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The effect of different wall materials on the production of suppressed-pungent capsaicin microparticles

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Abstract

The aim of this study was to investigate the effects of wall materials types on the production of double-layered and suppressed-pungent capsaicin microparticles via spray chilling method. For this purpose, palm oil and different proteins (gelatin, sodium caseinate and whey protein) were used as wall materials, while soy lecithin was selected as stabilizer. Sample encapsulated only with palm oil (single-layered) was used as control. Centrifuge stability and kinetic stability were analyzed on the prepared emulsions. Total and surface capsaicin, microencapsulation efficiency, melting point temperature and fusion enthalpy analysis were carried out on the capsaicin microparticles obtained by spray chilling.

Keywords: *Capsaicin; Spray chilling method; Pungency; Double-layered microparticles*

1. Introduction

Red hot chili peppers from plants of the Capsicum genus in the Solanaceae family are the pungent and carmine fruits generally used as spice to give pungent or hot sensation to many dishes. Capsaicinoids, the group of secondary metabolites, are responsible for the pungency of chili peppers. The main compound of the capsaicinioids is capsaicin (8-methyl-N-vanillyl-6-nonenamide) and it has a high bioavailability with a lot of benefits on human health especially on the cancerous cells. In addition, it can relieve neuropathic pains and is used in symptomatic therapy of arthritis, muscle and join pains. Conversely to its advantages, capsaicin can cause irritation on the human digestive system based on overconsumption. It can induce burning sensation on the mouth, alimentary canal and stomach of people who are more sensitive to pungency. Therefore, microencapsulation of capsaicin with an appropriate method and suppressing of its pungency via the relevant microencapsulation method is important for taking advantages of capsaicin and reducing its negative effects on the human health [1]. Spray chilling method is an alternative technique for microencapsulation of functional components because it serves rapid, easy-to-use and relatively cheap process [2]. Spray chilling technology, which uses oil-based substances as wall materials, has been used increasingly day by day in different industries such as pharmaceutical and food. The objective of the present study was to produce suppressed-pungent capsaicin microparticles by spray chilling method and to investigate the effects of wall materials type on the emulsion stability, physical properties and microencapsulation efficiency of capsaicin microparticles.

2. Materials and Methods

2.1. Materials

Capsaicin (66.7%) (Xian Sobeo Biotech, China) was used as core material. Ethanol (Sigma-Aldrich, Germany) used for dispersing capsaicin. Hydrogenated palm oil was supplied by Felda Iffco (Izmir, Turkey), while refined sunflower seed oil, whey protein isolate, gelatin, sodium caseinate and soy lecithin were obtained by a local market.

2.2. Emulsion preparation

Capsaicin was dissolved in ethanol (1:1) using ultrasonic bath for 10 min. Then, the capsaicin solution was mixed with palm oil containing soy lecithin as a stabilizer. . This oily capsaicin mixture was transferred into aqueous protein solution drop by drop under Ultra Turrax homogenizer, worked at 10000 rpm for 3 min. The final mixture was then added into molten palm oil (in circulation water bath at 65°C) and homogenized again at 10000 rpm for 3 min. For control samples, with or without stabilizer, molten palm oil was mixed with ethanolic capsaicin solution and homogenized at the same conditions. Palm oil, gelatin, Na-Caseinate and whey protein isolate were used as wall materials while soy lecithin was used as stabilizer.

The composition of emulsions was given in Table 1, where the concentrations are given as ratios of components in total 200 g final mixture.

Table 1. The percent composition of emulsions

Exp. No	Palm oil	WPI	Na-Cas	Gelatin	Soy Lecithin	Sunflower oil	Ethanol	Water	Capsaicin
1	90.5	-	-	1.50	0.50	0.90	0.30	6	0.30
2	90.5	-	1.50	-	0.50	0.90	0.30	6	0.30
3	90.5	1.50	-	-	0.50	0.90	0.30	6	0.30
4	90.5	-	0.75	0.75	0.50	0.90	0.30	6	0.30
5	90.5	0.75	-	0.75	0.50	0.90	0.30	6	0.30
6	90.5	0.75	0.75	-	0.50	0.90	0.30	6	0.30
7	90.5	0.50	0.50	0.50	0.50	0.90	0.30	6	0.30
8	98.9	-	-	-	0.50	-	0.30	0	0.30
9	99.4	-	-	-	-	-	0.30	0	0.30

