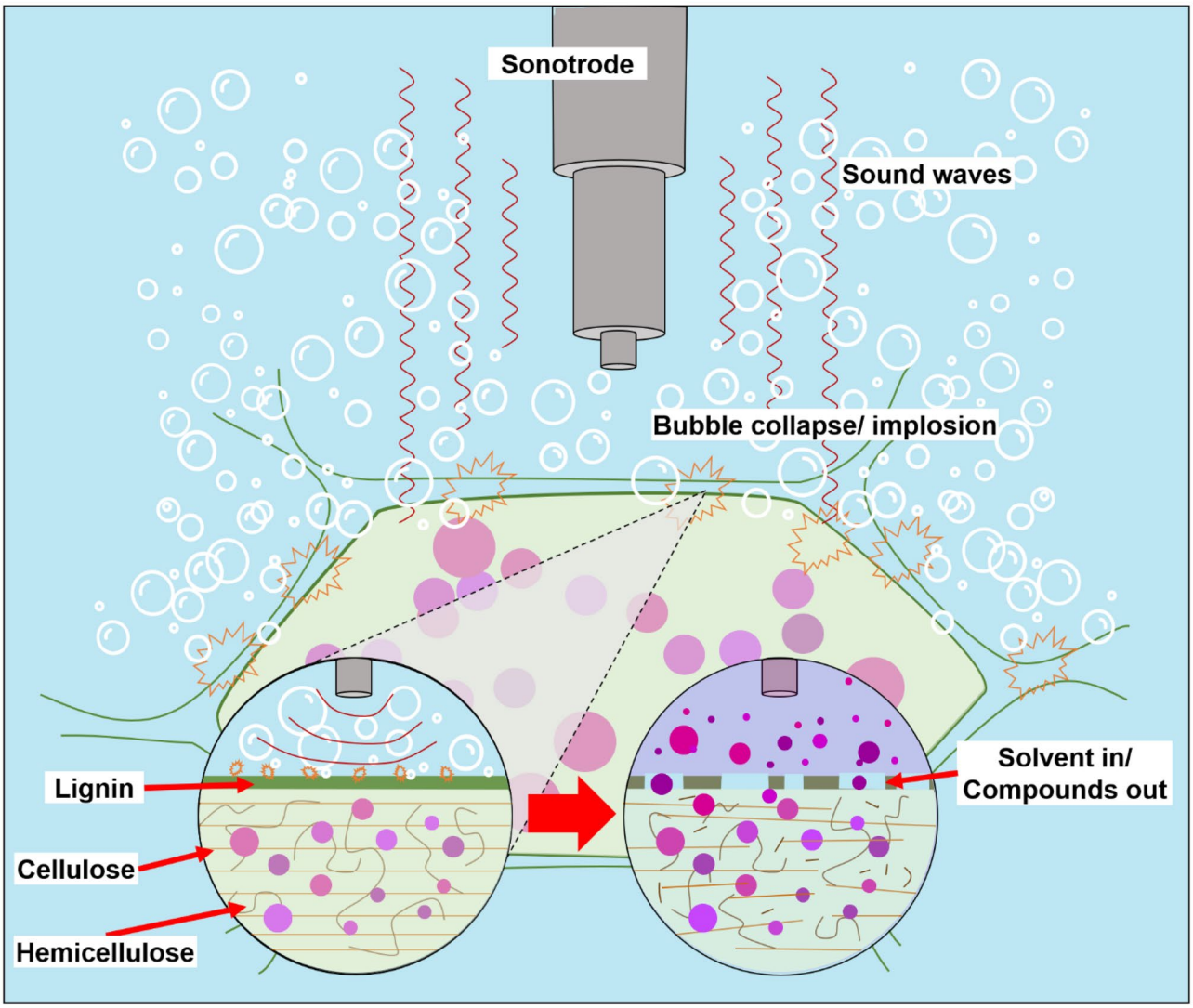


Fig. 6 Compound extraction by ultrasound assisted method (adapted from [91])

enzyme treatment left the extracted compounds exposed to thermal degradation during the drying, possibly due to the hydrolysis of compounds such as pectin that can help in encapsulation to form the wall; (c) the stability of the powders during storage did not improve; and (d) the costs and time of obtaining the compounds increased due to the extra step and the use of enzymes. Another important factor is the need to inactivate the enzymes, since some pigments present certain instability in their presence.

Pretreatments Before Spray Drying

The basic steps before spray drying consist of the extraction of the compounds of interest from the plant material; therefore, filtration and concentration are required; however, in some cases, there are other steps prior to obtain the powder by spray drying.

Concentration

The concentration of the extract is necessary after extraction of compounds by simple maceration or maceration assisted with any other technology such as ultrasound or microwaves.

Rotary Evaporator The most common method for concentration of compounds in extracts is applying evaporation at atmosphere or vacuum conditions. This consists of the removal of the solvent without the excessive use of heat. Due to the reduction of the pressure in the system, that lower the boiling point of the solvent, the extract remains relatively "intact" [100]. The use of a rotary evaporator has been

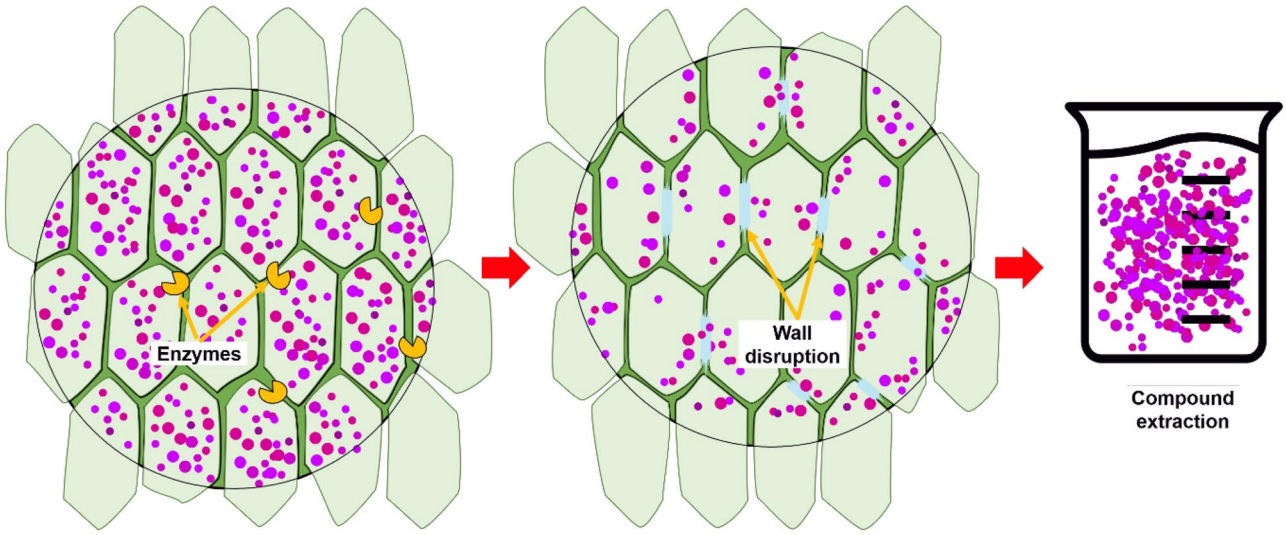


Fig. 7 Compounds extraction by enzymatic treatment method (adapted from [99])

reported in several research studies; the equipment is used to concentrate extracts of all types of pigments (Table 1). The typical temperature used for evaporation is between 35 and 55 °C, depending on the type of solvent used for the pigments extraction. A list of different solvents and suggestions for their distillation can be found in the literature of the Büchi company (manufacturer of spray dryers, rotary evaporators, and other equipment) [101] and by Portmann et al. [102]. As an example, Kaimainen et al. [103] used a rotary evaporator for concentration purple beet extracts. They pointed out that the intensity of the aroma and aftertaste of betalains from beets, microencapsulated with maltodextrin added in juices, decreased. They hypothesize that the decrease in aroma and aftertaste characteristics was due to the fact that in the evaporation, the beet aromatic compounds were removed.

Membranes Another unit operation is the concentration by ultrafiltration by membranes. It is based on the separation of compounds from a solution by applying hydrostatic pressure. The solution is obtained with the compounds that permeate the membrane and those that do not; it does not involve temperatures or chemical agents and is highly efficient [104]. It has been used for the concentration of anthocyanins, betalains, and carotenoids [104–108]. In some cases, it is necessary to carry out enzymatic liquefaction of the material prior to separation. Quirós et al. [108] studied the effect of both concentration at vacuum conditions and ultrafiltration for concentrating anthocyanins extracts from blackberry residues (*Rubus adenotrichos* Schltdl.). The concentration at vacuum conditions was 40 °C/70 mmHg/4 h, until reaching half of the original volume. For the ultrafiltration, the conditions were as follows: cross-flow membrane/2 kDa/total effective membrane area of 0.0139 m^2/constant temperature 35 °C/20 bar (permeate was collected every 5 min). Later on, the concentrated anthocynins were microencapsulated by spray drying using maltodextrin as carrier. The results showed that both concentrations at vacuum and ultrafiltration retained anthocyanins (92.8 and 48.4%, respectively), being the vacuum approach the most effective in pigments retention; both the color and the amount of anthocyanins were higher than in the membranes approach.

Heating

Other researchers suggest that heating prior to the extraction (blanching of the plant material) or to the extract (pasteurization) may help with the inactivation of enzymes or reduction of the natural microbial load. Hernández-Martínez et al. [56] boiled the extract of beet (*Beta vulgaris*) in order to inactivate periplasmic enzymes, which can degrade pigments to an unstable form [109]. On the other hand, blanching of the material can help with the extraction of pigments due to the cells disruption [70]. However, the heat on both pretreatments could cause a loss of pigments due to deglycosylation, decarboxylation, dehydrogenation or isomerization reactions [110], or leaching in the liquid used during blanching [70]. Sun-Waterhouse and Waterhouse [61] mention that heating of the mixture (50 °C) before spraying can also inactivate enzymes such as polyphenol oxidase and peroxidase, helping to maintain pigments and their color;however, there could be changes in the physicochemical and bioactive properties [61]. Janiszewska-Turak [24] evaluated the effect of blanching (90 °C/5 min) of carrots (*Daucus carota)* before the extraction of pigments for encapsulation; they reported a decrease

in carotenoids content and an increase in the particle size and density of the powders.

