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Recent developments in extraction and encapsulation techniques of orange essential oil

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ABSTRACT

Orange production is constantly growing. The main orange by-product, the orange peel, is a usable source of essential oils with a lot of health benefits. Because of that, it is important to find the best recovery and encapsulation techniques in order to get the best bioavailability for human and to ensure the highest quality for food applications. Thus, the aim of this work is to summarize the complete process needed to obtain orange essential oil, from the pre-treatments to the encapsulation steps, carried out in the last years. This review is focused on the comparison of new and more innovative techniques in front of the most conventional ones used for extracting and encapsulating the orange essential oil.

1. Introduction

Recovery of bioactive compounds coupling to emerging technologies is a hot topic in agro-food research. In fact several authors have noticed as the use of emerging technologies allows a better recovery of the bioactive compounds and include environmental friendly processes (Barba, Galanakis, Esteve, Frigola, & Vorobiev, 2015; Roselló-Soto et al., 2015).

Production of orange in the world accounted for 53.84 million tons, from which 6 million tons are produced in the European Union in 2019, almost 10% of the world production. Orange is a rich source of soluble sugars, phenolic compounds, flavonoids, dietary fibres (cellulose, hemicelluloses, pectin), vitamins and essential oils (El Kantar et al., 2018; Putnik et al., 2018). The orange essential oil (OEO) has been reported to have antioxidant, anti-cancer, anti-inflammatory, cardioprotective, neuroprotective, anti-bacterial and anti-mycotic activities (Magalhães et al., 2020). Nowadays consumers are taking care of what they eat attempting to protect themselves and their immune system (Galanakis, 2020). Because of that, the use of these orange essential oils as nutraceutical is interesting due to all these bioactivities they have (Galanakis, Aldawoud, Rizou, Rowan, & Ibrahim, 2020).

In general, orange essential oils are mainly localized in the peel and in a minor amount, in the leaves. Since recent times and more even in the new era of pandemic we are living, the sustainability of the food systems is a must. So all the researches are focused on the use of these unevaluated by-product derivatives to produce valuable ingredients for the food industry (Galanakis, 2020).

Briefly, OEO is composed by hundreds of ingredients. Volatile components account for 85–99%, mainly consisting on terpenoids (monoterpenes) and its derivates (oxygenated monoterpenes and sesquiterpenes). The main compound is limonene and account more than 90% of the total essential oil (Wang et al., 2019). More extended, the composition of OEO from *Citrus aurantium* and *Citrus sinensis* attending to the extraction method, the orange variety and the regional and seasonal changes are collected in Tables 1 and 2, respectively. Finding the best technique for recovery and encapsulate these valuable essential oils is a current challenge. The encapsulation procedure is the most important step attending to the stability and bioavailability of these compounds, in order to ensure the reaching to the target systems or tissues in the human organism for displaying the bioactivity (Froiio et al., 2019). The bioactive encapsulated compounds also have several potential applications in food (Vanamala, Reddivari, Yoo, Pike, & Patil, 2006) and cosmetics (Galanakis, Tsatalas, & Galanakis, 2018). The encapsulation of the OEO has been also carried out because it is an excellent strategy to overcome the problem of degrading its volatile compounds and the problems related to its use of applying in food

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Table 1
Composition of the *Citrus aurantium* essential oil.

References	(Gülay Kirbaşlar, Tavman, Dülger, & Türker, 2009)	(Azhdarzadeh & Hojjati, 2016)	(Dugo et al., 2011)	(Rowshan & Najafian, 2015)	(Jabri Karoui & Marzouk, 2013)	(Sanei-Dehkordi, Sedaghat, Vatandoost, & Abai, 2016)	(Ali et al., 2012).	(Sarrou, Chatzopoulou, Dimassi-Theriou, & Therios, 2013)	(Bourgou, Rahali, Ourghemmi, & Saïdani Tounsi, 2012)
Variety									
Recollection place	Turkey	Iran			Tunisia	Iran	Pakistan	Greece	Tunisia
Recollection date	November 2006	December 2014				October 2012	January	October	2009
Extraction procedure	Hand pressing	Hydrodistillation	Cold-pressed	Hydrodistillation	Hydrodistillation	Hydrodistillation	Hydrodistillation	Hydrodistillation	Hydrodistillation
Drying procedure	Fresh				Fresh	Fresh	Fresh	Fresh	
Monoterpenes		91.76							
Limonene	94.10	81.6	96.52	93.47	90.25	94.81	91.98	94.67	90.95
β-myrcene	1.80	5.74	1.56	1.78	0.04	1.00	2.57	2	0.07
β-ocimene		0.52			0.36	0.19	0.2	0.3	0.02
α-pinene		1.96	0.34	0.43	0.55	0.3	0.60	0.53	0.36
β-pinene	0.5	0.56	0.10	0.10	0.44	0.65	0.21	0.62	0.38
Sabinene		0.46			0.48		0.19	0.18	0.20
Terpinolene		0.05			0.28	0.44			0.02
α-terpinene		0.02			1.1				1.66
α-thujene		0.01							0.44
ϒ-terpinene		0.05				0.01			0.31
Sesquiterpenes		0.6							
α-caryophyllene		0.02							0.13
β-caryophyllene	0.10	0.22	0.04		0.05	0.03			
ϒ-elemene					0.05				
β-farnese					0.15				
Germacarene		0.22	0.03			0.08	0.06		0.04
Aldehydes									
Citral		0.45					0.13		
Critronellal		0.02							
Decanal	0.20		0.09	0.20			0.08	0.16	
Geranial	0.1								
Neral					0.13				
Nonanal		0.07				0.02			0.18
Octanal		0.83	0.07	0.68			0.14	0.24	
Alcohols									
Geraniol					0.16		0.28		
Linalool	0.40	2.19	0.06	1.25	1.56		1.78	0.76	0.10
linalool oxide		0.94			0.36	0.06			0.09
Nerol		0.23			0.08				0.12
Nerolidol					0.35	0.06	0.05		
Octanol		0.13				0.13			0.02
α-terpineol		1.03			0.56			0.13	0.35
Terpinene-4-ol		0.15			0.06		0.09		0.01
Esters									
Geranyl acetate	0.08	0.33	0.08	0.13	0.11	0.13	0.08		0.02
Linalyl acetate	1.20		0.08	0.22	0.29	0.32		0.18	0.01
Neryl acetate		0.12			0.23	0.03	0.04	0.10	

Table 2
Composition of the *Citrus sinensis* essential oil.