2.3. Emulsion stability

Emulsion stability of capsaicin emulsions was evaluated with kinetic and centrifuge stability analysis. In order to analyze the kinetic stability of obtained emulsions, 10 ml of each emulsion was transferred to a test tube and kept in water bath at 65°C for 4 hours. The volume of lower phase measured after 2 hours. The kinetic stability of the emulsions was calculated as sedimentation index via following equation (1):

$$SI = (H_{l@t}/H_i) \times 100 \quad (1)$$

where SI, $H_{l@t}$ and H_i indicate sedimentation index, lower phase height at *t* time and initial height of emulsion, respectively [3].

The centrifuge stability of samples was measured as follow; 10 mL of emulsion was immediately poured into graduated centrifuge tube and centrifuged at 10000 rpm for 10 minutes. Then the centrifuge stability of the emulsion was calculated by equation (2):

$$CS = (H_l/H_i) \times 100 \quad (2)$$

where CS, H_l and H_i indicate centrifuge stability, height of lower phase and initial height of emulsion, respectively [4].

2.4. Spray chilling

Capsaicin emulsions fed into the spray chilling system in order to obtain solid lipid capsaicin microparticles. Spray chilling process was carried out by using Bakon-B15 (Izmir, Turkey).

The inlet temperature of cooling air (10°C), the temperature of nozzle (60°C), the feeding temperature (65°C) and air flow rate (10L/min) were kept constant during spray chilling.

2.4. Melting point temperature (T_m) and fusion enthalpy (ΔH)

T_m and ΔH of capsaicin microparticles were determined by using DSC (Perkin-Elmer DSC 6000, Turkey). For this purpose, 10 mg of capsaicin microparticles was weighed directly into the DSC aluminum sample pan and sealed with a lid. The purge gas was nitrogen (50mL/min) and the temperature ranged from 25 to 90°C with a heating rate of 15°C/min. The data were then processed using Pyris Manager Software (Perkin-Elmer).

2.5. Powder yield

The powder yield was defined as the ratio between the total collected powder and theoretical powder quantity from the sprayed solution and calculated as shown in equation (3).

$$Powder\ yield\ (\%) = \frac{Powdered\ capsaicin\ microparticles(g)}{Feeding\ emulsion\ (g)} \times 100 \quad (3)$$

2.6. Total and surface capsaicin content and microencapsulation efficiency (ME)

The total and surface capsaicin contents of the microparticles were determined using high performance liquid chromatography (Shimadzu Corporation, Kyoto, Japan) according to Consoli et al. [3] with some modifications. The chromatography conditions were: 60°C column temperature, 5 µl sample volume, 1.5 ml/min flow rate, acetonitrile:water (50:50) mobile phase and 222 nm with UV-detector. Microencapsulation efficiency was calculated by the following equation (4).

$$ME\ (\%) = (TC - SC/EC) \times 100 \quad (4)$$

where ME, TC, SC and EC indicate microencapsulation efficiency, total capsaicin amount of microparticles, surface capsaicin amount of microparticles and total capsaicin amount in emulsion, respectively.

2.7. Statistical analysis

All measurements were performed in triplicate. Results are expressed as mean ± standard deviations. All results were analyzed using SPSS version 15.0 Windows program (SPSS Inc., Chicago, IL). To observe significant differences between the samples, paired t-test was used at a 5% significance level.

3. Results and Discussions

3.1. Emulsion stability

The emulsions are thermodynamically unstable systems. Therefore, protein based emulsifiers are mostly used to improve the stability of emulsions. These emulsifiers vary widely in their ability to form and stabilize emulsions depending on their molecular and physicochemical

characteristics [5]. The emulsion stabilities of capsaicin emulsions (emulsion number 1-7) prepared with different wall materials were evaluated as kinetic and centrifuge stability. The stability analysis for control samples (emulsion number 8 and 9) were not performed due to being a dispersion not an emulsion.

