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MICROENCAPSULATION OF THE AROMA FROM *CAPSICUM CHINENSE* JACQ. CV. HABANERO

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An aroma distillate with the odour note described as 'fresh Habanero chilli pepper' was obtained from hydrodistillation of the fruit. GC-MS analysis of the volatile constituents from the aroma distillate allowed the identification of 100 compounds, most of them esters followed by aldehydes, alcohols, terpenes, ketones, and acids. Encapsulation process of the aroma distillate by spray drying was optimised using response surface methodology. Independent variables were inlet air temperature (150–200 °C) and carrier (maltodextrin 10 DE and gum arabic in 2:1 ratio) content (10–20% wb), while response variables were powder moisture and volatiles retention. Moisture content of the powder varied inversely proportional to the air temperature, while the volatile retention was directly related. Retention of volatiles in the powder increased when the carrier content increased, while this factor negatively affected moisture content. Based on the optimisation model of the response variables, the powder with the highest flavour quality was obtained with an air inlet temperature of 200 °C and 20% wb carrier content, with 4% moisture content and 88.6% volatiles retention.

Keywords: Habanero chilli pepper, aroma distillate, hydrodistillation, spray drying, optimisation, volatiles retention

Habanero chilli pepper (*Capsicum chinense* Jacq. cv. Habanero) is widely used for culinary purposes due to its characteristic flavour and colour. The Habanero chilli pepper is very aromatic and is one of the hottest peppers in the world. The typical aroma is one of the most attractive properties, representing a quality parameter for the consumer (Sousa et al., 2006; Pino et al., 2007; Menichini et al., 2009).

Although nowadays there is significant production of flavouring materials, those coming from natural raw materials are still important in the manufacture of flavoured products due to numerous reasons, such as the fact that the composition of natural products is often too complex to be reproduced by the combination of synthetic compounds. Besides, the characteristic compounds not always can be synthesised at a competitive price, so the use of natural materials in the manufacture of certain commercial flavours is required (Surburg & Panten, 2006).

Among the numerous isolation techniques for volatile compounds in foodstuffs, hydrodistillation is the simplest and oldest method widely used. During the process, the volatilisation and subsequent collection of a liquid product occurs by condensation on a refrigerant. In a similar manner, the recovered aqueous essences are produced from fruit juice

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concentrates by fractional distillation. The process has since been widely applied to improve the flavour of most fruit concentrates (Reineccius, 2006). However, the aroma distillate has important handling problems due to its liquid state and the instability of the compounds associated with the presence of water. Likewise, there is a constant demand for flavourings in the form of powder, which are utilised more easily in the food industry. Powdered flavourings have many advantages over aqueous extracts, such as their low humidity, which allows their direct use in dry mixes and seasoning, compact packaging, easier handling and transport, and longer shelf life (Phisut, 2012; Shishir & Chen, 2017).

Thus, the aims of this research were to isolate the volatile compounds evaporated off from Habanero chilli pepper and recovered in the water phase during hydrodistillation, and the preservation of the isolated compounds in the form of microcapsules, which could be used as an ingredient in food processing.

1. Materials and methods

Freshly harvested Habanero chilli peppers, grown at Yucatan, Mexico, were purchased from a local retail market. Fruits were selected at ripe mature stage, when the whole fruit colour turned from green to orange. Carriers used for microencapsulation were maltodextrin (MD) 10 DE (Industrializadora de Maíz S.A. de C.V., Guadalajara, Mexico) and gum arabic (GA) from *Acacia senegal* (Industria Ragar, S.A. de C.V., Mexico City, Mexico).

Ripe chilli peppers (400 g) were homogenised with the same amount of 5% wb sodium chloride solution in a commercial blender for 5 min. The mixture was submitted to hydrodistillation in a flask attached to a Vigreux fractionation column (40 cm × 3 cm) and a condenser at 8 °C. The collecting flask was immersed in an ice bath. The process was maintained until 30% of the original mixture was distilled. Subsequently, the aroma distillate was collected and stored at 4 °C for further experiments. This procedure was evaluated in preliminary studies and used in all experiments.