References	(Singh et al., 2010)	(Velázquez-Nuñez, Avila-Sosa, Palou, & López-Malo, 2013)	(Bourgou et al., 2012)	(Bustamante et al., 2016)		(Jablaoui et al., 2019)		(Kamal, Anwar, Hussain, Sarri, & Ashraf, 2011)			(Hosni et al., 2010)			
Variety		*Valencia*	*Maltaise*	*Navel Navelate*				*Valencia*			*Thomson Navel*	*Valencia Late*	*Meski*	*Maltaise blanc*
Recollection place	India	Mexico	Tunisia	Spain		Burkina Faso		Pakistan			Tunisia			
Recollection date	July 2008		2009			December 2010		2010						
Extraction procedure	Hydrodistillation	Hydrodistillation	Hydrodistillation	Hydrodistillation	Microwave assisted hydrodistillation	Hydrodistillation	Instant controller pressure drop	Hydrodistillation			Hydrodistillation			
Drying procedure	Fresh	Fresh		Fresh		Oven dried		Fresh	Air dried	Oven dried	Air dried			
Monoterpenes				98.56	99.34									
Limonene	90.66	96.62	85.35	96.75	97.38	91.94	92.33	95.80	69.9	64.1	96.60	96.30	97.30	96.00
β-myrcene	1.71	1.72	0.71	0.74	0.79	2.44	2.53	1.80	3.27	4.05	0.05	0.03		0.03
β-ocimene	0.21		0.03							0.28	0.03	0.03		0.03
α-pinene	0.36	0.47	0.7	0.32	0.39	0.95	0.66	0.50	1.27	1.62	0.22	0.21	0.30	0.26
β-pinene	0.46	0.53	0.97	0.05	0.06				0.40		1.48	1.45	1.50	1.82
Sabinene	0.37		0.36	0.49	0.50					0.49	0.17	0.16	0.10	0.29
Sabinene hydrate	0.42		0.14			0.37	0.30							0.02
Terpinolene	0.23			0.20	0.18						0.07	0.07		
α-terpinene	0.02		0.93											
ϒ-terpinene			0.43		0.04					0.23	0.04	0.04		0.02
Sesquiterpenes											0.19	0.18		0.15
δ-cadinene						0.30	0.20			0.88				
α-caryophyllene			0.34								0.02	0.04		0.02
β-caryophyllene						0.14	0.09		1.39					
α-copaene						0.19	0.16		0.89		0.04	0.05		0.05
β-cubene						0.07	0.06		0.84	0.37				
β-elemene						0.02	0.02				0.05	0.06		0.02
α-farnesene									0.48	0.36				
Germacarene									1.07	0.30	0.03	0.04		0.02
Aldehydes														
Critronellal									0.78	0.45				
Decanal	0.02							0.10	2.33	7.71				
Dodecanal									0.42	0.54				
Geranial								0.10						
Neral								0.10						
Octanal	0.43							0.30		1.09				
Alcohols														
Geraniol														
Linalool			0.13	0.05	0.05	2.62	2.87	0.40	1.10	2.00	0.04	0.05	0.20	0.22
linalool oxide			0.03						0.60					
α-terpineol			0.52	0.01	0.01	0.18	0.28	0.10		1.10				
Terpinene-4-ol			0.26	0.01	0.01	0.08	0.09				0.01	0.02		0.02
Esters														
Bornyl acetate			0.42								0.02	0.03		0.02
Geranyl acetate			0.10											0.01
Linalyl acetate	2.80		0.10			0.03	0.05				0.01	0.01		0.01
Neryl acetate											0.02	0.03		0.02

matrixes for its conservation (Silva, Oliveira, Caetano, Vinhas, & Cardoso, 2018; Souza Pedrosa et al., 2019). The food applications of the encapsulated OEO are mainly for its antimicrobial activity, so it has been studied by several authors. It has been applied in apple and orange juices in order to inactive the autochthonous spoilage (Souza Pedrosa et al., 2019). The inclusion of the encapsulated OEO in films for using them in food packaging has been carried out. In fact, it has been included into corn starch films reducing the tensile strength and elongation of the film, and increasing the moisture content, permeability and morphological heterogeneity (do Evangelho et al., 2019). OEO has been also added to poly-vinyl-chloride films (30%) providing a protective effect of the polymer after sterilization by gamma radiation (Silva et al., 2018). A film made of chitosan coating combined with encapsulated OEO has been used for extending the shelf life of deep water pink shrimp to 15 days in front of the 7 days that gives only chitosan films (Alparslan & Baygar, 2017). OEO encapsulated added to sorbitol egg white protein powder based edible films have been used to keep the physico-chemical and antibacterial properties of kashar cheese, enhancing the antimicrobial effect and the appearance of the film, making it brighter and more transparent (Kavas & Kavas, 2016). Films made of poly (butylene adipate-*co*-terephthalate) with encapsulated OEO have been developed in order to reduce the microbial growth rate of *E. coli* during the storage as an active packaging. The OEO did not alter the thermal stability neither the melting temperature. Despite the film presented pores and it decreases the mechanical properties, the obtained values were still above the threshold for usage packaging (de Andrade et al., 2020). OEO has been successfully used in cakes delaying the fungal spoilage from 30 to 150 days (Kringel, da Silva, et al., 2020). For this activity, it has been also used to produce zein films to protect margarines from heat resistant moulds (Gucbilmez, Oksuz, & Arici, 2019). Encapsulated OEO has been studied for applying to potato slices to protect them from natural fungi spoilage with an inhibition higher than 70% mainly attributed to its limonene content (Shi, Huang, He, Wu, & Yang, 2018).

The present review focuses in all the procedures to have into account when trying to obtain essential oils from *Citrus sinensis* and *Citrus aurantium* agro-food residues as peels and leaves. Firstly, the main pre-treatments carried out in order to extract OEO from size reduction to dehydration process are presented. Furthermore, a review of the extraction methods, traditional and newer ones, used for obtaining the essential oils is summarized. Finally, the recent literature data related to encapsulation of OEO, sorting out the process between physical, physical–chemical and chemical methods is reviewed and an outlook to future research is reported. To sum up, this review pretends to give an exhaustive view of all the steps, including innovative technologies, that are necessary to produce encapsulated orange essential oils taking into account the literature of the last decade.

2. Pre-treatments of orange by-products

OEO is mainly contained in the fruit peel cells. So, in order to extract these valuable essential oils, the pre-treatments applicated to the orange peel are an important step in which focus on. The two principal most used pre-treatments are size reduction and dehydration. The main purpose for these pre-treatments are to keep the peels from the microbial contamination and to make them more accessible for the posterior extraction (Chua, Chong, Chua, & Figiel, 2019).