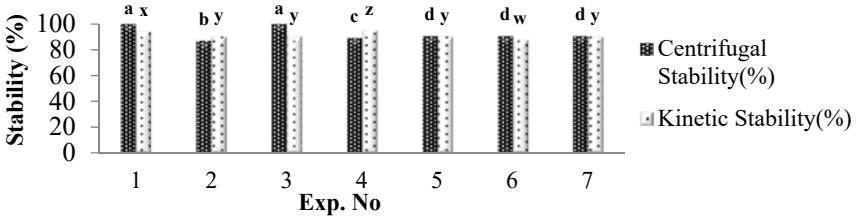

Fig. 1 Kinetic stability and centrifugal stability results of capsaicin loaded emulsions

The emulsion stability study revealed that most of the emulsions were kinetically (87.5-100%) and centrifugally (86.75-100%) stable as shown in Fig 1. Consoli et al. [5] also found similar stability results for gallic acid encapsulation with spray chilling method. The emulsion prepared with gelatin was found to be the most stable kinetically while adding Na-Caseinate and whey protein isolate to emulsions resulted in a decrease in kinetic stability. Gelatin has good water holding capacity because the small pore size of the gel network tightly holds water molecules by capillary forces [5]. Therefore, the separation of aqueous phase was prevented in capsaicin emulsions. The results of centrifugal stability of emulsions showed that emulsions prepared with whey protein and gelatin had the highest stability values. Whey proteins are much more rigid as compared to caseins [6]. Therefore, high concentrations of sodium caseinate resulted in low stabilities of emulsions as reported previously [7].

3.2. T_m and ΔH

The melting point temperature (T_m) and fusion enthalpy (ΔH) of encapsulated capsaicin microparticles were given in Table 2. Adding whey protein into emulsions as wall material resulted in an increase T_m of the capsaicin particles, while gelatin-containing microparticles had a lower melting point temperature. On the other hand, Na-caseinate did not have a significant effect on the melting point of the microparticles, but it caused higher melting point than that of gelatin-containing microcapsules. As shown in Table 2, melting points of double-layered samples (except 3th sample) were calculated lower than that of the single layered sample. It was probably due to the creation of the less ordered structure of the inner structure of the lipid matrix and/or the small size effect which could be explained by Gibbs-Thomson equations [8]. Wang et al. [9] reported that microencapsulation of capsaicin decreased the melting temperature. The capsaicin, nanocapsulated with single coacervation in that study [10], melted at 75°C (T_m) and absorbed 160.7 J/g energy. In our study, capsaicin was encapsulated with gelatin (single layered) and melting point temperature and absorbing

energy were calculated as 100°C and 199.9 J/g, respectively. Differences between T_m and ΔH values of the previous and the present study can be resulted from the encapsulation form (single or double layered) and encapsulation technique.

3.3. Product yield

The high product yield is one of the advantages of the spray chilling. The product yield values of the samples obtained from spray chilling were reported as over 75% [11]. Product yield of the capsaicin microparticles obtained from spray chilling method in the present study agreed with the data in the literature. Encapsulation form (single or double layered) did not significantly affect the product yield results and the results were in the range of 77-87%. The samples obtained from the emulsions added by whey protein and gelatin had the higher product yields as compared to that of containing Na-caseinate. The lower product yield value of the samples added by Na-caseinate could be resulted from viscoelastic properties and high gelling power of the Na-caseinate. Therefore, caseinate containing emulsions could stick to drying chamber in spray chilling equipment due to its adhesive structure [12].