Aqueous solutions of MD and GA, in 1:2 weight ratio, were prepared by dispersing both materials in 200 g of aroma distillate using a blender. The amounts of MD-GA given to the aroma distillate were 20, 30, and 40 g to provide carrier content of 10, 15, and 20% wb in the feed mixture. The feed mixtures were dried in a spray dryer SD-05 (LabPlant, Huddersfield, England) with a 0.5 mm diameter nozzle. The mixture was fed into the main chamber through a peristaltic pump at a feeding rate of 0.6 l h^{-1}. Drying air flow rate was 63 m^3 h^{-1} and compressor air pressure was 0.4 MPa. Inlet air temperatures used were 150, 175, and 200 °C, while outlet air temperatures were between 70 and 90 °C. The powders were stored in airtight polyethylene bags in a glass desiccator at 20 °C until analysis.

Moisture content of the powders was measured with an automatic digital moisture balance, CRODE (Merida, Yucatan, Mexico).

The volatiles were extracted by HS-SPME using conditions adapted from a previous report (Cuevas-Glory et al., 2015). A sample of 0.5 g of aroma distillate or powder was placed in a 15-ml vial with a silicone septum. A PDMS/DVB/CAR 50/30 (Supelco, Bellefonte, CA, USA) was exposed to the headspace for 45 min at 55 °C, with a previous equilibrium time of 15 min. The samples were stirred magnetically (100 min^{-1}) during the extraction. For determination of the volatiles retention, dispersions of an equivalent amount of aroma distillate (0.450, 0.425, and 0.400 g, respectively), mixture of MD-GA in 1:2 weight ratio (0.050, 0.075, and 0.100 g, respectively) and Milli-Q water (6.55, 6.57, and 6.60 ml,

respectively), representing 10, 15, and 20% wb feedstock, were prepared. The fibre was introduced to the injection port of the gas chromatograph, and the volatile compounds were desorbed in splitless mode at 250 °C for 2 min. After this time, the fibre was left in the injector for 10 min. Analyses were carried out in triplicate.

Analyses were performed in a gas chromatograph coupled to a mass detector Shimadzu GC 2100 Ultra (Kyoto, Japan) in EIMS mode. A DB-5 ms column (30 m × 0.25 mm i.d. × 0.25 μm; J & W Scientific, Folsom, CA, USA) was used. Helium carrier gas flow rate was 1 ml min^{-1}. The oven temperature program was 50 °C for 2 min, 50–240 °C at 4 °C min^{-1}, and 240 °C for 5 min. EIMS, electron energy, 70 eV; ion source and connecting parts temperature, 250 °C. Acquisition was made in scanning mode (35–400 m/z). The identification of compounds was carried out by their linear retention indices and their mass spectra. The linear indices of retention were calculated by means of linear relative interpolation to the retention times of *n*-alkanes C_5–C_{24}, and were compared with the standards and data of the literature (Adams, 2007). Mass spectra were compared with corresponding reference standard data reported and mass spectra from NIST 05, Wiley 6, NBS 75 k, and in-house Flavorlib libraries. The constituents of the aroma distillate were quantified after the areas of each detected compound were normalised and expressed as a percentage area. Process optimisation for the spray drying and volatiles retention was done by using the total area of the GC-MS chromatogram. The volatiles retention was calculated as the ratio between the initial content of total volatile compounds present in the microcapsules and the total volatiles content in the aroma distillate used to produce them. The total volatiles content in aroma distillate and microcapsules was determined using the procedure described previously.

Sensory evaluation was carried out with a panel constituted of five trained judges in evaluation of foods and spice essential oils. The microencapsulated powder obtained at the optimal parameters dissolved in water to 2% wb and the aroma distillate were placed in amber glass flasks just before evaluation. Aroma was characterised with free descriptor terms as described earlier (Rodríguez-Burruezo et al., 2010). Six adequate descriptors were then selected to profile the overall aroma: pepper, green, sweet, fruity, spicy, and woody. Additionally, the intensity of each descriptor was judged on a five-point scale (0=very weak, 1=weak, 2=moderate, 3=strong, 4=very strong).