2.1. Size reduction

On the one hand, it is well known that when the particle size has been reduced, it is improved the extractive capacity because the solid–liquid contact of orange peel and solvent is increased, leading to less diffusion resistance. There are different levels of size reduction depending on the product to extract; for example, as studied by Jablaoui et al. (2019) orange peel in pieces of 5 mm × 2 mm × 5 mm had a starting accessibility ratio of 1%, really small in comparison with orange peel grinded with a granulometry repartition of 36% between 600 and 800 μm, 20% between 400 and 600 μm and 40% lower than 280 μm, that had a starting accessibility ratio of 50% and higher essential oils yields (Jablaoui et al., 2019). Briefly, as lower particle size has been obtained, better solvent accessibility for the following extraction was reached. On the other hand, Ayala et al. (2017) optimized the grinding time for obtaining the maximum yield with hydrodistillation as extraction method. The optimal conditions were obtained at 2 min of grinding time (Ayala et al., 2017). The peel size has been also optimized to obtain the highest yield of OEO with microwave assisted hydrodistillation (Franco-Vega, Ramírez-Corona, Palou, & López-Malo, 2016). A size of 3.5 cm of particle is enough for obtaining high yields of extraction of essential oil, meanwhile with lower or higher particle size it demonstrated difficulties for losing water and with higher particle size it had higher transference resistances (Qadariyah, Amelia, Admiralia, Bhuana, & Mahfud, 2017). With supercritical fluid extraction with CO_2 of the OEO, the maximum particle size for a rapid extraction is reported to be 2 mm. At 0.3 mm is has been reported to extract $\geq$ 75% of the total content of essential oil (del Valle, Calderón, & Núñez, 2019). So, depending on the extraction method used, the size needed for achieving the higher yields of extraction is quite different. However, it is an important step to have into account.

2.2. Dehydration

Dehydration has been demonstrated to be an essential step in order to obtain extracts rich in bioactive compounds or essential oils so as to reduce water levels minimizing the microbial and enzymatic degradation of the orange. Orange peel is characterized for having high humidity (greater than50%) and high sugar content, being a perfect growth substrate for microorganisms. Thus, drying is necessary to ensure the stability, and for providing long storage life (Chua et al., 2019); however, the drying method is responsible of the loss of bioactive compounds as the essential oils. Over the last few decades, some methods have been studied to be applied in orange peel (Farahmandfar, Tirgarian, Dehghan, & Nemati, 2020b).

The most known conventional method is sun drying. This technique uses the solar energy in assistance of the wind at low temperatures. In spite of being a low-cost technique, it needs large times and spaces, it depends on the weather, it has a slow drying rate and it does not give an uniform drying (Chua et al., 2019). Similar to this, another conventional technique is the shade drying with the difference of not letting the samples exposed to sunlight (Chua et al., 2019). These two methods are able to retain heat-sensitive compounds in orange peels but they increase the probability of metabolic processes after treatment. Because of that they are not commonly used nowadays.

Another drying technique is oven drying or hot air drying, that is the most used method in orange peels due to its easiness to be carried out and its low-cost. It implies the use of a continuous flow of hot air at constant temperature for removing the moisture. Llavata et al. (2020) studied the temperature range from 30 to 90 °C on citrus peel; obviously, the higher temperatures required smaller times (2 h), meanwhile lower temperatures required large times (8 h). The results reported that from 80 °C case-hardening effect was observed. The highest losses of pectin, essential oils, antioxidant capacity and polyphenols were at the highest temperatures and at the highest drying times with the lowest temperatures. The best results with the less destruction of interesting components were seen between 50 and 70 °C (Llavata, García-Pérez, Simal, & Cárcel, 2020). Deng et al. (2020) studied the hot air-drying technique on sweet orange peel, specifically in the range of 50–70 °C, and they concluded that the higher amounts of essential oils, ascorbic acid and antioxidant capacity were noticed at 65 °C during 75 min (Deng et al., 2020). Kamal et al. (2011) evaluated the effect of the drying step against the yield of extraction of OEO comparing shade drying (or drying at room temperature) and oven drying. The results obtained showed that both drying technique were worth if compared with the yield obtained

in the fresh peel, but also let clearly that oven drying had higher yield (1.07%) than shade drying (0.50%) (Kamal et al., 2011). Franco-Vega et al. (2016) used the oven drying to optimize the moisture level of the orange peels in order to obtain the highest yields of essential oils; they reported that a moisture of 10% is the good enough to obtain high essential oil yields (Franco-Vega et al., 2016). The main problem of this technique is that it is slow and rate limiting because of the water transport from inside to outside the samples, followed by its evaporation. The unique option for improving the moisture diffusivity is increasing the temperature; however, it results in the degradation of heat-sensitive compounds with high nutritional value. In addition, the time could also be an inconvenient because the long exposure to oxygen can degrade the bioactive compounds (Chua et al., 2019). To avoid these inconvenients, vacuum oven drying could able to use lower temperatures in shorter times, decreasing the oxygen degradation, however it is more expensive.

Freeze drying or lyophilization is regarded as the best method for retaining the higher number of bioactive compounds. This technique is a dehydration process that occurs in vacuum conditions in which the samples are crystallized at low temperature followed by the sublimation of the water contented. Nevertheless, it is expensive, energy consuming and time consuming owning to the slow drying rates (Chua et al., 2019). Recently, in a study made on bitter orange peel, the five drying methods (sun, shade, oven 45 °C, oven 60 °C, vacuum oven 45 °C, vacuum oven 60 °C, microwave 360 W, microwave 600 W and freeze drying) are compared. Despite all the methods showed good performance results, freeze drying confirmed the highest essential oil yield (6.90%), total phenol content and antioxidant activity (Farahmandfar et al., 2020b; Farahmandfar, Tirgarian, Dehghan, & Nemati, 2020a).

3. Extraction methods of essential oils from orange by-products

Several methods could be used to extract the essential oil from orange by-products. The most used are: distillation, cold pressing, microwave assisted extraction, and supercritical fluid extraction.

3.1. Distillation

Distillation is the most traditional method used for essential oil extraction. To apply this technique, it is not necessary to dehydrate the raw material before the extraction, and it is important to be had into account that organic solvents could be not involved in the extraction process. Three main processes take place during the extraction: hydrodiffusion, hydrolysis and decomposition by heat (Oreopoulou et al., 2019). Table 3 summarize the main results obtained with this technique.

Several authors have used the hydrodistillation (distillation with water as solvent) for obtaining OEO. Bourgou et al. (2012) reached a maximum yield of 0.46% (Bourgou et al., 2012), and Strano et al. (2014) obtained a yield of 2.1% (Strano et al., 2014). Jablaoui et al. (2019) optimized the essential oil extraction from sweet orange peel via hydrodistillation (1.63% yielded) and with instant instant-controlled pressure drop technology (1.66% yielded), noticing no significant difference between them (Jablaoui et al., 2019). These results are similar to those obtained by González-Rivera et al. (2016) (1.59%) (González-Rivera et al., 2016), and a bit lower than those obtained by Bustamante et al. (2016) (1.80%) (Bustamante et al., 2016). The highest recovery yield of the OEO reported has been obtained by Liu et al. (2019) (2.14%) with the optimized conditions: water 8.4:1 (v/w), sodium chloride concentration of 5.3%, and distillation time of 3.5 h (Liu et al., 2019).

Organic solvents could be used to improve the extraction yield. Among others, hexane has been the preferred one (Geraci, Di Stefano, Di Martino, Schillaci, & Schicchi, 2017). Alternatively, bio-based solvents were successfully used by Ozturk et al. (2019) through the extraction of limonene (the main essential oil in orange peel). The best green solvent seems to be cyclopentyl methyl ether and 2-methyl-tetrahydrofuran with limonene extraction yields 50% higher than with hexane as distillation solvent (Ozturk et al., 2019).

