


Microencapsulation of oregano essential oil by spray-drying using maltodextrin: gum arabic blends

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ABSTRACT

The effect of processing parameters on microencapsulation of oregano essential with maltodextrin:gum arabic using a disk atomiser spray-dryer was evaluated. By means of response surface methodology, the feed flow rate and inlet air temperature were optimised. Powder yield, moisture content, essential oil retention, and antioxidant activity of microparticles were evaluated. The best conditions to produce microencapsulated oregano essential oil were 0.6 L h⁻¹ for feed flow rate and 200 °C for inlet air temperature. With this combination a microencapsulated powder with 89.8% powder yield, 2.1% moisture content, 92.1% essential oil retention, 76 s solubilisation time, 12.9 g of water/100 g of dry matter, 0.3371 g mL⁻¹ bulk density, 0.5826 g mL⁻¹ tapped density, and 8.2 µm of average particle size was produced. The microencapsulation of oregano essential oil preserves the antioxidant and antimicrobial activities of its bioactive compounds.

KEYWORDS

oregano essential oil, microencapsulation, spray drying, optimisation, maltodextrin, gum arabic

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1. INTRODUCTION

Oregano (*Origanum vulgare* L.) is an aromatic plant belonging to the Lamiaceae family commonly occurring in many parts of the world. The species is used as culinary herb in gastronomy but also used in folk medicine, and previous researches demonstrated the potential of the essential oil to preserve foods (Beirao da Costa et al., 2012; Teixeira et al., 2013; Kosakowska et al., 2021). Incorporation of essential oils in food is commonly achieved in a solid form resulting from the microencapsulation of the essential oil by a wall material (Assadpour and Jafari, 2019).

The microencapsulation technique by spray-drying has currently been preferred in food and pharmaceutical industries for several purposes including controlled delivery of bioactive compounds and flavours, as well as the stabilisation of antimicrobial substances and antioxidants. This technology offers good protection of the encapsulated compounds from adverse environments and limits chemical changes or losses of key substances (Jafari et al., 2008; Saifullah et al., 2019; Plati and Paraskevopoulou, 2022).

The most common wall materials (WM) used to protect the microencapsulated core are modified polysaccharides, gums, proteins, or their combinations. The characteristics of the WM, the emulsification of the hydrophobic core and aqueous phase, and the drying parameters influence the retention and stability of the core. Gum arabic is successfully used as WM, because it provides excellent emulsifying properties with essential oils and provides low solution viscosities. Combinations of maltodextrin:gum arabic are known to have lower costs and higher encapsulation efficiency and stability (Bringas-Lantigua et al., 2011, 2012). Oregano essential oil has been microencapsulated by spray-drying (Baranauskiene et al., 2006; Botrel et al., 2012; Beirao da Costa et al., 2012; Da Costa et al., 2013; Hernández-Hernández et al., 2014; Asensio et al., 2017). However, in these studies other WM in place of maltodextrin:gum arabic had been used, in many of them the processing parameters were fixed, and the studies were done in mini co-current flow spray-dryer, except the study of Baranauskiene et al. (2006), where a disk atomiser was used. Disk atomiser spray-dryers are commonly used in the industrial sector. Baranauskaite et al. (2016) optimised the spray-drying process conditions for microencapsulation of a Turkish oregano (*Origanum onites* L.) hydroalcoholic extract using maltodextrin:gum arabic combinations as WM, but the chemical composition of the extract is very different from that of an essential oil as nonvolatile compounds are solubilised in the extraction solvent.

The aims of this work were to evaluate the microencapsulation of oregano essential oil with maltodextrin:gum arabic using a disk atomiser spray-dryer and to select the optimum combination of feed flow rate and inlet air temperature by means of response surface methodology.

2. MATERIALS AND METHODS

2.1. Materials

Oregano (*O. vulgare* L.) essential oil (OEO) was supplied by Aromalab (Madrid, Spain). Gum arabic (Chunchi, China) and 20 DE maltodextrin (Qinhuangdao Lihua Starch Co., Ltd., Hebei, China) were used as wall materials.