In addition to the inactivation of enzymes, preheating of the extract to be fed to the spray dryer can decrease the viscosity, avoiding the clogging of the atomizer and optimizing the drying efficiency [61]. This could be due to a better solubilization of compounds in the extract such as pectins or due to the rheological behavior of encapsulating agents, since some tend to decrease their viscosity when increasing temperature.

Biological Pretreatments

Some researchers have focused on reducing the viscosity of the feeding mixture, as this could cause problems during spraying; to avoid this, enzymatic liquefaction can be used [111, 112, 63, 63]. The enzymatic treatment with Pectinex Ultra SP-L 1.5% (v/w), together with Celluclast 1% (v/w)/2.5 h/50 °C), of puréed banana (*Musa acuminata*) reduced 88% of the viscosity [112]. Other studies have reported a reduction of 94 (using Celluzyme and Pectinex Ultra, Pulp 1:1, 1%/3 h/37 °C) [113] and 97.6% (using Pectinex Ultra SP-L 1% v/w and Celluclast 0.5% v/w, for 2.5 h/50 °C) [113] of the viscosity of jackfruit puree (*Artocarpus heterophyllus*), before microencapsulation.

As it is well-known, microorganisms (fungi, bacteria, yeasts) can produce a great variety of enzymes [114] during respiration (cells growth) and fermentation (production of metabolites) processes. Thus, during fermentation, pectinases can be produced, which can replace commercial enzymes, since they have the advantage of not requiring special means for their production [115]. Along with the reduction in viscosity, due to enzymatic activity, fermentation degrades sugars, especially the fermentable ones, which are those of low molecular weight (low glass transition temperature) [116], helping to the powder obtained not to be adhered to the walls of the dryer [20]. Kumar and Giridhar [117] fermented extracts of *Basella rubra* L., rich in betalains, with *Saccharomyces cerevisiae* to reduce the amount of sugars, and the subsequent encapsulation by spray drying using maltodextrin as the encapsulating agent. In a previous work, the authors reported that the betalains content increased with the previous fermentation procedure [118]. Czyżowska et al. [119] observed that beet (*Beta vulgaris*) juice treated with different strains of lactic acid bacteria stabilized betalains as well.

Homogenization Methods

Before the spray drying process, the incorporation of encapsulating agents (carriers) is necessary. Carriers protect pigments. To carry out the incorporation of encapsulating agents, a homogenization method must be used. Some researchers have demonstrated the effect of the homogenization method on some characteristics of the powders obtained, for example, in particle size or hygroscopicity [120], which are very important for adequate stability and handling of the powder.

Magnetic Agitation

One of the most used methods to obtain a good mixture of extract and carrier is the magnetic stirring. It is the most common method and the most used in different industries. Its use is very common for water-soluble pigments (anthocyanins or betalains); however, it has also been used for mixtures of fat-soluble pigments (carotenoids) (Table 1). It consists of a magnet that creates a rotating magnetic field that rotates a bar (made of Teflon or glass, chemically inert that can be sterilized) immersed in the liquid to be homogenized with the encapsulating agent; the system may or may not include heating [121]. Some disadvantages are the amount or volume they can mix, the viscosity of the system, and the exposure to the environment, if the system is not closed [30]. Due to the low shear effect, the mixing times can be prolonged. Times between 30 and 60 min have been reported to carry out an adequate homogenization using speeds between 200 and 1000 rpm [38, 39, 45]. On the other hand, magnetic stirring has also been used to rehydrate encapsulating agents before mixing with pigments [51, 53, 56, 121]. This makes it possible to manipulate the encapsulating agents in case if the heating is necessary for a good homogenization of the agents before incorporating them to the pigments.

Mechanic Agitation (High Shearing)

If higher stirring force is needed for dissolving the encapsulating agent or mixing immiscible liquids, more powerful methods are used. High-speed or high-shear mixers such as ultraturrax have the advantage of being able to disperse an immiscible phase in the continuous phase. However, the speed will depend on the equipment used. Speeds of up to 20,500 rpm have been reported [69], with the advantage of using short mixing times (between 3 and 10 min), which depends on the speed (Table 1). Mechanical stirring is common for the preparation of fat-soluble pigment emulsions, such as carotenoids. The equipment consists of a motor that rotates a propeller, with fins (blades) at the bottom [121], to which different types of fins or heads can be easily adapted to reduce the particle size in the emulsion [122] and facilitate homogenization, improving the final physical properties of the powder. One of the disadvantages of using this type of equipment is the incorporation of oxygen due to high shear and the formation of the vortex during homogenization, which can affect the stability of the pigments.

Other Methods

Another method of homogenization is the use of ultrasound. This is based on the application of sound waves propagating in the liquid, creating small bubbles that, when collapse, generate cavitation/microjet phenomenon (generating pressures around 2000 atm), promoting the reduction of particle size [121]. In addition, it produces a rapid dissolution and homogenization of materials, due to the breakdown of molecular interactions [121]. The usual operating frequency conditions are between 16 and 100 kHz [123].

Selected Encapsulated Agents

The addition of encapsulating agents is necessary due to the low glass transition temperature (*Tg*) of some components in the extracts: glucose, fructose, and sucrose [124]. These components can cause powder to stick in the drying chamber, lowering yields, and creating problems with powder handling. Multiple researchers have mentioned that the selection of the encapsulating agent is very important, since it will influence the behavior of the powder. Encapsulating agents must be able to form a wall (a shield), be able to form an emulsion, have low viscosity at high concentrations, have low hygroscopicity, be biodegradable, be resistant to the gastrointestinal tract, and be stable and inert in the food product during processing, storage and consumption [23, 25, 43]. However, in some cases, the addition of the encapsulating agent may not be necessary. Pereira et al. [122] obtained a juçara (*Euterpe edulis*) powder without the addition of encapsulating agents. According to the authors, obtaining the powder without an encapsulating agent was due to the low amount of carbohydrates and organic acids.

Maltodextrin and Some Mixtures with Maltodextrin

The most used agents for the encapsulating pigments are maltodextrin (MDX), gum arabic (GA), starches from different sources, modified starches, proteins from different sources, gelatin, alginate and pectins (Table 1). The concentration of encapsulating agents varies widely; it can be from 1 to 50%. The selection of the type of encapsulated agents and concentration will depend on factors such as the optimum viscosity for the formation of drops during spraying and the characteristics of the equipment [122].

Maltodextrin has been used for the encapsulation of all types of pigments (Table 1). The use of maltodextrin as an encapsulating agent has been shown to improve the stability of pigments. In order to test whether the combination of MDX with other agents improves the properties of powders, different authors have used combinations with other agents, the most common being gum arabic (GA). Akhavan Mahdavi et al. [30] reported that the combination of MDX-GA for the encapsulation of barberry (*Berberis vulgaris*) anthocyanins presented the best encapsulation efficiency compared to mixtures such as MDX-gelatin or MDX alone. In this same study, the authors report that the MDX-GA blend had the best physical properties. Sarabandi et al. [39] observed that the combination of MDX-GA improved the performance of sour cherry (*Prunus cerasus* L.) anthocyanins, as well as MDX or GA with concentrated protein isolate by at least 10%; however, the different combinations had a decrease in the value of the a^* color parameter, unlike MDX alone. Jimenez-Gonzalez et al. [32] report more "vivid" colorful anthocyanins, obtained with MDX-GA from garden ginger (*Renealmia alpinia*), compared to those obtained with GA and MDX alone. The MDX-GA mixture has also been used in the encapsulation of beet betalains (*Beta vulgaris*); it was observed that the MDX (10DE)-GA mixture presented the highest yields and values in the a^* color parameter [48]. For the encapsulation of norbixin from annatto (*Bixa orellana*) seeds, Tupuna et al. [64] tested different ratios of MDX and GA; the 50:50 combination produced the best performance (about 80%), but an encapsulation efficiency of about 30%, unlike using GA: the efficiency and yield were about 50 and 60%, respectively. Kang et al. [69] observed in the encapsulation of chlorophylls that the increase in the amount of MDX with respect to GA improved the encapsulation efficiency and the stability of chlorophylls. They also observed that chlorophylls were bound encapsulating agents through hydrogen bonding and/or esterification [69].

Martins et al. [120] evaluated the effect of different combinations of carriers (binary and tertiary combinations of MDX, GA and chitosan) and size of the spray nozzle (0.7, 1.0, and 1.2 mm) on the encapsulation of Roselle calyxes (*Hibiscus sabdariffa*) anthocyanins. Unlike that reported by other authors, the best combination to obtain the powders was not with MDX-GA, but with GA-chitosan (using a 1 mm nozzle); the powder presented the best characteristics in anthocyanin content, antioxidant capacity, and homogeneous structure, but not the highest color intensity; this was for MDX, GA, or their combination.

The combination of MDX with pectin has been used to encapsulate blue corn (*Zea mays* L.) anthocyanins [40] and pitaya (*Stenocereus griseus*) betalains [40]. In both studies, the combination improved the stability of the pigments during storage: homogeneous and well-formed particles were obtained. The addition of pectin increased the *Tg*, which is related to some mechanisms of deterioration of the microcapsule and the active compound [51]. On the other hand, Ruiz Canizales et al. [125] mention that the combination of MDX and pectin favors the release of anthocyanins during the intestinal phase but reduce the absorption.

Mesquite Gum

Currently, a replacement for GA is mesquite gum (MesG). MesG is an exudate from the mesquite tree (*Prosopis* spp.), native to Mexico [126]. MesG is highly soluble in water and its solutions are of low viscosity with an amber color; its use is common in foods [127]. In general, few studies have reported the use of mesquite gum. Jiménez-Aguilar et al. [128] encapsulated blueberry (*Vaccinium corymbosum*) anthocyanins by spray drying. In this work, the drying temperature was the main factor that affected the degradation of the pigments. In a more recent study, Ochoa-Velasco et al. [31] encapsulated hibiscus calyxes (*Hibiscus sabdariffa*) anthocyanins. They tested different concentrations of MesG (1, 2, 3, 4, and 5%). The results obtained showed that increasing the amount of MesG in the feeding mixture in the spry drying had no effect on the moisture of the final product, but the yield increased. The authors mention that the excessive addition of gum to the extract generates powders with low nutritional quality due to the ratio between encapsulating agent/active compound. Furthermore, the increase in the concentration of MesG affected the color of the product, decreasing the values of the a^* color parameter. However, thanks to the use of MesG, the anthocyanins content and color can be maintained with slight changes, at least, up to 1 year under refrigerated conditions (4 °C).