Overall, the main limitation of the distillation technique is that at higher extraction temperatures, higher loss of volatile and thermolabile compounds were obtained. Moreover, a drawback is that it requires a significant amount of material and has a high energy consumption because of the long extraction times (Oreopoulou et al., 2019).

To shorten the extraction time and to improve the extraction of the OEO, steam hydrodistillation has been studied and applied in orange by-products (Dewi et al., 2018). This method consists of the injection of steam with higher temperatures and pressures into the sample (Manousi, Sarakatsianos, & Samanidou, 2019). According to Golmohammadi et al. (2018), the optimal extraction conditions were set at 170 °C, 8 bar during 4 min. Comparing this technique with hydrodistillation, the time of extraction was reduced eight times and higher limonene amounts were obtained (Golmohammadi et al., 2018). In addition, it has been used the vacuum fractional hydrodistillation for extracting essential oil from the orange peel, demonstrating that the separation of the compounds occurs with the leaving of the lighter terpenes, followed by the heavier terpenes and part of the oxygenated functions. However, in the OEO total content it was not found any improvement in front of steam hydrodistillation, but it is an advantage that all the fractions can be separated if only it were interesting some compounds (Perini et al., 2017).

Comparing the distillation processes took into account, the hydrodistillation let to obtain higher yields than the other methods with OEO with less susceptibility to hydrolysis, meanwhile steam hydrodistillation

Table 3
Experimental conditions and yield of orange essential oils obtained by distillation.

Sample	Variety	Procedure	Conditions	Yield (v/w)	Reference
Fruit	*C. sinensis malteise*	Hydrodistillation	100 g of sample with water, 120 min	0.46%	(Bourgou et al., 2012)
Flavedo	*C. sinensis Tarocco*		100 g of sample with water, 3 h	2.1%	(Strano et al., 2014)
Peel	*C. sinensis*		Water 15:1 (v/w), 4 h	1.63%	(Jablaoui et al., 2019)
Biomass waste	*Citrus* peel		water 6:1 (v/w), 150 min	1.59%	(González-Rivera et al., 2016)
Biomass waste	*C. sinensis Navel Navelate*		Water 10:1 (v/w), 240 min, 100 °C	1.80%	(Bustamante et al., 2016)
Flavedo	*C. sinensis Navel Gannanza*		Water 8.4:1 (v/w), sodium chloride 5.3%, 3.5 h.	2.14%	(Liu et al., 2019)
Biomass waste	Orange peel	Bio-based solvent distillation	Cyclopentyl methyl ether 10:1 (v/w), 120 min	0.81%	(Ozturk, Winterburn, & Gonzalez-Miquel, 2019)
Leaves	*C. sinensis Baby Java*	Steam hydro-distillation	2.5 L of water, 100–115 °C, 4 h.	0.63%	(Dewi, Prastyo, & Wijana, 2018)
Peel	*C. sinensis Valencia*	Steam explosion	100 g of sample, 170 °C, 8 bar, 4 min	1.12%	(Golmohammadi, Borghei, Zenouzi, Ashrafi, & Taherzadeh, 2018)
Peel	*C. sinensis*	Vacuum fractional hydrodistillation	10 kPa, 70 °C	1.13%	(Perini, Silvestre, Agostini, Toss, & Pauletti, 2017)

produce less oil with higher susceptibility to hydrolysis but a shorter period of extraction time is needed (Reyes-Jurado, Franco-Vega, Ramírez-Corona, Palou, & López-Malo, 2015).

3.2. Cold pressing extraction

Cold pressing is commonly used in the production of essential oils. It consist of a physical process in which the vegetable matrix is crushed or broken to release the oil resulting in an oil/water emulsion typically separated by centrifugation (Forde et al., 2014). The main advantage of this method is that it does not use heat during the process (Reyes-Jurado et al., 2015). It has been used by several authors for obtaining essential oil from the orange by-product (Guo et al., 2018). With this technique it is possible to obtain an OEO mainly composed by limonene, myrcene, alpha-pinene, linalool, octanal and decanal (Njoroge, Phi, & Sawamura, 2010). Huang et al. (2010) compared the extraction of the OEO by steam distillation and cold pressing techniques. Although the volatile composition extracted is similar with both techniques, the cold pressing let obtain higher yield. However, in comparison with the fresh orange peel, the essential oils extracted by these techniques have lower content of hexanal, 2-hexenal, decanal, citronella, neral, geranial, linalool, beta-caryophyllene and valencene, but higher content of perilla aldehyde, alpha-terpineol and p-mentha-1,8-dien 9-ol (Huang et al., 2010). A similar comparison was reported by Radan et al. (2018); despite more components were found in the oil obtained by cold pressing, the content of linalool and hexadecenoic acid were higher in detriment of the limonene content. The increased temperatures and extended time during hydrodistillation are the responsibles of losing some volatile compounds, but the limonene content is higher than the obtained by cold pressing.

However, cold pressing isolate non-aroma active fats, which is a disadvantage (Radan, Parčina, & Burčul, 2018). So, the drawback of obtaining an OEO with low purity could be avoid using pre-treatments as enzymes that have been researched in other matrixes to enhance de quality and amount of the essential oils obtained (Reyes-Jurado et al., 2015).

3.3. Microwave assisted distillation

Microwave assisted distillation works essentially the same as traditional distillation, but with the difference that the extraction occurs as the result of changes in the cell structure caused by microwaves. In this way, the extraction of the essential oils is reached in shorter times without changes in the essential oil composition compared to the distillation (Beoletto, de las Mercedes Oliva, Marioli, Carezzano, & Demo, 2016). Table 4 reports the main results obtained for orange essential oil extraction using microwave assisted distillation.

Bustamante et al. (2016) optimized the OEO extraction using microwave assisted hydrodistillation in two cycles for a total of 30 min of extraction with a yield of 2.06% (Bustamante et al., 2016), higher result than the obtained by Ciriminna et al. (2017) also in two cycles of 90 min in total (1.63%) (Ciriminna et al., 2017). González-Rivera et al. (2016) also optimized the essential oil extraction with microwave assistance at 400 W during 18 min having a lower yield than the techniques explained before (1.18%). Going further, they also combined ultrasound at 2700 W with coaxial microwave irradiation at 500 W during 80 min reaching a yield of 1.63% of orange peel essential oils (González-Rivera et al., 2016). A study (Duran Baron & Villa, 2014) reported that without water as solvent, the orange peel was carbonized when the microwave power was higher than 600 W. They also reported that the limonene content extracted by microwave assisted hydrodistillation did not depend on the maturity stage of the orange peels. Franco-Vega et al. (2016) optimized the conditions extraction of OEO by using microwave assistance. The yields obtained ranged from 0.9 to 2.5%. They reported that the microwave power only has influence in the time of the extraction. Thus, they corroborate as the time of extraction using 360 W is higher than that is necessary when 540 W were applied (Franco-Vega et al., 2016). The extraction of limonene has also been optimized using microwave assistance using hexane at 110 °C for 30 min. The yield obtained with this technique is 11.1% in front of 4.7% obtained with conventional heating without microwaves (Attard et al., 2014).