2.2. Spray drying of the essential oil

For the spray drying an emulsion was prepared with 7% w/w OEO, 9.3% w/w gum arabic, 18.7% w/w maltodextrin and 65% deionised water (35% w/w total solids feed). The maltodextrin and gum arabic were hydrated overnight in deionised water, then the essential oil was added using an Ultra Turrax T-25 (IKA, Germany) at $10,000 \text{ min}^{-1}$ for 10 min. The drying was achieved in a Mobile Minor rotary atomiser spray dryer (Niro Atomizer Ltd., Copenhagen) with the atomiser's rotating speed at $25,000 \text{ min}^{-1}$, feed flow rates at 0.6, 0.8, and 1 L h^{-1} , inlet air temperatures at 160, 180, and $200 \text{ }^\circ\text{C}$, and outlet air temperature at $75 \pm 5 \text{ }^\circ\text{C}$. A feed solution (600 g) was made for each experimental run. These parameters were chosen based on preliminary experiments, which yielded what appeared to be an acceptable spray dried powder. These conditions almost cover all previous works. The powders were recovered from the spray dryer, packed in polyethylene bags, and kept in a desiccator at $20 \text{ }^\circ\text{C}$ until analysis. Also, two replicates were made under the best spray dryer conditions.

2.3. Powder analytical methods

2.3.1. General methods. Powder yield was estimated by the total mass of recovered powder and feed mass ratio, both at dry basis (Botrel et al., 2012). Moisture content was determined at $105 \text{ }^\circ\text{C}$ by a thermobalance MA35 (Sartorius, Göttingen, Germany). Solubility was evaluated according to Miravet et al. (2016) with minor modifications: 2 g of powder were dissolved in 50 mL of water using a 20-mm cylindrical magnetic stirrer at $1,000 \text{ min}^{-1}$. Hygroscopicity was determined by the procedure of De Barros Fernandes et al. (2016). Briefly, 1 g of powder was placed in a desiccator with a saturated NaCl solution (75.29% RH), and after one week the powder samples were weighed. Bulk and tapped densities were measured by the procedure described by De Barros Fernandes et al. (2016). Particle shape and average particle size D_{43} were assessed with a scanning electron microscope 5130 SB (Tescam, Prague, Czech Republic) operated at 10 kV. All determinations were done in triplicate.

2.3.2. Essential oil retention. The spray-dried microcapsules were analysed for total essential oil by HS-SPME technique (Sánchez-Cabrera and Pino, 2011). HS-SPME parameters: 100 μm polydimethylsiloxane fibre, 0.2 g powder, 8 mL Milli-Q water, pre-extraction 15 min, extraction 25 min at $35 \text{ }^\circ\text{C}$, and desorption at $250 \text{ }^\circ\text{C}$ for 2 min. Gas chromatography was performed in a QP-2010 Ultra instrument (Shimadzu, Japan). GC parameters: SGE Analytical Science BP-5ms capillary column ($30 \text{ m} \times 0.25 \text{ mm} \times 0.25 \mu\text{m}$); carrier gas He at 1 mL min^{-1} ; temperature program: $70 \text{ }^\circ\text{C}$ hold 2 min, $4 \text{ }^\circ\text{C min}^{-1}$ – $230 \text{ }^\circ\text{C}$, hold 10 min; splitless time 2 min. MS parameters: ion source $220 \text{ }^\circ\text{C}$; ionisation EI 70 eV; from 35 to 350 mass; and scan time 0.2 s. Identification of compounds was made by comparison of the linear retention index and mass spectra of authentic compounds, as well as by comparison with those in mass databases (Flavorlib, NIST 02, and ADAMS 2001). Semi-quantification of components was performed from the chromatographic peak areas without considering correction factors. Essential oil retention was calculated as the total peak areas of the essential oil in the powders to that in the prepared emulsion to be spray-dried (dry weight basis). Dispersions of powder (200 mg) or a corresponding quantity of OEO (40 mg), maltodextrin (53 mg), and gum arabic (107 mg), which represented 20% theoretical oil content assuming ideal retention, were prepared for analyses. All determinations were done in triplicate.