Some Mucilages

Other authors have proposed the use of tender *Opuntia* cladodes [53, 50] and chia *(Salvia hispanica)* mucilages [54] for the encapsulation of betalains from cacti fruits (pitaya (*Stenocereus queretaroensis*) and jiotilla (*Escontria chiotilla*)) and beets, respectively. The cladodes mucilage, alone or in combination with MDX, showed good results in the encapsulation and stabilization of the pigments of pitaya (*S. queretaroensis*) and jiotilla (*E. chiotilla*). The encapsulated pigments showed low moisture content, uniform and spherical shape, and the advantage of containing a high content of dietary fiber. On the other hand, chia mucilage (especially that obtained by lyophilization) improved the retention capacity of betalains from beet (*Beta vulgaris*) in the encapsulated agents compared to the use of MDX alone or the MDX-GA mixture. The authors also report good stability at different pHs and temperatures, when powders were dissolved.

Fructans

Fructans, alone or in combination, have also been used for pigments encapsulation. Additionally, they can act as prebiotics in the intestines. Fructans, found in different varieties of plants and produced by some bacteria and fungi, are widely used in the food industry as substitutes for fat [129] and for the encapsulation of pigments (Table 1). Femat-Castañeda et al. [71] used agave (*Agave* spp.) fructans for chlorophylls encapsulation. Fructans produced more stable encapsulates, compared to those obtained with MDX. Both MDX and fructans, under normal storage conditions, extending the shelf life of encapsulated chlorophylls. In the same study, it was shown that the *Tg* of fructans is lower than that of MDX which can influence the drying and storage temperature. Fructans have also been used for the encapsulation of anthocyanins [45, 35] and betalains [57, 55].

Other Agents

On the other hand, Rubio et al. [36] used yeast biomass as an encapsulating agent of anthocyanins from Cabernet Sauvignon (*Vitis vinifera*) and *Vitis labrusca* grapes. They reported that brewer's yeast worked well as an encapsulating agent. The authors also demonstrated that the powder obtained had adequate characteristics for storage: low moisture content, low hygroscopicity, and a particle size not sensory perceptible. Yeast, in addition to helping to keep pigments stable, made them more bio-accessible during gastrointestinal digestion compared to free pigments.

Color Values of Spray-Dried Pigments

Color

Encapsulation protects and extends the shelf life of pigments; however, encapsulating agents may cause changes in color of the pigments. The most common color measurement system in foods is the *CIEL*a*b** system. It provides more uniform color differences according to what is perceived by the human eye [130]. The system consists of two color coordinates (a^*, green-red and b^*, blue-yellow) and one of luminosity (L^*, black-white). Table 1 shows the values of L^*, a^*, and b^* color parameters obtained after encapsulation of different natural pigments. The tristimulus values of the *CIEL*a*b** system are used to calculate the color attributes *Chroma* (color saturation or purity, *C*) and *Hue* angle (H °, *hue*): 0° = red, 90° = yellow, 180° = green and 270° = blue. The calculation of these two color attributes is obtained by the following equations (Eqs. 1 and 2).

$$Chroma(C*) = \sqrt{a^{*2} + b^{*2}} \tag{1}$$

$$Hue(H^\circ) = \tan^{-1}\left(\frac{b^*}{a^*}\right) \tag{2}$$

However, it is necessary to take into account the following considerations to obtain the correct angle according to

the color diagram: (i) the reported values must be in degrees; (ii) if a^* and b^* are positive, the value of $H°$ is kept as is since it is in the first quadrant (red-yellow, 0–90°) of the color space; (iii) if a^* is negative and b^* is positive, the color is in the second quadrant (yellow-green, 90–180°) of the color space; therefore, it is necessary to add 180° to the value of $H°$; (iv) if a^* and b^* are negative, the color is in the third quadrant (green-blue, 180–270°) of the color space, and it is necessary to add 180° to the value of $H°$; (v) if a^* is positive and b^* negative, the value of $H°$ is in the fourth quadrant (blue-red, 270–360° (0°)) of the color space, and it is necessary to add 360° to the value of $H°$ [131]. A representation is shown in Fig. 8.

Some studies show the relationship between loss of color and loss of pigments. Tools such as statistics and probabilistic are used to observe the behavior of the encapsulation materials and the active compound under different storage conditions. The changes that occur during storage depend on physical factors such as temperature, exposure to light, and oxygen, among others. Table 2 summarizes some works that have studied the degradation of different compounds under different storage conditions.

Color Degradation

The degradation of pigments (anthocyanins, betalains, carotenoids, and chlorophylls) can occur in different ways. Anthocyanins, when found in aqueous media with changes in pH, can change their color due to changes in their structure. Anthocyanins exist as flavylium cation at pHs less than 2, at pHs between 3 and 6; they exist as a carbinol pseudobase which leads to their chalcone form, both colorless [151]. Due to thermal processes, polymerization or bond breakage may occur; however, this depends on type of anthocyanidin, source, and degree of polymerization, pH, and temperature, among other factors [151]. On the other hand, betacyanins can have changes due to isomerization, hydrolysis, decarboxylation and dehydrogenation causing changes in color [152]. Regarding carotenoids, the isomerization of the E-carotenoids to Z-carotenoids molecules, caused by heat, acids or exposure to light, is accompanied by oxidation and loss of color [152, 152]. Finally, the main change in chlorophylls is due to the formation of pheophytin produced by heat treatments or pH reduction [153].

The color of natural pigments can degrade due to different physicochemical factors. The kinetic model that describes the behavior of the data usually complies with first order kinetics (Eqs. 3 and 4). Sometimes a two-stage behavior has been reported, where the first stage corresponds to the degradation of the pigments on the surface of the microcapsules and the second stage corresponds to the degradation of the pigments inside the capsule [132, 139].

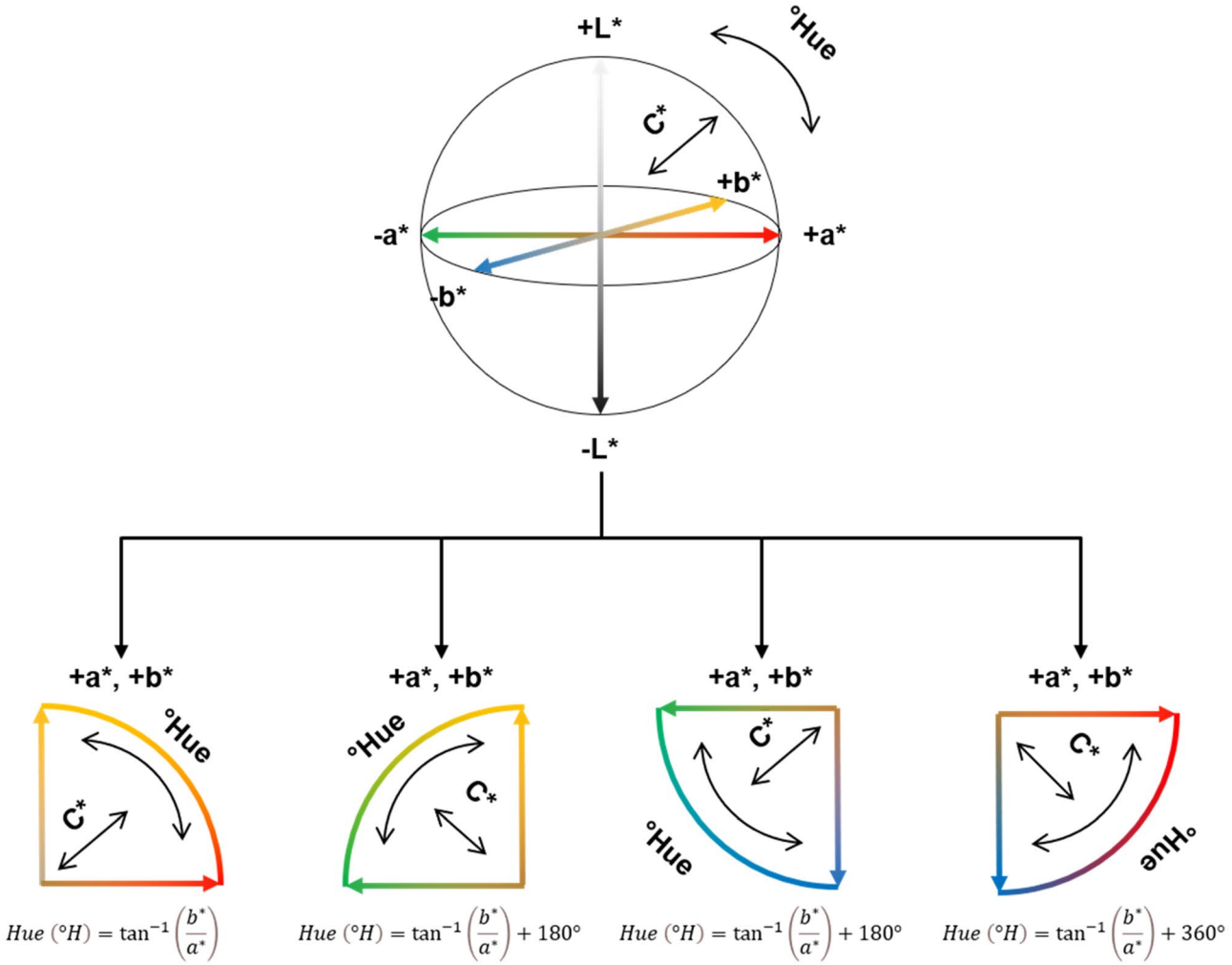
$$Log\left(\frac{C(t)}{C_0}\right) = -kt \tag{3}$$

$$t_{1/2} = -\frac{\ln(2)}{k} \tag{4}$$

where C_0 corresponds to the initial concentration of pigments present in the microcapsule, C (t) is the concentration of pigments at different storage times (t) (min, h, days, or months), k is the first-order kinetic constant (/min, /h, / day, or /month), and $t_{1/2}$ is the half-life time (min, h, days, months) of pigment degradation. Next, some physicochemical factors that affect the degradation of microencapsulated pigments are discussed.