Process optimisation for the spray drying of chilli pepper was performed using response surface methodology (RSM). Independent variables were inlet air temperature and carrier content, while volatiles retention and moisture were the response variables. A three-level factorial design model by Design-Expert version 8 (Stat-Ease Inc., Minneapolis, MN) generated the 12 experimental runs. The objective of the RSM optimisation was to find the optimum conditions of the microencapsulation or to determine the region of the space of the factors, in which the values of certain desired characteristics are satisfied (Montgomery, 2013).

2. Results and discussion

As result of the sensory evaluation, the aroma of the hydrodistillated extract was described as ‘fresh Habanero chilli pepper aroma’, which indicates that the important volatile compounds of the characteristic aroma of Habanero chilli pepper were recovered in the hydrodistillate. GC-MS analysis of the volatile constituents from the aroma distillate allowed the identification of 100 compounds (Table 1). Most of the compounds identified were esters, followed by aldehydes, alcohols, terpenes, ketones, and acids. Major compounds were (*Z*)-3-hexenyl

3-methylbutanoate, hexyl 3-methylbutanoate, hexyl pentanoate, 3,3-dimethylcyclohexanol, and (*E*)-2-hexenal, which have been found in earlier studies (Pino et al., 2011, Cuevas-Glory et al., 2015) and are important aroma-active compounds in this species (Gahungu et al., 2011; Pino et al., 2011).

Table 1. Volatile constituents of Habanero chilli pepper aroma distillate

Compound	LRI	Area %
Ethanol	537	0.1
(*E*)-2-Butenal	624	0.2
3-Methylbutanal	654	tr
2-Methylbutanal	658	tr
1-Penten-3-one	678	tr
2-Ethylfuran	707	tr
3-Methylbutan-1-ol	738	tr
1-Methyl-1*H*-pyrrole	743	0.1
2-Methylpentan-3-one	748	tr
(*E*)-2-Pentenal	753	0.1
Methyl 3-methylbutanoate	765	tr
Methyl 2-methylbutanoate	772	tr
2-Methylthiophene	775	tr
Hexan-2-one	792	tr
Hexanal	802	1.1
3-Methyl-2-butyl acetate	832	tr
4-Methylpentan-1-ol	838	0.2
(*Z*)-2-Hexenal, diethyl acetal	841	0.1
(*E*)-2-Hexenal	856	5.7
(*Z*)-3-Hexen-1-ol	859	0.4
(*E*)-2-Hexen-1-ol	862	0.4
Hexan-1-ol	871	0.6
Heptan-2-one	892	0.1
Heptanal	902	tr
2-Methylpropyl 2-methylpropanoate	913	tr
Anisole	918	0.2
(*E*)-2-Heptenal	958	tr
Benzaldehyde	961	tr
Heptan-1-ol	967	0.2
1-Octen-3-one	980	tr
6-Methyl-5-hepten-2-one	986	tr
Octanal	998	0.1
2-Methylpropyl 3-methylbutanoate	1006	0.2
δ-3-Carene	1011	0.1

Table 1 (continued)

Compound	LRI	Area %
3-Methylbutyl 2-methylpropanoate	1013	tr
2-Methylbutyl 2-methylpropanoate	1017	tr
Limonene	1027	tr
2-Ethylhexan-1-ol	1032	0.1
Phenylacetaldehyde	1042	0.1
Butyl 3-methylbutanoate	1047	tr
Pentyl 2-methylpropanoate	1058	tr
3-Methylbutyl 3-methylbutanoate	1104	0.1
Octan-1-ol	1068	0.1
cis-Linalool oxide (furanoid form)	1075	tr
Methyl benzoate	1091	0.1
Linalool	1096	1.3
Nonanal	1101	0.3
3-Methylbutyl 3-methylbutanoate	1104	1.2
2-Methylbutyl 2-methylbutanoate	1107	0.2
Pentyl 2-methylbutanoate	1142	0.2
Pentyl 3-methylbutanoate	1150	1.5
Hexyl 2-methylpropanoate	1152	0.5
3-Methyl-3-butenyl 3-methylbutanoate	1154	1.4
Methyl 2-methyloctanoate	1158	tr
(*E,Z*)-2,6-Nonadien-1-ol	1160	0.1
(*E*)-2-Nonenal	1163	0.4
2-Methoxy-3-(2-methylpropyl)-pyrazine	1183	0.5
Decan-2-ol	1186	0.6
α-Terpineol	1189	tr
Methyl salicylate	1194	2.3
Hexyl 2-methylbutanoate	1236	3.4
Hexyl 3-methylbutanoate	1244	14.9
3-Methylbutyl hexanoate	1260	0.1
Heptyl 2-methylpropanoate	1300	0.1
(*Z*)-3-Hexenyl 2-methylbutanoate	1293	tr
(*Z*)-3-Hexenyl 3-methylbutanoate	1295	19.5
Hexyl pentanoate	1298	12.8
(*E*)-2-Hexenyl 3-methylbutanoate	1299	3.0
9-Decanolide	1308	0.3
Hexyl tiglate	1328	0.1
Heptyl 2-methylbutanoate	1333	0.7
Heptyl 3-methylbutanoate	1338	3.5
(*Z*)-3-Hexenyl hexanoate	1380	0.2
Hexyl hexanoate	1382	0.6
α-Copaene	1384	tr
2,3-Dimethylcyclohexanol	1389	1.7