2.3.3. Antioxidant capacity. The antioxidant capacity was determined using the FRAP assay (Suravanichnirachorn et al., 2018). Calculation of the antioxidant activity was performed using a calibration curve of Fe^{2+} with Mohr's salt as standard. The tests were performed in triplicate.

2.3.4. Microbiological analysis. The effect of the powder on bacterial strains with food relevance was evaluated. The selected microorganisms were *Salmonella enterica* U822s, *Staphylococcus aureus* ATCC 25923, *Escherichia coli* ATCC 25922, *Listeria monocytogenes* ATCC 19115, and *Bacillus cereus* ATCC 10876. The bacterial inocula were prepared from young cultures of less than 24 h, by suspensions of sterile solutions of sodium chloride (0.85%), which were shaken, homogenised, and the microbial concentration was calculated by diluting with sterile solutions until adjusting a concentration of 10^6 CFU mL^{-1} using the McFarland scale. A determined amount of OEO (7 mg) or powder (35 mg) was dissolved in 10% (v/v) Tween 80. The antimicrobial activity was evaluated by the tube dilution method to estimate the minimum inhibitory concentrations (MIC). The concentrations studied were between 0.005 and 10 mg L^{-1} from powder dilutions in dimethyl sulfoxide. In test tubes, 1 mL of the powder dilutions was mixed with 1 mL of the microbial inoculum (10^6 CFU mL^{-1} suspended in Mueller Hinton broth). The tubes were incubated at $32 \pm 2^\circ\text{C}$ for 24 h. Then, 1 mL of the content of the tubes was seeded, using Mueller Hinton broth and incubated at $32 \pm 2^\circ\text{C}$ for the growth of each microorganism. The MIC was determined as the lowest concentration of the powder capable of inhibiting microbial growth (Murbach Teles Andrade et al., 2014).

2.4. Statistical analysis

A IV-optimal design was used to perform the tests, considering two factors (independent variables): feed flow rate and inlet air temperature. This design gives a total of nine combinations (Table 1). The polynomial models were assessed by the analysis of variance and the model adequacy tests according to the Design-Expert 8.07 (Stat-Ease Inc., Minneapolis, USA). Numerical optimisation was carried out to find the conditions, which maximised powder yield and essential oil retention and minimised the moisture content.

Analysis of variance by *t*-Student test was performed to determine the significance of the effects on each major compound of the OEO before and after microencapsulation under the best spray drying conditions.

Table 1. Experimental design for spray drying assays

Feed flow rate (L h^{-1})	Inlet air temperature ($^\circ\text{C}$)	Yield (%)	Moisture (%)	Essential oil retention (%)	FRAP ($\mu\text{M Fe}^{2+}/\text{g}$ powder)
0.8	180	86.8	4.28	90.6	410.8
0.6	200	90.0	2.09	92.1	435.2
0.8	160	86.3	4.06	84.7	403.8
1.0	200	83.3	3.34	93.2	427.8
0.8	180	85.5	4.25	89.0	410.2
0.8	160	85.6	4.18	84.2	404.3
1.0	160	82.0	4.33	85.5	398.0
0.8	180	87.2	4.30	89.8	401.7
0.6	180	88.5	3.34	88.6	413.0



3. RESULTS AND DISCUSSION

The influence of feed flow rate and inlet air temperature on process yield, moisture content, and essential oil retention are presented in Table 1, while Table 2 shows the coded regression models. The models were significant and lack of fit were insignificant, and thus a good agreement between adjusted and predicted R-square were found, adequate precision was over four, and the residuals were minimal. Therefore, the regression models provide adequate predictions of the response variables for average results.

Powder yields were in the range from 82 to 90% (Table 1). In other reports the data were lower than in the present study: 59.7% for OEO microencapsulated into modified starch:gum arabic:maltodextrin (Botrel et al., 2012), 17.18% for ginger essential oil microencapsulated into maltodextrin:gum arabic 1:1 w/w (De Barros Fernandes et al., 2016), and 31.1–51.8% for OEO essential oil microencapsulated into hydroxypropyl methyl cellulose-maltodextrin-colloidal silicon dioxide blends (Asensio et al., 2017).