Effect of Temperature The increase in temperature during storage generates an increase in the values of the degradation constant k, which is reflected in the loss of pigments and a shorter $t_{1/2}$ [133, 128, 44, 40, 64, 146]. On the contrary, the refrigeration temperatures decrease the value of k, since storage at low temperatures limits the mobility of the water molecules present in the microencapsulated pigments. Temperatures below the *Tg* make pigment degradation reactions less likely to occur. However, the reduction in temperature below *Tg* is not the only parameter on which the stability of the microencapsulated pigments depends [154].

Effect of the Encapsulating Agent Tonon et al. [132] observed the influence of four types of encapsulating agents (GA, MDX 10DE, MDX 20DE, tapioca starch) on the stability of açai (*Euterpe oleracea*) anthocyanins. They maintained the storage conditions at two temperatures (25 and 35 °C) and two different a_w values (0.328, 0.529). They observed that MDX 10DE generated the most resistant microencapsulated pigments in all the storage conditions evaluated, showing lower k values compared to the microencapsulates of MDX 20DE, GA, and tapioca starch. In the encapsulation of blackberry (*Rubus adenotrichus*) anthocyanins, MDX presented better stability compared to GA alone and the MDX:GA (1:1) mixture [134]. Pavón-García et al. [136] showed that the addition of mesquite gum increased the stability of maltodextrin when applied to muicle (*Justicia spicigera*) anthocyanins at 20, 35 and 40 °C and different relative humidities. The addition of cladodes mucilage and MDX in the encapsulation of prickle pear (*Opuntia ficus-indica*) betalains decreased the life time of the microencapsulated pigments by almost half time than that of using only MDX [50]. The use of modified starches can improve or increase the stability of the pigments; such is the case of the use of Capsul or Hi-cap (commercial brands) in the encapsulation of carrot (*Daucus carota)* carotenoids: the degradation constant values and the half-life time values decreased more



a*	b*	$\tan^{-1}$(b*/a*) without considerations	$\tan^{-1}$(b*/a*) with considerations
20.00	35.00	60.26	60.26
-20.00	35.00	-60.26	119.74
-20.00	-35.00	60.26	240.26
20.00	-35.00	-60.26	299.74

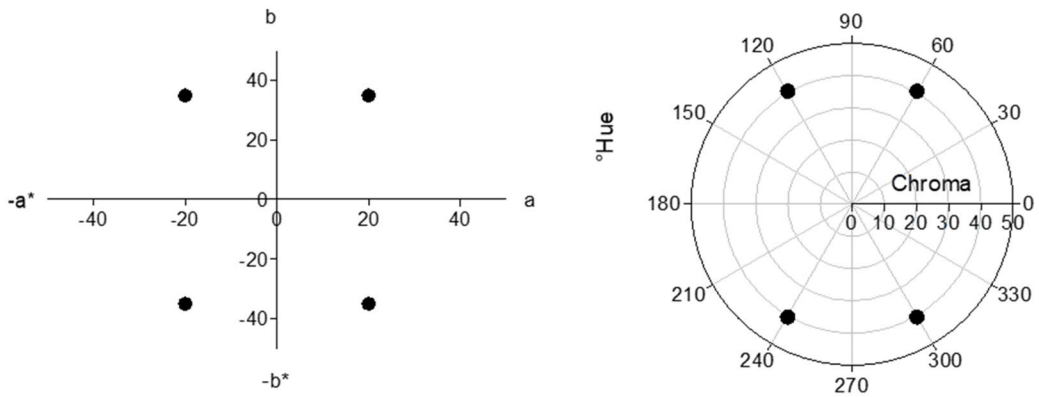


Fig. 8 Schematic considerations to hue value calculation

Table 2 Degradations kinetics modeling of spray drying encapsulated pigments throughout storage

Product	Storage condition	Wall material	K (constant rate) (/days)	$t_{1/2}$ (half-life)	Reference
Açai (*Euterpe oleracea*)	$a_w = 0.328$ $T = 25$ °C	MDX 10DE	1.4×10^{-3a}–0.5×10^{-3b}	1248.29 (days)	Tonon et al. [132]
	$a_w = 0.529$ $T = 25$ °C		2.6×10^{-3a}–0.6×10^{-3b}	962.58 (days)	
	$a_w = 0.328$ $T = 35$ °C		2.1×10^{-3a}–0.7×10^{-3b}	880.35 (days)	
	$a_w = 0.529$ $T = 35$ °C		5.7×10^{-3a}–1.0×10^{-3b}	411.55 (days)	
	$a_w = 0.328$ $T = 25$ °C	MDX 20DE	3.2×10^{-3a}–0.6×10^{-3b}	909.58 (days)	
	$a_w = 0.529$ $T = 25$ °C		4.5×10^{-3a}–0.7×10^{-3b}	677.64 (days)	
	$a_w = 0.328$ $T = 35$ °C		3.9×10^{-3a}–1.1×10^{-3b}	516.15 (days)	
	$a_w = 0.529$ $T = 35$ °C		8.4×10^{-3a}–1.0×10^{-3b}	260.55 (days)	
	$a_w = 0.328$ $T = 25$ °C	GA	2.9×10^{-3a}–0.6×10^{-3b}	978.75 (days)	
	$a_w = 0.529$ $T = 25$ °C		4.9×10^{-3a}–0.6×10^{-3b}	826.25 (days)	
	$a_w = 0.328$ $T = 35$ °C		4.1×10^{-3a}–0.7×10^{-3b}	767.07 (days)	
	$a_w = 0.529$ $T = 35$ °C		9.1×10^{-3a}–1.0×10^{-3b}	328.55 (days)	
	$a_w = 0.328$ $T = 25$ °C	Tapioca starch	1.5×10^{-3a}–0.8×10^{-3b}	813.81 (days)	
	$a_w = 0.529$ $T = 25$ °C		3.0×10^{-3a}–0.8×10^{-3b}	695.18 (days)	
	$a_w = 0.328$ $T = 35$ °C		2.8×10^{-3a}–0.9×10^{-3b}	655.27 (days)	
	$a_w = 0.529$ $T = 35$ °C		5.7×10^{-3a}–1.8×10^{-3b}	248.14 (days)	
Black carrot (EX) (*Daucus carota*)	$T = 4$ °C dark	Stardri 10 (10DE)	0.8×10^{-3}	28 (months)	Ersus and Yurdagel [133]
	$T = 25$ °C dark		2.3×10^{-3}	10 (months)	
	$T = 4$ °C dark	Glucodry 210 (20–23DE)	0.8×10^{-3}	28 (months)	
	$T = 25$ °C dark		2.3×10^{-3}	10 (months)	
	$T = 4$ °C dark	MDX 28–31DE	0.9×10^{-3}	25 (months)	
	$T = 25$ °C dark		2.5×10^{-3}	9 (months)	
	$T = 25$ °C light (3000 lx)	Stardri 10 (10DE), Glucodry 210 (20–23DE), MDX 28–31DE	2.4×10^{-3}	9 (months)	
Blackberry (*Morus nigra*)	$T = 25$ °C 32.8% RH	MDX	0.0019	373.8 (days)	Ferrari et al. [134]
	$T = 35$ °C 35.8% RH		0.0032	213.86 (days)	
	$T = 25$ °C 32.8% RH	GA	0.0023	297.48 (days)	
	$T = 35$ °C 35.8% RH		0.0031	225.36 (days)	
	$T = 25$ °C 32.8% RH	MDX-GA (1:1)	0.002	347.68 (days)	
	$T = 35$ °C 35.8% RH		0.0027	253.15 (days)	
Blueberry (*Vaccinium corymbosum*)	$T = 4$ °C dark	Mesquite gum	0.0185 (/week)	37 (weeks)	Jiménez-Aguilar et al. [128]
	$T = 25$ °C light		0.0681 (/week)	10 (weeks)	
	$T = 25$ °C dark		0.0415 (/week)	17 (weeks)	
Jucara (*Euterpe edulis* M.)	$T = 40$ °C 75% RH	GA (5%) DT = 160 °C	48.78×10^{-3}	14.2 (days)	Bicudo et al. [135]
		Gelatin (5%) DT = 160 °C	78.20×10^{-3}	8.9 (days)	
		MDX (5%) DT = 160 °C	62.03×10^{-3}	11.2 (days)	
Justicia spicigera	$a_w = 0.555$ $T = 20$ °C	GA-MDX (1:1)	1.06×10^{-3}	653.91 (days)	Pavón-García et al. [136]
	$a_w = 0.592$ $T = 35$ °C		1.37×10^{-3}	505.94 (days)	
	$a_w = 0.627$ $T = 40$ °C		1.46×10^{-3}	474.75 (days)	
	$a_w = 0.581$ $T = 20$ °C	Mesquite gum-MDX (1:1)	0.87×10^{-3}	790.77 (days)	
	$a_w = 0.587$ $T = 35$ °C		1.04×10^{-3}	666.48 (days)	
	$a_w = 0.648$ $T = 40$ °C		1.20×10^{-3}	577.62 (days)	
Rose (*Rosa rugosa*)	$T = 70$ °C	GA-MDX (1:1)	1.45×10^{-3}	7.97 (h)	Yu and Lv [44]
	$T = 80$ °C		2.28×10^{-3}	5.07 (h)	
	$T = 90$ °C		4.07×10^{-3}	2.84 (h)	

Table 2 (continued)