Table 1 (continued)

Compound	LRI	Area %
3,3-Dimethylcyclohexanol	1392	7.8
β-Cubebene	1394	0.2
Benzyl 3-methylbutanoate	1396	1.5
Dodecanal	1420	tr
(*E*)-α-Ionone	1431	0.4
Octyl 2-methylbutanoate	1438	0.4
Octyl 3-methylbutanoate	1440	0.5
γ-Himachalene	1483	1.3
(*E*)-β-Ionone	1489	1.3
n-Pentadecane	1500	0.3
δ-Cadinene	1523	0.1
γ-Dehydro-*ar*-himachalene	1532	tr
trans-Cadina-1(2),4-diene	1535	tr
α-Calacorene	1546	tr
Dodecanoic acid	1564	0.1
(*Z*)-3-Hexenyl benzoate	1567	0.2
Hexyl benzoate	1576	0.2
Phenylacetic acid	1579	0.1
n-Hexadecane	1600	0.1
Tetradecanoic acid	1760	0.3
Benzyl benzoate	1763	tr
Pentadecanoic acid	1868	0.1
(*Z*)-9-Hexadecenoic acid	1938	0.2
Hexadecanoic acid	1960	0.8

LRI: Lineal retention index in DB-5ms; tr: <0.1%

The microencapsulation of the aroma distillate was studied by response surface design (Table 2). The moisture of powders varied from 4 to 6%, which is similar to those reported in several studies of fruit spray drying (Sarabandi et al., 2017; Thirugnanasambandham & Sivakumar, 2017). Factor inlet air temperature had the major effect on moisture content (Table 3). The coefficient of the first order term with the coded variable showed that moisture content decreased with the increase of inlet air temperature. The negative sign of the coefficient of the first order term of this variable indicates that moisture content of the powder decreased when inlet air temperature increased.

When high inlet air temperature was used with the feed matrix, it led to higher efficiency of heat and mass transfer, and therefore, higher evaporation rate to evaporate moisture from the droplets (Shishir & Chen, 2017). The moisture content reveals a reduction with an increase in carrier content (Table 3). This behaviour could be explained by the fact that additional amount of biopolymer resulted in an increase in feed solids and a reduction in total moisture.

Table 2. Experimental conditions and response values of the response surface design

Inlet air temperature (°C)	Carrier (%)	Moisture (% wb)	Volatiles retention (%)
150	10	5.94	69.9
150	15	5.80	74.3
150	20	6.00	79.0
175	10	6.00	72.4
175	15	5.94	75.8
175	15	5.98	77.3
175	15	5.94	78.1
175	15	6.00	78.7
175	20	5.00	85.1
200	10	4.90	83.2
200	15	5.00	85.3
200	20	4.00	87.5

Table 3. Main effects and interactions of the response surface models

Regression coefficient	Moisture	Volatiles retention
Intercept	5.92	77.67
X_T	–0.64**	5.47**
X_C	–0.31*	4.35**
X_T^2	–0.43*	1.74
X_C^2	–0.33	0.69
X_TX_C	–0.24	–1.20
R^2	0.929	0.946
Model (P-value)	0.001	0.001
Lack of fit (P-value)	0.065	0.215

X_T: Inlet air temperature; X_C: Carrier content.
*Significant at P<0.05; **Significant at P<0.001

Volatiles retention was between 69.9–87.7% (Table 2). Throughout drying, some volatiles losses are inevitable, since a part of the volatiles are evaporated from the surface of the drop at a faster rate than water during the constant drying stage. However, the content of volatile compounds in the powders was similar to the level of volatile compounds that are retained in some spray-dried fruit products (Komes et al., 2007).