Feed flow rate was the factor that most influenced process yield (Table 2). This variable negatively affected powder yield probably by low heat and mass transfer rates. Similar behaviour has been found in microencapsulation based on spray drying with other materials (Tonon et al., 2008; Phisut, 2012). However, Botrel et al. (2012) studying OEO microencapsulated by spray drying using modified starch:gum arabic:maltodextrin (5.1:3.6:1.3 m m⁻¹) reported that a rise in inlet temperature and feed rate produced higher powder recoveries, but they worked at 10% total solids content.

Moisture contents of microcapsules were in the range from 2.09 to 4.33% (Table 1), which was similar to 1.30–3.65% and 0.92–2.13% for OEO (Botrel et al., 2012; Da Costa et al., 2013), 4.9–7.1% for lime essential oil (Bringas-Lantigua et al., 2012), and 3.1–5.1% for OEO (Asensio et al., 2017).

Both factors presented significant influence on powder moisture (Table 2). In fact, a decrease in moisture content of the microcapsules was found when inlet air temperature increased and feed flow rate decreased. This behaviour could be explained considering that at

Table 2. Summary of statistics and parameters of the coded regression models

Parameter	Yield	Moisture	Essential oil retention	FRAP ($\mu\text{M Fe}^{2+}/\text{g powder}$)
Intercept	86.19	4.28	89.80	413.16
X_{FFR}	-3.18**	0.59**	0.93	-2.21
X_{IAT}	0.48	-0.53**	4.23*	13.58
X_{FFR}^2	-	-0.35*	-0.27	-
X_{IAT}^2	-	-0.69**	-1.12	-
$X_{\text{FFR}} \cdot X_{\text{IAT}}$	-	0.03	-0.38	-
<i>F</i> model	40.27**	319.93**	34.92*	12.14*
<i>F</i> lack of fit	0.82	0.20	0.25	3.65
<i>R</i> ²	0.931	0.998	0.983	0.801
Adjusted <i>R</i> ²	0.908	0.995	0.955	0.735
Predicted <i>R</i> ²	0.863	-	-	0.477
Adequate precision	17.04	51.64	15.66	8.606

X_{FFR} , X_{IAT} coded feed flow rate and inlet air temperature, respectively.

*: $P \leq 0.01$; **: $P \leq 0.001$.



high feed rates, the contact time of the droplets with the drying air could not be sufficient to completely evaporate the water. An increase in inlet air temperature gives a decrease in relative humidity of the air, which could remove more moisture from the droplets during spray drying and, therefore, causing fast removal of water (Tontul and Topuz, 2017). These results agree with those found for other essential oils by several reports (Botrel et al., 2012).

The powders have an essential oil retention between 84.2 and 93.2%, which were higher than those found: 73.2% and 81.3% in OEO microencapsulated into milk protein-based matrices (Baranauskiene et al., 2006), <80% in OEO microencapsulated using modified starch:gum arabic:maltodextrin (Botrel et al., 2012), and 33.10–77.39% in OEO into gum arabic:modified starch:maltodextrin blends (Da Costa et al., 2013).

The factor with the higher influence on the essential oil retention was the inlet air temperature (Table 2). The increased inlet air temperature led to higher essential oil retention. According to Jafari et al. (2008), this effect occurs by a reduction in the time necessary to create a semi-permeable film at the drying particle surface. This trend agrees with other studies (Bringas-Lantigua et al., 2011, 2012; Botrel et al., 2012).

The antioxidant potential of microencapsulated OEO as a food ingredient was also evaluated (Table 1). Several works (Kosakowska et al., 2021; Plati et al., 2021) have reported the antioxidant activity of the essential oil of plants of the *Origanum* genus. The antioxidant capacity ranged from 398.0 to 435.2 $\mu\text{M Fe}^{2+}/\text{g}$ powder. Neither the feed flow rate nor the inlet air temperature significantly affected the antioxidant capacity of the powders, but there is a tendency that an increase in the inlet air temperature favoured a higher antioxidant capacity, which could be related to a higher essential oil retention when the value of this factor increased, as explained above.