Product	Storage condition	Wall material	K (constant rate) (/days)	$t_{1/2}$ (half-life)	Reference
Blue maize (*Zea mays* L.)	$T=25$ °C	MDX	7.47×10^{-4}	93.13 (days)	Ruiz Canizales et al. [40]
	$T=35$ °C		8.77×10^{-4}	81.47 (days)	
	$T=45$ °C		9.97×10^{-4}	70.56 (days)	
	$T=25$ °C	MDX-pectin	5.80×10^{-4}	120.87 (days)	
	$T=35$ °C		7.37×10^{-4}	94.51 (days)	
	$T=45$ °C		8.13×10^{-4}	85.48 (days)	
Red onion (*Allium cepa*)	$a_w=0.108$ $T=35$ °C	MDX	3.52×10^{-3}	196.92 (days)	Pascual-Pineda et al. [137]
	$a_w=0.215$ $T=35$ °C		1.95×10^{-3}	355.46 (days)	
	$a_w=0.318$ $T=35$ °C		2.37×10^{-3}	292.47 (days)	
	$a_w=0.436$ $T=35$ °C		7.41×10^{-3}	93.54 (days)	
	$a_w=0.515$ $T=35$ °C		9.99×10^{-3}	69.38 (days)	
	$a_w=0.627$ $T=35$ °C		22.26×10^{-3}	31.14 (days)	
	$a_w=0.742$ $T=35$ °C		28.91×10^{-3}	23.98 (days)	
	$a_w=0.821$ $T=35$ °C		27.88×10^{-3}	24.86 (days)	
Wine grape by-products (seeds and skin)	32.8% RH $T=25$ °C	MDX (10%) DT = 130 °C	0.00294	233.5 (days)	de Souza et al. [138]
	32.8% RH $T=25$ °C	MDX (10%) DT = 150 °C	0.0022	311.31 (days)	
	32.8% RH $T=25$ °C	MDX (10%) DT = 170 °C	0.0026	267.4 (days)	
	32.8% RH $T=25$ °C	MDX (20%) DT = 130 °C	0.0019	365 (days)	
	32.8% RH $T=25$ °C	MDX (20%) DT = 150 °C	0.0018	383.4 (days)	
	32.8% RH $T=25$ °C	MDX (20%) DT = 170 °C	0.0015	461.2 (days)	
	32.8% RH $T=25$ °C	MDX (30%) DT = 130 °C	0.0016	441.1 (days)	
	32.8% RH $T=25$ °C	MDX (30%) DT = 150 °C	0.0018	381.1 (days)	
	32.8% RH $T=25$ °C	MDX (30%) DT = 170 °C	0.0018	391.3 (days)	
Cactus fruit (*Opuntia megacantha*)	$T=18$ °C 90% RH	MDX-CM (1:0.225)	1.85×10^{-3}	37.48 (days)	Otálora et al. [49]
	$T=18$ °C 57% RH	MDX-CM (1:0.225)	1.11×10^{-3}	62.68 (days)	
Cactus fruit (*Opuntia ficus-indica*)	90% RH $T=18$ °C	EX-MDX-CM (1:1:0.225)	0.049	13.9 (days)	Otálora et al. [50]
	75% RH $T=18$ °C		0.013	51 (days)	
	57% RH $T=18$ °C		0.012	54.1 (days)	
	90% RH $T=18$ °C	EX-MDX (1:1)	0.029	23.8 (days)	
	75% RH $T=18$ °C		0.007	103.4 (days	
	57% RH $T=18$ °C		0.006	117.4 (days)	
Cactus pear (*Opuntia ficus-indica*)	$T=60$ °C, force air oven	Pulp-MDX	1.06×10^{-2b}	1.9 (months)	Saénz et al. [139]
		Pulp-Inulin	1.07×10^{-2b}	2.1 (months)	
		EX-MDX	7.84×10^{-2a}–1.28×10^{-2b}	9 (days[a])–54 (days[b])	
		EX-Inulin	3.07×10^{-2a}–0.58×10^{-2b}	23 (days[a])–4 (months[b])	
Purple cactus Pear (*Opuntia ficus-indica*)	$T=60$ °C, force air oven	Pulp-SPI	0.9×10^{-2}	2.6 (months)	Robert et al. [140]
		Pulp-SPI + MDX (1:1)	0.5×10^{-2}	4.6 (months)	
		Pulp-SPI + Inulin (1:1)	0.8×10^{-2}	2.9 (months)	

Table 2 (continued)

Product	Storage condition	Wall material	K (constant rate) (/days)	$t_{1/2}$ (half-life)	Reference
Beetroot (*Beta vulgaris*)	pH 3 buffer sol (30 °C)	MDX	158.9×10^{-3}	4.3 (days)	Antigo et al. [47]
	pH 4 buffer sol (30 °C)		163.2×10^{-3}	4.2 (days)	
	pH 5 buffer sol (30 °C)		189.4×10^{-3}	3.6 (days)	
	pH 6 buffer sol (30 °C)		215.6×10^{-3}	3.2 (days)	
	pH 3 buffer sol (30 °C)	MDX-XG	127.3×10^{-3}	5.4 (days)	
	pH 4 buffer sol (30 °C)		119.1×10^{-3}	5.8 (days)	
	pH 5 buffer sol (30 °C)		108.4×10^{-3}	6.3 (days)	
	pH 6 buffer sol (30 °C)		118.8×10^{-3}	5.8 (days)	
Amaranthus	32% RH $T=25$ °C	MDX 10DE	0.0159 (/week)	43.6 (weeks)	Cai and Corke [141]
	32% RH $T=25$ °C	MDX 25DE	0.0141 (/week)	49.02 (weeks)	
	32% RH $T=25$ °C	MDX 10DE-MDX 25DE (1:3)	0.0109 (/week)	63.6 (weeks)	
Cactus pear (Betaxanthins)	$T=20$ °C dark	MDX 6DE	3.25×10^{-3}	7.10 (months)	Fernández-López et al. [142]
	$T=4$ °C dark		0.43×10^{-3}	4.41 (years)	
	$T=-20$ °C dark		0.23×10^{-3}	8.34 (years)	
Rosa mosqueta (*Rosa rubiginosa*)	$T=25$ °C dark	Starch	6.7×10^{-3} (/h)	103.5 (h)	Robert et al. [143]
	$T=40$ °C dark		2.5×10^{-2} (/h)	27.7 (h)	
Trans-β-carotene	$T=55$ °C dark		5.1×10^{-2} (/h)	13.6 (h)	
	$T=25$ °C dark	Gelatin	1.9×10^{-3} (/h)	364.8 (h)	
	$T=40$ °C dark		9.5×10^{-3} (/h)	73 (h)	
	$T=55$ °C dark		3.7×10^{-2} (/h)	18.7 (h)	
Trans-L-lycopene	$T=25$ °C dark	Starch	7.5×10^{-3} (/h)	92.4 (h)	
	$T=40$ °C dark		2.2×10^{-2} (/h)	31.5 (h)	
	$T=55$ °C dark		4.0×10^{-2} (/h)	17.3 (h)	
	$T=25$ °C dark	Gelatin	4.6×10^{-3} (/h)	150.7 (h)	
	$T=40$ °C dark		1.5×10^{-2} (/h)	46.2 (h)	
	$T=55$ °C dark		4.1×10^{-2} (/h)	16.9 (h)	
Trans-rubixanthin	$T=25$ °C dark	Starch	8.0×10^{-3} (/h)	86.6 (h)	
	$T=40$ °C dark		2.9×10^{-2} (/h)	23.9 (h)	
	$T=55$ °C dark		6.4×10^{-2} (/h)	10.8 (h)	
	$T=25$ °C dark	Gelatin	3.2×10^{-3} (/h)	216.6 (h)	
	$T=40$ °C dark		1.8×10^{-2} (/h)	38.5 (h)	
	$T=55$ °C dark		3.4×10^{-2} (/h)	20.4 (h)	
Yellow tamarillo, Banana, and mango peel powder	90% RH $T=18$ °C	EX:MDX 20DE:	0.05	13.9 (days)	García et al. [144]
	75% RH $T=18$ °C	MP:BP (1:1:0.5:0.1)	0.044	15.8 (days)	
	50% RH $T=18$ °C	DT = 130 °C	0.029	23.9 (days)	
	90% RH $T=18$ °C	EX:MDX 20DE:	0.036	19.3 (days)	
	75% RH $T=18$ °C	MP:BP (1:1:1:0.1)	0.036	19.3 (days)	
	50% RH $T=18$ °C	DT = 130 °C	0.032	21.7 (days)	

Table 2 (continued)

Product	Storage condition	Wall material	K (constant rate) (/days)	$t_{1/2}$ (half-life)	Reference
Paprika oleoresin (Red fraction)	$a_w = 0.108$ $T = 35$ °C	GA	33.09×10^{-3}	20.95 (days)	Rascón et al. [145]
	$a_w = 0.318$ $T = 35$ °C		55.73×10^{-3}	12.44 (days)	
	$a_w = 0.515$ $T = 35$ °C		123.86×10^{-3}	5.6 (days)	
	$a_w = 0.743$ $T = 35$ °C		31.60×10^{-3}	21.94 (days)	
	$a_w = 0.108$ $T = 35$ °C	SPI	197.95×10^{-3}	3.5 (days)	
	$a_w = 0.318$ $T = 35$ °C		201.32×10^{-3}	3.44 (days)	
	$a_w = 0.515$ $T = 35$ °C		196.45×10^{-3}	3.53 (days)	
	$a_w = 0.743$ $T = 35$ °C		40.15×10^{-3}	17.26 (days)	
(Yellow fraction)	$a_w = 0.108$ $T = 35$ °C	GA	37.33×10^{-3}	18.57 (days)	
	$a_w = 0.318$ $T = 35$ °C		55.41×10^{-3}	12.51 (days)	
	$a_w = 0.515$ $T = 35$ °C		130.17×10^{-3}	5.32 (days)	
	$a_w = 0.743$ $T = 35$ °C		31.60×10^{-3}	21.94 (days)	
	$a_w = 0.108$ $T = 35$ °C	SPI	190.3×10^{-3}	3.64 (days)	
	$a_w = 0.318$ $T = 35$ °C		209.75×10^{-3}	3.3 (days)	
	$a_w = 0.515$ $T = 35$ °C		217.81×10^{-3}	3.18 (days)	
	$a_w = 0.743$ $T = 35$ °C		38.55×10^{-3}	17.98 (days)	
Carrot (*Caucus carota*)	$T = 4$ °C	MDX	0.0045 (/week)	154.03 (weeks)	Shaaruddin et al. [146]
	$T = 25$ °C		0.0051 (/week)	135.91 (weeks)	
	$T = 40$ °C		0.0057 (/week)	121.6 (weeks)	
	$T = 25$ °C light		0.0205 (/week)	34.66 (weeks)	
	$T = 4$ °C	Resistant MDX	0.0049 (/week)	141.46 (weeks)	
	$T = 25$ °C		0.0085 (/week)	81.55 (weeks)	
	$T = 40$ °C		0.0112 (/week)	61.89 (weeks)	
	$T = 25$ °C light		0.0187 (/week)	32.74 (weeks)	
	$T = 4$ °C	Capsul	0.0022 (/week)	315.07 (weeks)	
	$T = 25$ °C		0.0036 (/week)	192.54 (weeks)	
	$T = 40$ °C		0.0044 (/week)	157.53 (weeks)	
	$T = 25$ °C light		0.011 (/week)	63.01 (weeks)	
	$T = 4$ °C	Hi-cap	0.0016 (/week)	433.22 (weeks)	
	$T = 25$ °C		0.0018 (/week)	385.08 (weeks)	
	$T = 40$ °C		0.0029 (/week)	239.02 (weeks)	
	$T = 25$ °C light		0.0098 (/week)	70.73 (weeks)	
Cagaita (*Eugenia dysenterica* DC.)	$T = 25$ °C 32.8% RH	GA (10%, DT = 120)	0.0029	238.6 (days)	Daza et al. [147]
		GA (20%, DT = 120)	0.00313	221.4 (days)	
		GA (30%, DT = 120)	0.00253	274.3 (days)	
		GA (10%, DT = 140)	0.00311	222.8 (days)	
		GA (20%, DT = 140)	0.00303	228.9 (days)	
		GA (30%, DT = 140)	0.00257	270 (days)	
		GA (10%, DT = 160)	0.00363	190.9 (days)	
		GA (20%, DT = 160)	0.00259	267.4 (days)	
		GA (30%, DT = 160)	0.00282	246.1 (days)	