3.4. Total and surface capsaicin contents and microencapsulation efficiency (ME)

Total and surface capsaicin contents and microencapsulation efficiency results of the capsaicin microparticles were shown in Table 2. The results showed that capsaicin could be effectively encapsulated with the oil-based wall materials and the amount of surface capsaicin could be reduced. In terms of surface capsaicin content, the microparticles had relatively less amounts of capsaicin on their surfaces. Maximum surface capsaicin content was calculated as 2.08 ppm for single layered (only palm oil) encapsulated sample. On the other hand, double layered (palm oil-soy lecithin-whey protein) encapsulated sample showed the best encapsulation efficiency with the surface capsaicin content of 2.04 ppm. Microencapsulation efficiency of the emulsions increased with an increase in whey protein concentration in the emulsions. In addition, microencapsulation efficiency of the emulsions containing gelatin was higher than that of containing Na-Caseinate. This could be attributed to the viscoelastic properties and gelling power of casein. The maximum microencapsulation efficiency of 99.73% was determined for double-layered (palm oil-whey protein) encapsulated sample, while the minimum result was calculated as 80.48% for the samples produced by single-layered (only palm oil) encapsulation. The results showed that the melting point temperatures significantly affected the microencapsulation efficiency of the samples. The microencapsulation efficiency increased with the increasing melting point. In the previous study with ascorbic acid encapsulation by spray chilling [13], palm and palm seed oil (43°C melting point) were used as wall material while soya lecithin was used as stabilizer. The encapsulation efficiency values of the samples varied from 68.2 to 72.5%. Differences in the encapsulation efficiency results of the previous study and the present study can be resulted from the double-layered microencapsulation used in our study.

Table 2. Total and surface capsaicin, microencapsulation efficiency and thermal characteristics of the capsaicin microcapsules

Exp. No	TC (ppm)	SC (ppm)	ME (%)	T_m (ºC)	ΔH (J/g)
1	$39.87\pm0.27^{c,d}$	2.04 ± 0.15^{c}	$94.57\pm0.30^{c,d}$	48.81 ± 0.09^{a}	$85.13\pm0.48^{c,d}$
2	$35.76\pm2.62^{a,b}$	$1.92\pm0.01^{b,c}$	$84.61\pm6.52^{a,b}$	49.40 ± 1.01^{a}	$85.17\pm2.91^{b,c}$
3	41.93 ± 0.62^{d}	2.04 ± 0.06^{c}	99.73 ± 1.68^{d}	50.05 ± 0.16^{a}	$83.48\pm3.75^{a,b,c}$
4	$38.65\pm0.02^{b,c,d}$	$1.71\pm0.02^{a,b}$	$92.36\pm0.08^{b,c,d}$	49.06 ± 0.34^{a}	79.13 ± 4.44^{a}
5	40.75 ± 0.34^{d}	2.04 ± 0.08^{c}	$96.78\pm1.04^{c,d}$	48.88 ± 0.32^{a}	$83.67\pm2.47^{b,c,d}$
6	41.53 ± 0.27^{d}	$1.93\pm0.02^{b,c}$	99.01 ± 0.72^{d}	48.80 ± 1.08^{a}	92.54 ± 4.21^{d}
7	$40.23\pm0.09^{c,d}$	2.05 ± 0.08^{c}	$95.44\pm0.41^{c,d}$	48.65 ± 0.10^{a}	$80.25\pm4.28^{a,b}$
8	$37.31\pm0.76^{a,b,c}$	1.53 ± 0.05^{a}	$89.45\pm1.77^{b,c}$	48.53 ± 0.02^{a}	$85.95\pm2.90^{a,b,c}$
9	34.27 ± 0.07^{a}	2.08 ± 0.07^{c}	80.48 ± 0.02^{a}	49.54 ± 0.85^{a}	$84.76\pm2.77^{b,c}$

Different letters (a, b, c or d) above the columns indicate significant difference between the emulsion formulation

4. Conclusions

Capsaicin has a high bioavailability with a lot of benefits on human health. However, it is inconsumable for some people because of irritating effect. Therefore, it can be microencapsulated to suppress its pungency. In the present study, capsaicin is effectively microencapsulated by spray chilling technique by using palm oil and whey protein (as wall materials) and soy lecithin (as stabilizer). The encapsulation efficiencies of the whey protein based emulsions were higher than that of the other emulsions. Furthermore, the sensible pungency of capsaicin can be reduced from 40 ppm to 1.71 ppm (the minimum surface capsaicin content) with double-layered microencapsulation by spray chilling. By this way, capsaicin was converted to a suppressed-pungent component that can be consumed by sensitive people.

5. Nomenclature

DSC	differential scanning calorimeter
min	minutes
μm	micrometer
rpm	revolutions per minute

6. Acknowledgments

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