The predicted optimal conditions were feed flow rate 0.6 L h^{-1} and inlet air temperature $200 \text{ }^\circ\text{C}$, with a desirability function of 0.96. Baranauskiene et al. (2006) fixed $190 \pm 5 \text{ }^\circ\text{C}$ to microencapsulate OEO with whey protein concentrate or skimmed milk powder by means of a rotary atomiser spray-dryer, while Da Costa et al. (2013) used the inlet and outlet air temperatures at $180 \pm 2 \text{ }^\circ\text{C}$ and $105 \pm 2 \text{ }^\circ\text{C}$, respectively, with inulin as WM by means of a co-current mini spray-dryer. Botrel et al. (2012) found that also a high temperature ($185 \text{ }^\circ\text{C}$) and a moderated feed rate (0.63 L min^{-1}) were the best spray drying conditions using modified starch:gum arabic:maltodextrin by means of a co-current atomiser, while Hernández-Hernández et al. (2014) fixed an inlet and outlet air temperature of $190 \text{ }^\circ\text{C}$ and $100\text{--}110 \text{ }^\circ\text{C}$, respectively, with modified starch via a co-current mini spray-dryer. Because the powder reaches the outlet air temperature, it is important to reduce this parameter. Lower outlet air temperature provided higher retention of bioactive compounds at constant inlet air temperature (Shishir and Chen, 2017). According to the outlet air temperature of our study ($75 \pm 5 \text{ }^\circ\text{C}$), it can be stated that the applied procedure can provide a powder more stable.

Under the optimal conditions, 89.8% powder yield, 2.1% moisture content, 92.1% essential oil retention, 76 s rehydration time, 12.9 g of water/100 g of dry matter hygroscopicity, 0.3371 g mL^{-1} bulk density, and 0.5826 g mL^{-1} tapped density were found. Botrel et al. (2012) reported hygroscopicity values between 22.30 and 26.27 g of water/100 g of dry matter, which means that the powder produced in our study absorbs less water during exposure to a high relative humidity. Observed tapped density values were higher than those found by Botrel et al. (2012) ranging from 0.338 to 0.451 g mL^{-1} , but powder density depends on chemical composition, particle size, powder moisture, processing parameters, and handling procedures.



When flavourings and/or bioactive compounds are microencapsulated, it is essential to check possible changes in the chemical composition. Therefore, the analysis of the constituents of the OEO and the powder obtained under the best spray drying conditions was performed by HS-SPME (Table 3). The composition of OEO was dominated by carvacrol, *p*-cymene, and (*E*)-caryophyllene, even using a nonpolar PDMS fibre, which was selected to favour the isolation of minor constituents. Carvacrol is a phenol, which shows wide-ranging uses and biological activities (Hernández-Hernández et al., 2014). Significant differences in composition were found after the microencapsulation (Table 3), which could be related to spray drying. Polar compounds like carvacrol, linalool, and decanal significantly increase their relative percentages in the microencapsulated essential oil, whereas many terpenic hydrocarbons decrease their relative concentrations. Jafari et al. (2008) suggested that a high inlet air temperature accelerates the formation of the crust around the drying droplets and, therefore, increases flavour retention. It is known that hydrophobic volatile compounds are less protected in the droplet and are more easily lost (Baranauskiene et al., 2006). These reasons could explain the higher losses of *p*-cymene and other terpenic hydrocarbons.

Table 4 shows that OEO inhibited the growth of bacteria, with MIC values of 0.1 mg L⁻¹ in all cases, while microencapsulated OEO showed higher MIC values, i.e. less effective antibacterial activity compared to OEO. In the case of microencapsulated OEO, considering the MIC values, *E. coli* ATCC 25922 and *S. enterica* U822s were the most susceptible among the strains tested, so it seems that Gram-negative bacteria were more sensitive in comparison with Gram-positive bacteria, a result similar to other researches (Kosakowska et al., 2021), but

Table 3. Major compounds (%) of oregano essential oil before and after microencapsulation under the best spray drying conditions