Table 2 (continued)

Product	Storage condition	Wall material	K (constant rate) (/days)	$t_{1/2}$ (half-life)	Reference
		Inulin (20%, DT = 120)	0.00593	116.9 (days)	
		Inulin (30%, DT = 120)	0.00371	186.6 (days)	
		Inulin (20%, DT = 140)	0.00679	102.1 (days)	
		Inulin (30%, DT = 140)	0.0053	130.7 (days)	
		Inulin (20%, DT = 160)	0.00497	139.3 (days)	
		Inulin (30%, DT = 160)	0.00423	163.5 (days)	
Annatto seeds (*Bixa orellana* L.)	$T = 40$ °C dark	GA	23.33×10^{-3}	29.71 (days)	Tupuna-Yerovi et al. [148]
	$T = 40$ °C light		74.64×10^{-3}	9.27 (days)	
Annatto seeds (*Bixa orellana* L.)	$T = 60$ °C	GA	1.30×10^{-4} (/min)	88.82 (h)	Tupuna et al. [64]
	$T = 90$ °C		8.10×10^{-4} (/min)	14.26 (h)	
	$T = 98$ °C		13.78×10^{-4} (/min)	8.39 (h)	
Spinach (*Spinacea oleracea*) β-carotene	$T = 4$ °C, vacuum	None	2.2×10^{-1}	31.62 (days)	Syamila et al. [149]
	$T = 20$ °C, vacuum		2.1×10^{-2}	31.99 (days)	
	$T = 40$ °C, vacuum		2.3×10^{-2}	30.62 (days)	
	$T = 4$ °C		2.2×10^{-2}	32.08 (days)	
	$T = 20$ °C		2.1×10^{-2}	31.99 (days)	
	$T = 40$ °C		2.1×10^{-2}	33.68 (days)	
	$T = 20$ °C, vacuum, light		2.5×10^{-2}	28.84 (days)	
Spinach Lutein	$T = 4$ °C, vacuum		1.2×10^{-2}	56.96 (days)	
	$T = 20$ °C, vacuum		1.2×10^{-2}	56.08 (days)	
	$T = 40$ °C, vacuum		1.4×10^{-2}	49.98 (days)	
	$T = 4$ °C		1.2×10^{-2}	58.25 (days)	
	$T = 20$ °C		1.2×10^{-2}	56.08 (days)	
	$T = 40$ °C		1.5×10^{-2}	45.63 (days)	
	$T = 20$ °C, vacuum, light		1.1×10^{-2}	58.94 (days)	
Spinach α-tocopherol	$T = 4$ °C, vacuum		0.8×10^{-2}	84.43 (days)	
	$T = 20$ °C, vacuum		1.0×10^{-2}	73.44 (days)	
	$T = 40$ °C, vacuum		1.6×10^{-2}	42.45 (days)	
	$T = 4$ °C		0.8×10^{-2}	85.37 (days)	
	$T = 20$ °C		1.0×10^{-2}	70.94 (days)	
	$T = 40$ °C		1.6×10^{-2}	42.34 (days)	
	$T = 20$ °C, vacuum, light		1.1×10^{-2}	63.19 (days)	

Table 2 (continued)

Product	Storage condition	Wall material	*K* (constant rate) (/days)	$t_{1/2}$ (half-life)	Reference
Spinach Carotenoids	*T* = 4 °C	None	0.587 (/month)	1.18 (months)	Çalışkan and Dirim [150]
	T = 20 °C		0.597 (/month)	1.16 (months)	
	T = 30 °C		0.626 (/month)	1.11 (months)	
Chlorophylls	*T* = 4 °C		0.433 (/month)	1.6 (months)	
	T = 20 °C		0.479 (/month)	1.45 (months)	
	T = 30 °C		0.549 (/month)	1.26 (months)	

EX extract, *DT* drying temperature, *MDX* maltodextrin, *GA* gum arabic, *SPI* soy protein isolate, *MP* mango poder, *BP* banana poder, *XG* xanthan gum, *CM* cladode mucilage

[a]Superficial

[b]Internal

than the double [146]. The foregoing may be related to the compatibility of the active compound with the encapsulating agent and a formation of more stable molecules [32].

Effect of Relative Humidity The effect of relative humidity (RH) has been evaluated by different researchers. Rascón et al. [145] evaluated the RH effect in paprika pigments microencapsulated with GA and Otálora et al. [50] in betalains of cactus fruit (*Opuntia ficus-indica*) microencapsulated with MDX and a mixture of MDX-cladode mucilage. Pascual-Pineda et al. [137] evaluated the RH effect in red onion (*Allium cepa*) anthocyanins microencapsulated with MDX, among others. In general, all studies conclude that the best storage conditions are below 50% RH, since higher RH values cause a collapse in the capsule structure and degradation of the encapsulated pigments [154]. In addition, it has been observed that above 70% RH, there is water absorption and changes in the physical state of the powder [137]. However, the authors also mention that this change of state generated a dough-like mass that acted as a protective barrier against oxygen, keeping the pigments for a longer time, but the change of state makes it difficult to add them to materials when used as coloring in foods. Specifically, Pascual-Pineda et al. [145] describe how the different stages of water affect the stability of microencapsulates: i) in the first stage (expanded phase, low water activities), the water molecules on the surface are compact and close to each other, which increases the vapor pressure on the surface and the pigments can be more stable; (ii) in the next stage (expanded-condensed phase transition), the water adsorbed on the surface becomes small drops, which correspond to the formation of water in the monolayer (according to the GAB equation) (here it could be observe intermediate stability due to chemical reactions); (iii) finally, at water activities higher than 0.5 (and temperatures higher than *Tg*), the wall of the microcapsules goes to the rubbery state, which decreases the viscosity and increases the molecular mobility, also causing a reduction in stability, generating dissolution, collapse, or caking.

Influence by Exposure to Light It has been observed that blueberry (*Vaccinium ashei* var. Rabbiteye) anthocyanins encapsulated with mesquite gum [128], extracts of black carrot (*Daucus carota*) encapsulated with different agents [133], orange carrot (*Daucus carota*) carotenoids encapsulated with different agents [146], annatto seeds (*Bixa orellana*) encapsulated with GA [148], and spinach (*Spinacea oleracea*) juice chlorophylls encapsulated [149] degraded faster by exposure to light during storage.

Microencapsulated Pigments Color Values

A graphic representation of the colors observed from encapsulated natural pigments is shown in Fig. 9. Although a great variety of shades and color intensities have been observed in the microencapsulation of pigments, most are red, yellow, and some green tones; however, green and blue colors are less common.

A lack of studies or reports of some natural pigments has been observed, which could be related to the stability of the pigment. For example, chlorophylls at moderate and high temperatures undergo a structural change; the loss of the Mg^{2+} ion from the porphyrin ring generates pheophytins, which have brown colors of different tones [155]. Some anthocyanins present blue tonalities due to pH, at neutral-alkaline values this coloration can be observed [156, 156]; However, under these conditions, they can be unstable, and the color could fade, especially if the temperature increases [157, 158], changes that may be due to physicochemical changes in the environment. An option for obtaining blue pigments is the use of indigo (*Indigofera tinctoria*) leaves; the fermented extract of the leaves of the plant generates a blue tone and the molecule is stable at alkaline pH [159]. A recent study showed that it is possible to obtain a blue pigment from betalamic acid from beets (*Beta vulgaris*), which also does not show toxicity to humans and maintains its blue color in aqueous solutions [160].