Solvent-free microwave distillation has been used to extract OEO, obtaining higher product yield and product quality than hydrodistillation. Moreover, it is less time and energy consuming, letting obtain more oxygenated compounds. A recent study compares the OEO extracted by solvent-free microwave assisted extraction at 200 W for 10 min at 100 °C, with the traditional hydrodistillation and cold-pressing techniques. The solvent-free microwave assisted extraction caused less modifications in the cellular structure than the other techniques, in addition it shows higher efficiency, faster kinetics and higher radical scavenging activity (Aboudaou et al., 2019). This extraction has also been optimized by other researchers obtaining as optimal conditions 400 W for 60 min (Qadariyah et al., 2017).

A new approach is to use microwave absorption mediums into fresh

Table 4
Experimental conditions and yield of orange essential oils obtained by microwave assisted distillation.

Sample	Variety	Procedure	Conditions	Yield (v/w)	Reference
Peel	*C. sinensis Navel Navelate*	Microwave assisted hydrodistillation	Water 10:1 (v/w) Two cycles: 1°, 982 W, 5 min 2° 250 W, 30 min	2.06%	(Bustamante et al., 2016)
Biomass waste	Orange peel		Water 2:1 (v/w) at 94 °C Two cycles: 1° 600 W, 10 min 2° 500 W, 80 min	1.63%	(Ciriminna et al., 2017)
Fruit	*C. sinensis Valencia*		Water 5:1 (v/w), 600 W, 10 min	0.58%	(Duran Baron & Villa, 2014)
Peel	*C. sinensis Valencia*		Water 2,8:1 (v/w), 540 W, 18 min	2.47%	(Franco-Vega et al., 2016)
Biomass waste	Orange peel	Microwave assisted hydrodistillation	Water 6:1 (v/w), 400 W, 18 min	1.18%	(González-Rivera et al., 2016)
		Coaxial microwave assisted hydrodistillation	Water 6:1 (v/w), 300 W, 76 min	1.59%	
		Coaxial microwave assisted hydrodistillation with ultrasounds	Water 6:1 (v/w), 500 W, 80 min with ultrasounds at 2700 W	1.63%	
Biomass waste	Orange peel	Microwave assisted distillation	Hexane 20:1 (v/w), 110 °C, 200 W, 30 min	1.11%	(Attard, Watterson, Budarin, Clark, & Hunt, 2014)
Peel	*C. sinensis Valencia*	Solvent-free microwave distillation	200 W, 10 min, 100 °C	0.40%	(Aboudaou, Ferhat, Hazzit, Arino, & Djenane, 2019)
Peel	*C. sinensis*		400 W, 60 min	1.67%	(Qadariyah et al., 2017)
Biomass waste	Orange peel	Solvent-free microwave distillation assisted with absorption medium	85 W, 30 min, 30 g of graphine powders for 100 g of sample	1.30%	(Ying, De-Sheng, & Zi-Ming, 2010)
Biomass waste	*C. sinensis Valencia*	Microwave assisted steam distillation	200 W, Flow rate of 25 g/min, 12 min	1.60%	(Farhat et al., 2011)

sample system in order to improve conventional microwave heating mode using solvent or water to absorb microwave energy. According to Ying et al. (2010), carbonyl iron powders and graphine powders are two absorption mediums which are suitable for extracting orange essential oils. With this technique of solvent-free microwave assisted extraction with absorption mediums, less time and low electricity consumption were obtained if compared with microwave-assisted hydrodistillation without absorption medium or conventional hydrodistillation (Ying et al., 2010). This extraction with carbonyl iron powder for extracting essential oils from the orange peel was also used by Ying et al. (2010), who also found that it took much less time of extraction (30 min) than microwave-assisted hydrodistillation (90 min) and conventional hydrodistillation (180 min). Also they reported that there were no composition changes in the essential oils extracted by the different methods (Ying et al., 2010).

The microwave assisted steam distillation has been optimized by Farhat et al. (2011); optimal conditions were obtained using a steam mass flow rate of 25 g/min and a microwave power of 200 W. They reported a reduction in more than three times in the time of the extraction needed in comparison with conventional steam distillation for obtaining high yields of extraction. Finally, a study compared the microwave assisted distillation and the steam distillation for extracting essential oil from the orange peel, and no significant changes in yield were noticed (Boukroufa, Boutekedjiret, Petigny, Rakotomanomana, & Chemat, 2015). This could mean that the implementation of steam in the microwave distillation does not give significative increments of yields and is more expensive.

3.4. Supercritical extraction

The supercritical extraction method has been developed in recent years. It is used to extract substances from a matrix using a solvent at supercritical conditions. Both the temperature and pressure of the supercritical fluid are higher than the critical point. Not only is this technique faster than conventional or traditional methods, it also is more environmentally friendly (Reyes-Jurado et al., 2015). Nevertheless, the main advantage of this technique is the possibility of modifying the selectivity of the process when the density of the fluid is changed for being soluble the targeted compounds (Lizcano, Dávila, & Hernández, 2019; Wang et al., 2016). Berna et al. (2000) studied the influence of different factors on the supercritical fluid extraction of OEO. On the one hand they studied the influence of the height of the particle bed on the kinetics of extraction. They did not observ the formation of masses of particles or lack of homogeneity in the fluid flow thanks to adding a diatomaceous earth, so the height of the bed has very little effect, at least on the same scale of operation (Berna, Tárrega, Blasco, & Subirats, 2000). On the other hand they studied the solubilization of the orange essential oil compounds as limonene and linalool in compressed carbon dioxide flow, finding that limonene is more soluble than linalool and that the best conditions seem to be elevated pressure and temperature near to the critical temperature of the carbon dioxide (Berna, Chafer, & Monton, 2000). However, according to del Valle et al. (2019) the optimal conditions found for obtaining an orange essential oil composed in 99.5% of limonene with this technique are 12.5 MPa and 35 °C, whereas for extracting linalool the conditions changed to 8 MPa and 35 °C (del Valle et al., 2019). Xhaxhiu and Wenclawiak (2015) also optimized the extraction of essential oils from the orange peel using supercritical CO_2 and ultrasonic extractions. The optimal condition obtained for the supercritical extraction were 200 atm, 40 °C, and flow rate of 1.6 ml/min during 15 min with a yield of 0.23%, very similar than the obtained by ultrasound extraction in a bath at 10 W during 60 min. Despite needing more time with the ultrasound technique, the composition of the essential oil was the same with both technique, with D-limonene as main compound followed by beta-myrcene, decanal, alpha-pinene, linalool and valencene (Xhaxhiu & Wenclawiak, 2015). Lin et al. (2010) also proposed the supercritical extraction of the orange essential oil with CO_2 at flow rate 3.5 kg/h, during 90 min at 48 °C and 100 atm obtaining extracts with high antimicrobial activity composed mainly by limonene, linalool and alfa-terpineol (Lin, Sheu, Hsu, & Tsai, 2010).