Volatiles retention was directly proportional to the main effect of inlet air temperature and carrier content (Table 3). These results could be explained by the fact that increasing the air temperature increases the speed of film formation in the droplets, which favours the retention of volatile compounds (Reineccius et al., 1982). On the other hand, the positive effect of the carrier content on the retention of volatile compounds may be due to the high rate of encapsulation by the increase of biopolymer content present in the feed mixture, which reduces the loss of volatile compounds during drying (Reineccius et al., 1982).

The response surface plot obtained for the optimisation of moisture and volatiles retention shows that the best responses can be obtained with the inlet air temperatures close to the highest temperature studied (199.8 °C) and highest carrier content (19.9% wb). The desirability function at this point was 1.0, which is the maximum goal. With these processing conditions, the powder was estimated to have 4.0% wb moisture content and 88.6% volatiles retention. Additional drying trials with an inlet air temperature higher than 200 °C and carrier content higher than 2% produced powders with lower typical Habanero chilli pepper aroma.

Odour is an important sensory quality attribute of foods that can be affected by different factors during the microencapsulation process. After reconstituting the microencapsulated powder in distilled water, it was found that the sample obtained with the optimised parameters has the odour descriptors not less than that of the original distillate counterpart in aroma (Fig.1). Obviously, the drying air temperature and carrier content had influence on the product quality, but the Habanero chilli pepper added with MD-GA at 20% wb and dried at 200 °C achieved the highest overall liking score. No significant differences were found (P=0.09) in the intensity of the descriptors of the Habanero pepper smell between the aroma of the distillate and its encapsulated powder. This result shows that volatile compounds responsible for the aroma in the aqueous phase were successfully trapped in the microcapsules.

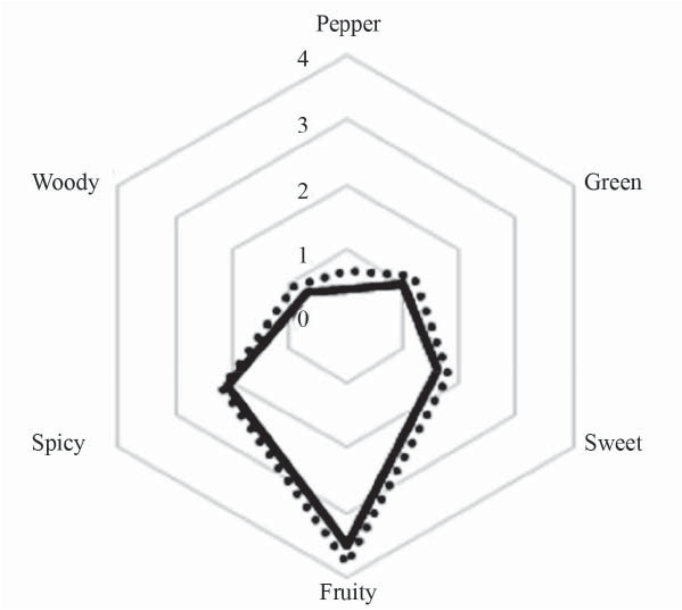

Fig. 1. Odour profiles of Habanero chilli pepper aroma distillate and power obtained with optimal parameters
·····: Aroma distillate; ——: Powder

3. Conclusions

The Habanero chilli pepper powder made by spray drying of an aroma distillate combined with 20% wb maltodextrin and gum arabic (2:1) and at 200 °C inlet air temperature achieved the highest flavour quality. Multiple response optimisation indicated that such processing conditions were predicted to provide 4.0% wb powder moisture content and 88.6% volatiles retention. These results are useful for the fruit powder producers and researchers.

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