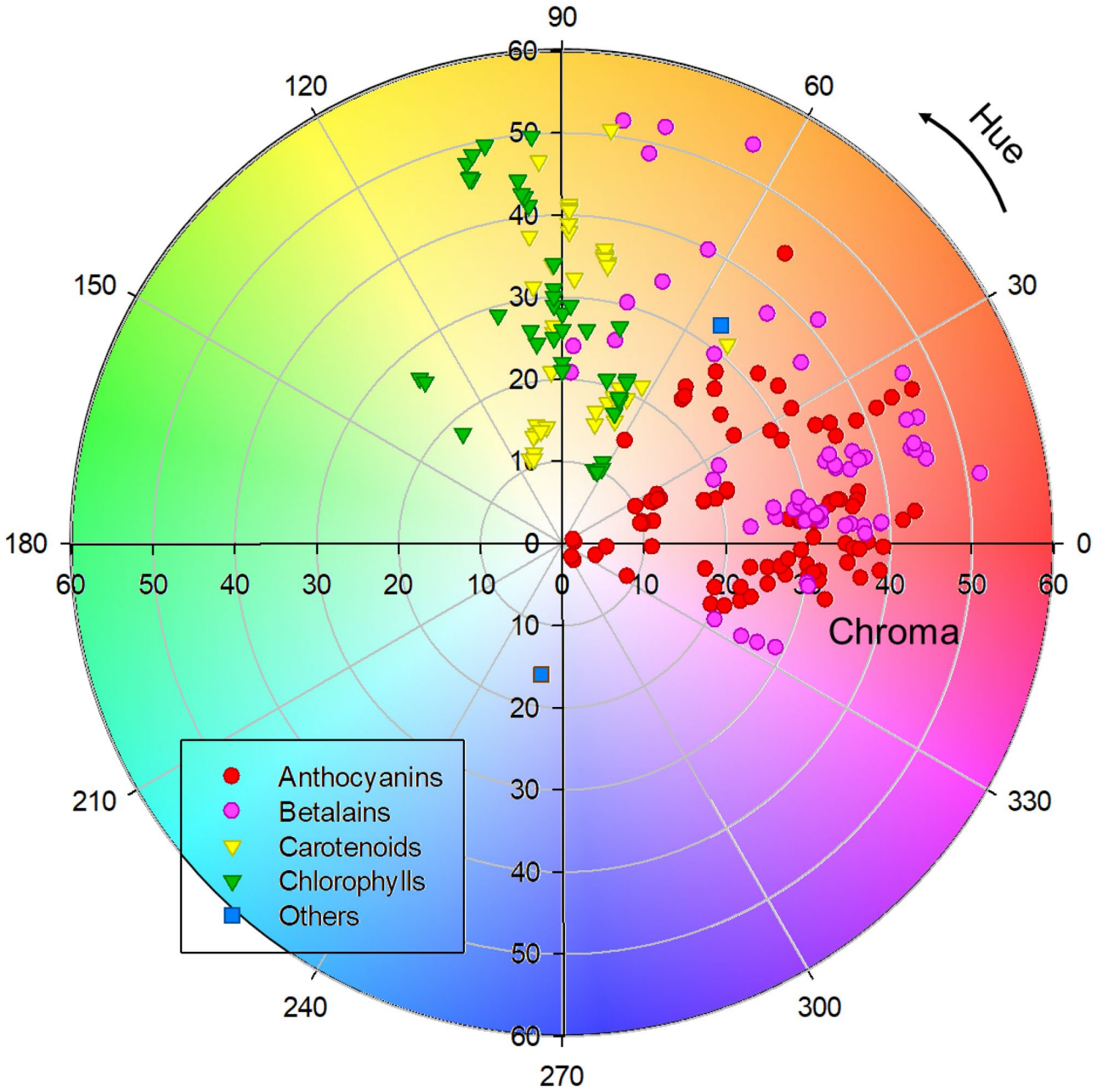


Fig. 9 Color values reported as chroma vs. hue plot (The background color does not correspond exactly to color.)

Experimental Design to Increase Encapsulated Pigments

Experimental Design

Some authors have focused on evaluating the effect that different variables have on one or more responses (physical, physicochemical, morphological, etc.) on spray-dried product. They have shown the use of mathematical methods and statistical inferences as a tool to explore the set of variables involved in spray drying. Some of the most studied variables are the process conditions during drying: feeding flow, pressure, inlet or outlet temperature, air velocity in cyclone, type of atomizer, the feeding mixture, the amount of solids, and the type and mixtures of encapsulating agent, among others.

Thanks to the use of experimental design, the behavior of the system can be known and the number of experiments reduced. For this, it is necessary to design a series of experiments that adequately describe the response of interest, that is, use mathematical models to fit the data and find the factors that

maximize or minimize the response [161]. If all the variables can be measured, the following expression is obtained (Eq. 5):

$$Y = f(x_1, x_2, x_3, \cdots x_k)k = 1,2,3,\cdots n \quad (5)$$

where Y is the response of the system that depends on n that is the number of independent variables x_k. Usually, the different models follow a behavior that can be described with a second order polynomial (Eq. 6) [162].

$$Y = \beta_0 + \sum \beta_j X_j + \sum \beta_{jj} X_j^2 + \sum \beta_{ij} X_i X_j \quad (6)$$

where Y is the response described by the coefficients β_o, β_j, $\beta_{jj,}$ and β_{ij}, which correspond to the intercept, linear coefficient, quadratic coefficient, and the coefficient of the interaction, respectively. X_i and X_j are the coded independent variables or factors [162]. This coding can be carried out using the following equation (Eq. 7).

$$Xi = \frac{x_i - x_0}{\Delta x} \quad (7)$$

where X_i is the coded dimensionless value of the factor or variable x_i; x_0 is the center point of x_i and Δx is the increment between the values.

Within the experimental designs, it can find screening designs (i.e., full factorial, fractional factorial, or Plackett-Burman) to select the factors or variables that have the greatest effect on the response (for further optimization) or those with the response surface (central composite, Box-Behnken, mixture simplex-centroid or mixture simplex-lattice, and Taguchi design), since applying the response surface methodology can optimize the process/formulation [163]. Some experimental designs will be described below.

Full Factorial Design

It is a two-level design denoted as 2 k, where k is the number of factors studied, usually coded as + 1 at the highest level and −1 at the lowest [163]. Design 2^3 has been used to evaluate the effect of the inlet temperature (T_{in}) (150 and 180 °C), the feeding rate (1.5 and 2 L/h), and the percentage of encapsulating agent (60 and 90%) on characteristics such as moisture content, yield, and color of microencapsulated pitaya (*Stenocereus griseus*) betalains. The authors reported that individual factors (T_{in}, feeding and concentration) affected moisture content and color. However, the performance was affected, mainly by the amount of encapsulating agent and the feeding rate under the following two optimal conditions: (a) 150 °C, 1.5 L/h and 90% of encapsulates and (b) 150 °C, 2.0 L/h and 90% of encapsulates [51].

Fractional Factorial Design

This type of model evaluates the effect of certain factors with a lower number of runs, unlike the full factorial design [163]. To obtain the highest amount of anthocyanins and the best physical properties, Ortiz-Basurto et al. [164] used a 3^{3-1} fractional factorial design to encapsulate pitanga (*Eugenia uniflora* L.), varying the type of encapsulating material (high degree polymerization agave fructans, high-performance agave fructans, MDX), the outlet temperature (110, 120, 140 °C), and the proportion between pigment and encapsulating agent (1:4, 1:5, 1:6). Of the nine experiments carried out, the type of encapsulating material had no effect on the physicochemical and fluidity properties of the encapsulates, but the retention of anthocyanins and the antioxidant properties depended on the proportion of pigment and encapsulating agent, with the ratio 1:6 being the greater amount of retention and protection offered.

Plackett-Burman Design

It is a two-level fractional factorial design to examine $N - 1$ factors where N is a multiple of 4. Even though it does not consider the interactions of two factors, very few experiments are necessary (107). This design considers insignificant dummy variables, which are used to estimate the experimental error and write a first order model (65). This design was used to find which variables were the most significant in the encapsulation of lycopene using dehumidified air: the effect of the content of dextrose equivalents (DE) of MDX (6 and 21), the active compound-encapsulating agent ratio (1:2, 1:6), the feeding temperature (30 and 60 °C), the inlet temperature (120 and 150 °C), the drying air flow (17.5 and 22.75 m^3/h), and the compressed air flow (500 and 800 L/h). Moisture content, density, rehydration capacity, lycopene isomerization, and storage stability of the microencapsulates were evaluated. The most significant parameters were the active compound-encapsulating agent ratio, the drying temperature, and the feeding temperature, which were used for a subsequent central composite design with which the optimal encapsulation conditions were found: active compound-encapsulating agent ratio, 1:3.3; feeding temperature, 52 °C; and inlet air temperature, 147 °C, obtaining an encapsulation efficiency of 93% [165].

Central Composite Design

This design combines a two-level factorial design and a star (or axial point) design with center points. A small number of experiments are necessary to evaluate a great variety of combinations, and it is also a robust model. The number of experiments is based on $k2 + 2k + \varepsilon$, where ε is the number of central replicas and k is the number of factors [163]. This design is one of the most used to explore the effect of different variables in the encapsulation process. Chong et al. [166] evaluated: (a) the effect of the inlet temperature (132–188 °C) and the concentration of maltodextrin

(6–34%) in microencapsulated amaranth (*Amaranthus gangeticus*) betacyanins and (b) the effect of the inlet temperature (140–190 °C), the concentration of β-cyclodextrin (5–19%), and the feeding flow (4–14 mL/min) for encapsulated amaranth betacyanins. The authors evaluated different characteristics related to drying (drying rate, droplet size, and drying time), noting the positive effect of the encapsulating agent and the inlet temperature on the particle size for both encapsulating agents. For the rest of the characteristics, they did not observe significant differences. Das et al. [167] used this experimental design for the optimization of the encapsulation of purple rice (*Oryza sativa*) bran anthocyanins using modified rice starch as the encapsulating agent. They varied the encapsulate concentration (5–10%), the pressure (2.76–5.52 MPa), and the inlet temperature (140–180 °C) in order to maximize the different responses (encapsulation efficiency, anthocyanin content, density, and a_w). After optimization of encapsulation efficiency, maximum anthocyanin content, density, and low a_w, the authors observed that the optimal process conditions were 6.01% starch, 4.96 MPa, and 168.78 °C. On the other hand, Santana et al. [168] studied the encapsulation of carotenoids from pequi (*Caryocar brasiliense* Camb.) using GA as encapsulating agent, varying the inlet temperature (140–200 °C), the surfactant concentration (0–5% Tween 80), and the GA concentration (10–20%). The dependent variables or responses were the physicochemical characteristics (moisture content, hygroscopicity, yield, and a_w), the physical characteristics (bulk and absolute density, porosity, particle size, morphology), the content of carotenoids and vitamin C. The variables had an influence on other characteristics, different to the carotenoid content, where none of the variables was significant. However, the process was optimized to obtain the highest content of carotenoids and vitamin C, the minimum water content, and the lowest hygroscopicity (T_{in} 152 °C, surfactant 1%, and GA 18%). Other works have been focused on optimizing the content of microencapsulated anthocyanins by varying the inlet temperature and the feeding flow [169] or encapsulating blackberry anthocyanins by varying the inlet temperature (100–150 °C) and the pulp:solids ratio (1:0.5–1:2) [170, 171].

Box-Behnken Design

It is applied to evaluate three or more factors with three levels. $2k(k-1)+$experiments are required where k is the number of factors and ε the number of center points. The efficiency of the model depends on the number of estimated coefficients (Eq. 6) [163]. Bazaria and Kumar [76] used it to evaluate the effect of three independent variables: inlet air temperature (160, 170, and 180 °C), feeding flow rate (400, 500, and 600 mL/h), and whey protein concentrated (WPC) concentration (5, 10, and 15%) on eight different responses, including the betalains content of beet (*Beta vulgaris*) juice in the microcapsule. The authors found that the increase in temperature decreases the content of betalains; these, together with the concentration, were the significant variables.