4. Encapsulation of essential oils of orange by-product

Briefly, encapsulation consists of entrapping a substance within another, producing particles with low diameters. The encapsulated substance, in this case the OEO, is the active agent, and the encapsulating substance is the core material. The nature of encapsulating agents and the process used to produce the capsule influence the protection of the OEO. Depending on its particle size, encapsulates can be nanocapsules (nm) or microcapsules (μm). To obtain them, there are physical, physical–chemical and chemical methods, and depending in the selected one, the encapsulated will have different properties. So, selecting the better one for the ingredients of interest is essential (Ye, Georges, & Selomulya, 2018).

4.1. Physical methods

4.1.1. Spray drying

This encapsulation technique consists of transforming a liquid into a powder by steam injection. During this process, the evaporation of the solvent is rapid and the entrapment of the compounds of interest occurs almost instantaneously (Ozkan, Franco, De Marco, Xiao, & Capanoglu, 2019). Spray drying is an unitary operation whereby a liquid product is atomized in a hot gas stream to instantly obtain a powder in an atomizer that is subsequently separated by a cyclone. The gas generally used is air or more rarely an inert gas such as nitrogen. The initial liquid that feeds the sprayer can be a solution, an emulsion or a suspension. Spray drying can produce a very fine powder with a particle size of 10 to 50 μm; or, large sizes that can be from 2 to 3 mm. The shapes and structure of the microcapsules vary from simple spherical shapes with a uniform thickness coating to irregular particles both inside and outside. One or more matrices can be trapped in the centre and the coatings can be made up of more than one layer (Sandoval-Peraza, Cu-Cañetas, Peraza-Mercado, & Acereto-Escoffié, 2016). The characteristics of the final product obtained depend on the drying temperature, the hot air flow rate, the feed flow rate, the atomization rate and the type and concentration of the carrier agent. This carrier agent can be polysaccharides such as Arabic gum, cyclodextrins and maltodextrin with different equivalent values of dextrose, proteins such as whey proteins, sodium caseinate, soy proteins and others, such as modified starch, gelatine, gellan gum and chitosan (Ozkan et al., 2019). According with Galmarini (2020) the aromatic profile and sensory properties of the OEO is determined in part by the matrix wall composition by spray-drying encapsulation. When encapsulation was obtained using a combination of maltodextrin-trehalose the attributes woody, marmalade, syrup, citrus terpenes and vitamin C are perceived, meanwhile the attributes perceived in encapsulated by maltodextrin-Arabic gum are peely, plastic, solvent and green (Galmarini, 2020). Carmona et al. (2013) encapsulated the OEO by spray drying using whey protein concentrate and maltodextrin (1:3) as wall materials obtaining small droplets with good oil retention (Carmona, Tonon, da Cunha, & Hubinger, 2013). Sosa et al. (2014) compared the use of mixtures of trehalose-maltodextrin and sucrose-maltodextrin as encapsulate agents demonstrating that the type of starch used as an emulsifier affect the retention of volatiles. So, trehalose formulations retain mainly limonene, while sucrose formulations retained mostly α-pinene and myrcene, both with good physical and sensorial characteristics (Sosa, Zamora, van Baren, & Schebor, 2014). Rojas-Moreno et al. (2018) used *N*-Lok modified starch for encapsulating OEO in a ratio 1:4 with high retention and encapsulation efficiencies (>75%) (Rojas-Moreno, Cardenas-Bailon, Osorio-Revilla, Gallardo-Velazquez, & Proal-Najera, 2018). Souza et al. (2020) used Arabic gum and maltodextrin as wall materials incorporated with cellulose nanofibrils. They showed that

the presence of cellulose nanofibrils increases the thermal stability of the microcapsules apart from acting as a physical barrier allowing stabilization of the interface oil/water, what contributes to the improvement of the properties of the essential oil microparticles. Otherwise Arabic gum was reported to show better equilibrium moisture than maltodextrin (de Souza et al., 2018; Souza et al., 2020). Marquez-Gómes et al. (2018) used a modified rice starch as wall material combined with native rice starch, maltodextrin and protein; they noticed that the best encapsulating agent was the one with more than 50% of modified rice starch, demonstrating a higher efficiency than commercial wall materials (Marquez-Gomez, Galicia-Garcia, Marquez-Melendez, Ruiz-Gutierrez, & Quintero-Ramos, 2018). Anyways, an aqueous solution made of Arabic gum and maltodextrin 1:1 also has been used to encapsulate *Nigella sativa* seeds essential oils commonly used in the fortification of processes food (Edris, Kalemba, Adamiec, & Piaotkowski, 2016). So, the discussed studied of encapsulating the OEO with this technique would let to use it as a nutraceutical or in food applications in a stable form.

The main advantage of the spray drying technique is that it has good encapsulation efficiency and good stability of the final product. It allows continuous scaling and production. However, the main disadvantage is the damage that high temperatures can cause to sensitive bioactive compounds. Furthermore, it is difficult to control the final size of the drops (Suganya & Anuradha, 2017). To solve the problem of the high temperatures, the effect of using reduced pressure and absence of oxygen during the spray drying process (vacuum spray drying) has been investigated by de Melo Ramos et al. (2019). To encapsulate OEO, they used octenyl succinic anhydride modified starch combined with maltodextrin. This innovative inclusion of vacuum to the conventional spray drying let obtain a powder with lower mean diameter and wettability besides higher encapsulation efficiency and moisture content. It does not affect the solubility of the samples and the particle does not present fissures or cracks although more morphological changes of the structure must be studied. However, this technique seems to be promising because of the lower temperature needed, which improve the stability of thermo-sensitive compounds (de Melo Ramos, Silveira Júnior, & Prata, 2019).

4.1.2. Freeze drying

Freeze drying, also known as lyophilization, in the context of encapsulation technique, is a multi-stage process consisting of a first stage of freezing, followed by sublimation (primary drying), and subsequent desorption (secondary drying). The most significant advantage of lyophilization is that it is a simple process carried out at low operating temperature with the absence of air that produces superior and prolonged quality products by preventing deterioration caused by oxidation or chemical modification. Briefly, lyophilization is the most suitable technique for the dehydration of almost all heat-sensitive substances (Ozkan et al., 2019). De Araújo et al. (2020) studied the microencapsulation of the OEO with maltodextrin and gelatine by emulsification followed by lyophilization with high yields and efficiency. Gelatine improves the thermo-oxidative stability of the encapsulated, so in combination with maltodextrin, the antimicrobial and antioxidant properties of the sweet OEO are maintained. In contrast, microspheres formulated only with maltodextrin provide more regular surfaces, better thermal stability and higher antioxidant properties. However, in both cases, the stability of the sweet OEO is prolonged and protected against environmental factors and prolonged storage time (de Araújo et al., 2020). However, the freeze-drying technique has some drawbacks, such as the long process time (more than 20 h), the high capital and the operating costs compared to others. The porous structure of lyophilized powders due to the sublimation of ice during the process is also one of the main limitations since they must be crushed or converted into fine powders after drying, and problems related to the lack of control over particle size can be found (Ozkan et al., 2019).