Compound	LRI	Before spray drying	After spray drying	Change %
α-Pinene	939	2.5 ^a	2.2 ^b	-0.3
Camphene	954	0.2 ^a	0.2 ^a	0.0
β-Pinene	979	0.9 ^a	0.8 ^a	-0.1
Myrcene	991	4.3 ^a	3.9 ^b	-0.4
δ-3-Carene	1,011	0.1 ^a	0.1 ^a	0.0
α-Terpinene	1,017	1.8 ^a	1.6 ^b	-0.2
<i>p</i> -Cymene	1,025	29.3 ^a	24.8 ^b	-4.5
Limonene	1,029	2.6 ^a	2.4 ^b	-0.2
γ-Terpinene	1,060	6.9 ^a	6.6 ^b	-0.3
Linalool	1,097	0.1 ^a	0.2 ^b	0.1
Nonanal	1,101	0.3 ^a	0.3 ^a	0.0
Decanal	1,202	0.5 ^a	0.6 ^b	0.1
Bornyl acetate	1,287	0.1 ^a	0.1 ^a	0.0
Carvacrol	1,299	29.3 ^a	36.3 ^b	7.0
α-Copaene	1,377	0.4 ^a	0.4 ^a	0.0
β-Elemene	1,391	0.4 ^a	0.4 ^a	0.0
(<i>E</i>)-Caryophyllene	1,419	18.5 ^a	17.5 ^a	-1.0
α-Humulene	1,455	1.7 ^a	1.6 ^a	-0.1
β-Bisabolene	1,506	0.1 ^a	0.1 ^a	0.0

LRI: lineal retention index. Different letters indicate statistical difference at $P \leq 0.05$.



Table 4. Minimum inhibitory concentration of microencapsulated oregano essential oil

Microorganism	Minimum inhibitory concentration (mg L ⁻¹)	
	Essential oil	Microencapsulated essential oil
<i>Salmonella enterica</i> U822s	0.1	0.5
<i>Staphylococcus aureus</i> ATCC 25923	0.1	0.6
<i>Escherichia coli</i> ATCC 25922	0.1	0.5
<i>Listeria monocytogenes</i> ATCC 19115	0.1	0.6
<i>Bacillus cereus</i> ATCC 10876	0.1	0.6

contradictory to those reported with microencapsulated OEO (Beirao Da Costa et al., 2012). The antibacterial activity of OEOs is related to their chemical composition. Essential oil compounds such as carvacrol deplete the intracellular ATP pool by decreasing its synthesis. In general, the antibacterial activity of the essential oils of the *Origanum* genus is well described (Teixeira et al., 2013). However, the comparison of activity between OEO and microencapsulated OEO is more limited. The results of the present study indicated a stronger antimicrobial activity of OEO than microencapsulated OEO, which may be due to the higher content of carvacrol and *p*-cymene, before and after spray-drying (Table 3).

Under the optimal spray drying conditions, the microcapsules exhibited well-formed spherical shapes, with few cavities and dents. This observation agrees with previous reports of spray dried OEO (Baranauskienė et al., 2006; Botrel et al., 2012; Da Costa et al., 2013; Hernández-Hernández et al., 2014). The D_{43} was $8.2 \pm 0.4 \mu\text{m}$, lower than $10 \mu\text{m}$ commonly found in similar products (Jafari et al., 2008). This value was in the $7.55\text{--}18.57 \mu\text{m}$ range for microencapsulated OEO using modified starch:gum arabic:maltodextrin (Botrel et al., 2012) and $6.3\text{--}9.2 \mu\text{m}$ range for microencapsulated OEO using inulin (Da Costa et al., 2013).

4. CONCLUSIONS

Powders of microencapsulated oregano essential oil via spray-drying with maltodextrin:gum arabic (1:2 w/w) can be an interesting ingredient for several formulations in the food industry.

Our findings demonstrated that feed flow rate and inlet air temperature had significant influence on powder yield, moisture content, and essential oil retention. Results obtained in the present study indicate that a low feed flow rate of 0.6 L h^{-1} and a high inlet air temperature of $200 \text{ }^\circ\text{C}$ were the best spray drying conditions for the microencapsulation of oregano essential oil. This procedure is an alternative to produce powders easy to handle and to incorporate in foods, and helps to preserve the antioxidant and antimicrobial activities of the bioactive compounds.

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