Mixture Simplex-Centroid Design or Mixture Simplex-Lattice Design

This type of design only evaluates the variables that can be mixed. It is used to optimize the composition of a mixture based on the proportion of its components. The mixture consists of k number of components equally separated between 0 and 1 (Eq. 8).

$$xi = 0, \frac{1}{m}, \frac{2}{m}, \frac{m-1}{m}1 \quad i = 1,2,3,\cdots k \tag{8}$$

For the adjustment of a first-degree function, only levels 0 and 1 are necessary. For a second-degree model, levels 0, 1/2, and 1 are necessary, and for a cubic model, levels 0, 1/3, 2/3, and 1 are needed. This design takes into account the pure components, binary combinations (simplex-lattice), and ternary (only in simplex-centroid design); therefore, its use is laborious, since it requires a high number of experiments. In the case of the simplex centroid design, it is necessary at least the inclusion of a ternary mixture.

Since this design describes the behavior in proportion of its components, it is necessary to consider the mathematical model as the sum of each of the components, therefore taking the following expression:

Making the following considerations $x_1+x_2=1$, $b_0=1\ b_0$, $x_{11}=x_1\ (1-x_2)$, and $x_{22}=x_2\ (1-x_1)$, the following expression is obtained:

$$y = (x_1 + x_2)b_0 + b_1x_1 + b_2x_2 + b_{11}x_1 (1-x_2) + b_{22}x_2(1-x_1) + b_{12}x_1x_2 \tag{10}$$

and reordering, the following is obtained:

$$y = (b_0+b_1 + b_{11})x_1 + (b_0+b_2 + b_{22})x_2 + (b_{12}+b_{11} + b_{22})x_1x_2 \tag{11}$$

$$y = b_1'x_1 + b_2'x_2 + b_{12}'x_1x_2 + \cdots + b_i'x_i + b_{ij}'x_ix_j \tag{12}$$

Thus, it is possible to obtain linear, quadratic, complete cubic, or special cubic models depending on the number of experimental points [172].

The simplex-lattice design has been used by Santana et al. [21] in the encapsulation of anthocyanins from juçara (*Euterpe edulis*) pulp using GA, modified starch, and whey protein isolate or soy protein isolate. The authors observed that the ternary mixtures showed better performance, solubility, encapsulation efficiency and retention of anthocyanins. Mahdavee-Khazaei et al. [173] encapsulated saffron (*Crocus*

sativus) petals anthocyanins with GA and two types of MDX with different dextrose equivalents (16-20DE and 4–7DE). One of the response variables was color. The authors observed that as the amount of GA decreased, fewer color differences were observed in the powders. On the other hand, the simplex-centroid design has been used in the encapsulation of pomegranate (*Punica granatum*) anthocyanins with Capsul (trademark), MDX, and GA. The highest pigment retention was seen with the 1:1 GA: capsul binary mixture [174]. Souza et al. [175] encapsulated tomato (*Solano lycopersicum*) lycopene with MDX, whey protein isolate and modified starch. In this case, MDX and modified starch alone and the 1:1 binary mixture showed both the highest amount of lycopene and the highest antioxidant activity [175].

Taguchi Design

The Taguchi model allows the study of multiple factors with several levels with high precision. Pal and Bhattacharjee [176] used this design of experiments for the encapsulation of lutein from African marigold flowers (*Tagetes erecta* L.) to optimize the maximum amount of encapsulated lutein. The following variables were established: concentration of encapsulating agent (30, 35, 40%), mixtures of MDX:GA (50:50, 60:40, 70:30), inlet temperature (150, 170, 190 °C), and air flow speed (473, 601, 742 L/h). The authors observed that the best conditions for encapsulation were 30% of encapsulating agent, 60:40 MDX:GA, 0.90 mL/min feeding rate, 170 °C inlet temperature, 742 L/h air flow, and a pressure of 60 mbar, obtaining up to 78.32% encapsulated lutein.

Uses

Even though encapsulation by spray drying allows obtaining highly stable pigments, very few articles report the use of pigments in food systems. Some of these studies are listed in Table 3. Pigments have been used mainly for coloring model beverage systems or for simulate juices [56, 103, 65], isotonic drinks [148], fermented and unfermented milk-based drinks [177, 178], ice cream [179, 180], yogurt [142, 56, 52], bakery products [181, 182, 183, 184, 146], and chocolate, chewy caramels, chewing gums, and hard caramels [180, 185, 186].

Tatar Turan et al. [180] and Papillo et al. [155] encapsulated blueberry anthocyanins (*Vaccinium corymbosum*) with guar gum and black rice anthocyanins (*Oryza sativa* L. var. Artemide) with GA, respectively. In both researches, microencapsulated pigments were incorporated in formulations for making bread. They observed that microencapsulation protected the pigments from thermal degradation, with no apparent changes in color. Thus, results were favorable since at high temperatures polyphenol compounds, such as anthocyanins, form *O*-quinones with brown tones [181]. On the other hand, the use of carotenoids has also been evaluated in bread. In lycopene encapsulated with modified starch, the encapsulation protected pigments during bread baking [182]. The incorporation of microencapsulated anthocyanins from Asian pigeonwings (*Clitoria ternatea*) flowers prevented the growth of microorganisms, acting as a bio-preservative for baked products [183].

The addition of encapsulated pigments allows to obtain colors similar to those of commercial products. For example, the addition of betaxanthins from yellow pulp prickly pear (*Opuntia* spp.) produces a lemon-like colored yogurt [142]. The betalains from "garambullo" or bilberry cactus (*Myrtillocactus geometrizans*) produce a color similar to that of strawberry yogurt [56]. The anthocyanins of purple cabbage (*Brassica oleracea* L. ssp. Capitata f. Rubra) added to fermented milk-based drinks produced colorations similar to those of commercial strawberry yogurt (pale pink color) [177]. Some authors suggest that the color varies with the storage time [52]. However, it may be more stable when the product is stored in dark conditions [142]. On the other hand, low temperature is a fundamental factor in the stability of the pigments added to some products [142] such as ice cream.

Another use for microencapsulated pigments is in candy. Microalgae (*Nannochloropsis oculata*; *Isochrysis galbana*) have been used in the formulations for the manufacture of chewing gums [68] and chewy candies. The authors highlight that the incorporation of the microencapsulated pigments increased the green color, especially in $-a^*$ parameter, making the products more pleasant. On the other hand, orange carrot (*Daucus carota*) carotenoids have been incorporated into formulations for hard candies; the physical characteristics of the food matrix minimized contact with the environment and better maintained the added pigments [146]. Da Silva et al. [186] report that the highest degradation of açai encapsulated anthocyanins was observed during the production of chewy candies, not during storage, moreover, the color was stable for up to 6 months.

Other uses of some encapsulated natural pigments have been reported. Garrido et al. [189] suggest that encapsulated natural pigments can be consumed directly as tablets, since the powders obtained from anthocyanins from maqui or Chilean wineberry (*Aristotelia chilensis*) presented good compactness properties and a high amount of beneficial compounds to humans. The authors also pointed out that the pigments obtained have potential in cosmetic applications, which has been evaluated by Azmin et al. [191]. They made a lip balm with betalains from beets, the final product presented high stability and good sensory acceptability. On the other hand, in the incorporation of blackberry (*Rubus fruticosus*) pigments in edible films, with high antioxidant capacity, a controlled release and degradation of the film was observed, which has made possible its application in foods packaging [188].

Table 3 Current application of microencapsulated pigments

Material	Applications	Reference
Blueberry (*Vaccinium corymbosum* L.)	Ice cream and cake	Tatar Turan et al. [180]
Clitoria ternatea flowers	Muffins	Ab Rashid et al. [181]
Açai (*Euterpe oleracea* Mart.)	Chocolate	Augusto et al. [185]
Jabuticaba residue (peel and seeds)	Sausages	Baldin et al. [187]
Açai (*Euterpe oleracea* Mart.)	Chewy candies	da Silva et al. [186]
Blackberry (*Rubus fruticosus*)	Edible films (direct and sparkling addition)	Ferreira Nogueira et al. [188]
Juçara pulp (*Euterpe edulis* M.)	Fermented and unfermented dairy beverage	Lima et al. [178]
Purple cabbage (*Brassica oleracea* L. ssp. *Capitata* f. *Rubra*) leaves	Fermented milk drinks	Espinosa Alvarez et al. [177]
Maqui berry (*Aristotelia chilensis*)	Compact tablets	Garrido Makinistian et al. [189]
Black rice bran (*Oryza sativa* L)	Biscuits	Papillo et al. [182]
Juçara (*Euterpe edulis* Marius)	Gelatins	Bernardes et al. [190]
Pitaya (*Stenocereus prunosus*)	Yogurt	Vargas-Campos et al. 52
Gomphera globosa L. flowers	Colored cookies	Roriz et al. [184]
Beetroot (*Beta vulgaris* L.)	Model juice	Kaimainen et al. [103]
Beetroot (*Beta vulgaris* L.)	Beverage, yogurt	Hernández-Martínez et al. [56]
Cactus pear (*Opuntia ficus-indica*) (yellow pulp)	Yogurt and softdrink	Fernández-López et al. [142]
Beetroot (*Beta vulgaris* L.)	Lip balm	Azmin et al. [191]
Mandarin (*Citrus unshiu*)	Mandarin beverage	Lee et al. [65]
Nannochloropsis oculata and *Isochrysis galbana*	Chewy gum	Palabiyik et al. [68]
Nannochloropsis oculata, *Diacronema vlkianum*, and *Porphyridium cruentum*	Ice cream	Durmaz et al. [179]
Carrot (*Daucus carota*)	Hard candy	Shaaruddin et al. [146]
Annatto seeds	Synthetic tangerine flavor isotonic drink	Tupuna-Yerovi et al. [148]
Commercial lycopene	Cake	Rocha et al. [183]

Final Remarks

Spray drying has proven to be efficient in obtaining microencapsulated compounds from natural sources that contain different types of pigments: carotenoids, anthocyanins, betalains, and chlorophylls. Thanks to microencapsulation, powder pigments are obtained that can be easily incorporated into food systems, maintaining their stability and physical properties for a longer time. However, it is essential to study the effect of processing on the incorporated pigments, as well as their addition in different food matrices. Even though different polymers have been tried for pigment encapsulation, maltodextrin has been and perhaps will continue to be the most widely used encapsulated agent. Future works should be focused on the exploitation of other sources of polymers for encapsulation and, at the same time, take advantage of agro-industrial waste to obtain natural pigments, as well as expanding the range of colors that they could display.

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Declarations

Conflict of Interest The authors declare no competing interests.

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