4.1.3. Fluid bed coating

A fluidized bed is used to coat solid core particles. The solid particles are suspended in the air and the encapsulating material is sprayed onto the particles, forming a coating. The encapsulating material may be a concentrated solution or dispersion, a hot melt, or an emulsion. Most encapsulating materials (i.e. fats, carbohydrates, emulsifiers, proteins) can be used in this process, allowing the development of particles with very different controlled release properties. This method can be used to give a secondary coating after spray-dried process adding more protection. As a limitation, it can only be used on solid compounds after grinding or milling to equalize the particle size (McClements, 2020). A study reports the encapsulation of OEO with this technique, using modified starch (*N*-Lok) in a spout-fluid bed dryer with a draft tube on a bed of inert solids; the efficiency obtained was lower than those obtained by using only spray drying. Contrary, it provided better protection to the essential oil and most compounds were encapsulated (94% in front of 70% in the spray dryer) (Velazquez-Contreras, Osorio-Revilla, & Gallardo-Velazquez, 2014).

4.2. Physical-chemical methods

4.2.1. Coacervation

Coacervation is a phase separation technique of a single polyelectrolyte or a mixture of polyelectrolytes of opposite charges from a solution and the deposition of the agglomerated colloidal particles (the matrix material) into an immiscible active nucleus resulting in the formation of a simple coacervate or a complex coacervate. Complex coacervates of oppositely charged biopolymers have been used in the food industry for encapsulation of active ingredients in foods such as flavours, actives and water-soluble oils. Many factors, including the type of biopolymer (molar mass, flexibility and charge), pH, ionic strength, concentration and the ratio of biopolymers affect the strength of the interaction between biopolymers and the nature of the complex formed (Ozkan et al., 2019). Although electrostatic interactions are considered to drive the interaction between oppositely charged biopolymers; hydrophobic interactions and hydrogen bonds can also contribute significantly to complex formation. Coacervates can form when a protein at a pH below its isoelectric point mixes with a polyanion (McClements, 2020). This complex coacervation has been carried out by Jun-Xia et al. (2011) to encapsulate OEO with soybean protein isolate and Arabic gum in ratio 1:1 and pH 4 with sucrose as cross-linker, obtaining good efficiency and yield. They showed that with this matrix, higher ionic strength reduced the coacervation. With these two biopolymers the microcapsules are spherical without holes on the surface and volatile compounds are well retained without loses of limonene (Jun-xia, Hai-yan, & Jian, 2011). Rojas-Moreno et al. (2018) employed the complex coacervation of the orange essential oil using whey protein isolate and Arabic gum in ratios 1:2, obtaining a retention and encapsulation efficiency of 25% lower than the conventional spray drying technique, and showing higher degradation after 4 months of storage (Rojas-Moreno, Cardenas-Bailon, et al., 2018). They also studied other polysaccharide wall material combinations with whey protein isolate as carboxymethylcellulose, sodium alginate and chitosan, with the help of tannic acid, sodium tripolyphosphate, oxidised tannic acid and transglutaminase enzyme as cross-linkers. Without the cross-linkers the coacervated microcapsules form solid cakes. The best systems found for encapsulating the orange essential oils were whey protein isolate combined with carboxymethylcellulose with tannic acid as cross linker, and whey protein isolate combined with chitosan with transglutaminase enzyme as cross linker, obtaining the higher yields of 47 and 50%, respectively (Rojas-Moreno, Osorio-Revilla, Gallardo-Velazquez, Cardenas-Bailon, & Meza-Marquez, 2018).

The coacervation usually goes followed by a physical method as spray drying or freeze drying. The coacervation technique is superior to other microencapsulation techniques due to its high load capacity, low temperature, lower losses due to evaporation or thermal degradation,

and compatibility to control the release of active materials. Furthermore, no specific equipment is required for its implementation and it has simple setup conditions and low agitation utilization. On the other hand, the high cost of the particle isolation procedure and the complexity of the technique must also be taken into account (Ozkan et al., 2019).

4.2.2. Emulsification

A two phases system composed by two immiscible liquids, which are commonly oil and water, is called emulsion. Depending on the majoritarian phase, the emulsions can be water in oil, or oil in water. In the case of the OEO, they are oil in water, being the OEO the disperser phase and the water or another liquid, the continuous or dispersing phase. Emulsions systems that can be applied in OEO can be classified as nano-emulsion and micro-emulsion depending on its physical properties and thermodynamic stability. Nano-emulsions have a droplet size of 20–200 nm and they are thermodynamically unstable but with a transparent or translucent appearance. Meanwhile, micro-emulsions have a smaller particle size (5–50 nm) and they are more thermodynamically stable and clearer (Jin et al., 2016).

Dat et al., 2020 developed an OEO in water nano-emulsion combined with nanosilver particles with a particle size of 42.9 nm by ultrasonic method obtaining an antibacterial media (Dat et al., 2020). Another study reported a stable nano-emulsion (12.68 nm) of OEO prepared with a mixture of Tween 80, combined soluble fractions of Persian gum and tragacanth gum and water by using sonication with amplitude of 94% during 138 s and 37 °C (Hashtjin & Abbasi, 2015). Carmona et al. (2013) optimized a stable micro-emulsion of the OEO in order to encapsulate it later by spray drying. The optimal conditions were total solid content of 30%, oil concentration of 15% and homogenization pressure of 650 bar. They also reported that the increase in the number of cycles resulted in larger oil droplets and lower oil retention (Carmona et al., 2013). It has been found in other matrixes as *Laurus nobilis* leaves essential oil (Lima Reis et al., 2020) and *Thyme* flowers essential oil (Bilenler, Gokbulut, Sislioglu, & Karabulut, 2015) that the emulsification process could be assisted by ultrasound technology doing shorter the encapsulation time and making the encapsulates more stable in short period time.

Emulsification technology is one of the most commonly used for protecting the volatile compounds against the environment and improving their antimicrobial capacities (Jin et al., 2016). However, the principal problem of emulsions is its stability. The main instability mechanisms are flocculation, cremation, sedimentation, coalescence, Ostwald ripening and phase separation. Because of that, this method must be followed by another one as spray drying or freeze drying (Karthik, Ezhilarasi, & Anandharamakrishnan, 2017).

4.2.3. Liposomes

Liposomes are spherical bilayer vesicles that are formed by dispersions of polar lipids (i.e. phospholipids, that have a hydrophilic head and a hydrophobic tail) in an aqueous media. The size and structure of the liposomes depends on the composition, the preparation method and the environmental conditions. Liposomes can be used as carriers for both hydrophilic, amphiphilic and lipophilic molecules. The trapped actives stabilize against changes in the environment (pH, temperature, ionic strength). The contents of the core are released when the gel-to-liquid transition temperature of the phospholipids used in the formulation is reached. Other unique properties of liposomes are their high bioavailability, biocompatibility, biodegradability and high permeability of the cell membrane (Ye et al., 2018). It has been developed a liposome (nanoethosome) based on ethanol, water, soybean phosphatidylcholine (SPC), Tween 80, and palm oil sucrose esters in optimized proportions for encapsulating OEO for its application in fragrances of long-duration with a positive correlation between particle size and sustained-release effectiveness (Chen et al., 2019). Other essential oils as *Artemisia annua* flowers essential oil has been encapsulate in nanoliposomes with cholesterol and SPC, also with good efficacy and stability and even with enhanced antimycotic activity in vitro analysis (Risaliti et al., 2020). The main limitations of liposome encapsulation are poor physical and chemical stability, a wide range of particle size distributions, lipid oxidation, and the need for complex post-treatment steps. In summary, although this method provides a high bioavailability of the orange essential oil, its low physical and chemical stability must be considered during its application (Ozkan et al., 2019).

4.2.4. Ionic gelation or electrospinning

Ionic gelation is a microencapsulation technique based on the creation of a gel in the presence of multivalent ions such as Ca^{2+}, Ba^{2+} and Al^{3+}. The interaction that occurs is a divalent ionic crosslinking between the divalent molecule ions and the envelope polymer. This technique was initially developed for cell immobilization (Sandoval-Peraza et al., 2016). It can be done by a simple extrusion process in which pressure is applied to force a hot biopolymer mass containing the active core to disperse through a hole in a hardening bath. This process has been widely used to microencapsulate flavours in glassy carbohydrate matrices. The flavour is injected into a hot mass of the molten biopolymer and extruded in a hardening bath, usually with isopropyl alcohol (McClements, 2020). By this way, the OEO has been encapsulated with this technique in chitosan microcapsules prepared with different Tween and Span emulsifiers. It has been showed that the microcapsules made with Tween 60 obtained the minimum particle size and in consequence needs the lowest energy for melting. So, the emulsifier modify the intermolecular interactions and it must be have in consideration (Li et al., 2018). Tavassoli-Kafrani et al. (2018) studied the encapsulation of OEO with electrospinning using gelatine and gelatine-cross linked tannic acid nanofiber as wall ingredients in ratios 1:1, finding with both encapsulation efficiency higher than 50% and good storage stability (Tavassoli-Kafrani, Goli, & Fathi, 2018). This electrospinning technique also has been developed in *Laurus nobilis* and *Rosmarinus officinalis* leaves essential oils using zein nanofibers coats. It lets to enhance their antimicrobial properties in food films applications when comparing with other traditional casting methods (Göksen, Fabra, Ekiz, & López-Rubio, 2020).

4.3. Chemical methods

4.3.1. Interfacial polymerization

Wall formation in this technique is characterized by polymerization, in which hydrophilic and lipophilic monomers interconnect in an oil–water emulsion and react to form a polymeric membrane on the surface of the droplet or particle. The performance and quality of the polymeric membrane manufactured using this technique could be optimized by controlling the process parameters, including monomer concentrations, temperature, mixing speed, and reaction time. Mainly four types of polymers have been developed to produce microcapsules by interfacial polymerization, consisting of polyamides, polyurethanes, polyureas, and polyesters. Velmurugan et al. (2017) used this emulsion polymerization technique to encapsulate OEO with chitosan and potassium per sulphate at 50 °C during 8 h. They obtained microcapsules with bimodal and uniform distribution without effects against the oil constituents (Velmurugan, Ganeshan, Nishter, & Jonnalagadda, 2017).

The interfacial polymerization technique has potential advantages, including possible control of mean capsule size and membrane thickness, high active compound loading, versatile and stable membrane chemical and mechanical properties, low cost, ease of scalability, simplicity and reliability of the process. On the other hand, there are also some factors that limit the application of this technique, such as the difficulty of producing this oil–water interface (McClements, 2020).

4.3.2. Complexation by molecular inclusion

Molecular inclusion is an encapsulation technique that takes place at the molecular level and involves the entrapment of the interest compound by a host (polymer) through physicochemical forces, such as hydrogen bonding, van der Waals forces, or hydrophobic interactions.

These complexes are formed through a reaction that takes place only in the presence of water. The most common "host" molecules are cyclodextrins, which are composed of a hydrophilic external part and an internal hydrophobic part (Ozkan et al., 2019). The formation of complexes with a hydrophobic molecule is due to the fact that the displacement of water from inside the cyclodextrin is energetically favourable. Complexation increases the solubility of hydrophobic host molecules in water and the ingredient is protected against degradation (McClements, 2020). For protecting the OEO from volatilization and oxidation, the molecular inclusion with beta-cyclodextrin has been optimized with high efficiency results (greater than50%) increasing its solubility and bioavailability (Li, Wu, Huang, Guo, & Dou, 2018). The OEO has been encapsulated with this technique with zein and beta-cyclodextrin inclusion complex and applied as a conservator for bakery products with good stability (Kringel, da Silva, et al., 2020). Otherwise, cyclodextrins produced from germinated wheat starch, has been applied successfully for molecular inclusion complex of orange essential oil increasing its thermal stability (Kringel, Baranzelli, et al., 2020). In general cyclodextrin has been studied for encapsulating orange essential oils by molecular inclusion because it attributes them with higher stability compared with physical mixtures (Kringel et al., 2017).

This technique is worth it because of the slow loss of the volatile compounds, the higher stability when compared with other techniques. Therefore, the results of the studies showed that the chemical composition, molecular size, and structure of the essential oil influence the characteristics of the inclusion complexes. The application of the cyclodextrins in the formation of inclusion complexes with essential oils can expand the potential applications in foods (Ye et al., 2018).

5. Conclusions

Orange essential oils are a good source of antioxidant and antimicrobial compounds that are commonly used for healthy and technological scope in foods. Thus, this review summarized the recent researches on the extraction and encapsulation techniques to obtain orange essential oil functional ingredients. It was underlined as several methodologies could be applied depending on the end use of the ingredient formulated.

6. Future trends or challenges

The use of OEO is a very promising topic for food and cosmeceutical industries. Beside several new technologies have been used to produce functional OEO, the use of other sustainable technologies, such as pulsed electric fields among others, should be applied to improve the recovery of essential oils from orange by-products. The information on encapsulation of OEO provided in this review may encourage further research on the use of this ingredient for different scope improving the application in food and cosmeceutical products. In this way, the application on active packaging and the study of controlled release could be very interesting research areas to develop.

CRediT authorship contribution statement

María del Carmen Razola-Díaz: Writing - original draft. **Eduardo Jesús Guerra-Hernández:** Supervision, Writing - review & editing. **Belén García-Villanova:** Writing - review & editing. **Vito Verardo:** Conceptualization, Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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