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Effects of spray drying process conditions on the quality properties of microencapsulated cream powder

Ahsen Burcin Himmetagaoglu [a, b], Zafer Erbay [b, *]

[a] Department of Gastronomy and Culinary Arts, Faculty of Tourism, Alanya Hamdullah Emin Paşa University, Antalya, Turkey
[b] Department of Food Engineering, Faculty of Engineering, Adana Science and Technology University, Adana, Turkey

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ABSTRACT

Effects of spray drying conditions including inlet drying temperature (150, 170 or 190 °C), feed flow rate (9.0, 19.5 or 30 mL min^{-1}), and aspiration rate (50, 75 or 100%) on the composition, water activity, reconstitution, density, colour, morphology, surface fat and free fatty acid content of microencapsulated cream powder were evaluated. The variation in the wettability, bulk and tapped densities, surface fat and volatile fatty acid contents of the powders showed similar tendencies with the spray drying parameters. In contrast, moisture content, fat content, water activity, solubility, and colour properties were not influenced by the processing conditions. High inlet drying temperature, low or high aspiration rates, and high feed flow rate caused undesirable powder properties. According to the results, processing conditions that cause high drying rates should not be applied for production of a high-quality microencapsulated cream powder.

1. Introduction

An important compound of milk is milk-fat and it is an economical source for manufacturing novel products such as dairy creamers, creamy condiments, and desserts (Danviriyakul, McClements, Decker, Nawar, & Chinachoti, 2002). There is a significant worldwide butter surplus that has stemmed from the change in dietary habits and the trend to remove fat from consumer products. As a result, storage of milk fat has gained importance. Although butter can be stored for about 4 weeks at room temperature, it is possible to increase the storage time, to decrease the storage costs and to increase the use of milk fat by converting it into a powder form (Danviriyakul et al., 2002; Onwulata, Smith, Cooke, & Holsinger, 1996; Onwulata, Smith, Craig, & Holsinger, 1994). Consequently, the largest production increases in dairy products were seen in the butter and dairy powder categories (IDF, 2015).

In the 1960s, powdered products became popular due to their convenience (Havea, Baldwin, & Carr, 2009). Cream powder that has a fat content higher than 42% (CODEX, 2011) is frequently used in dairy and food manufacturing (e.g., ice cream, cheese, infant formula, evaporated milk, sweetened condensed milk). It also functions as an ingredient in bakery products, soups, sauces, processed meats, convenience foods, snacks, seasoning and dressings, creamy condiments, coffee and tea whiteners (Chandan, 2011; Danviriyakul et al., 2002; Farkye, 2006; Havea et al., 2009; Rowe & Donaghy, 2011; Smiddy, Kelly, & Huppertz, 2009; Spreer, 1998). In recent years, the production of cream powders in industry has increased and the trade of milk fat in the powder form increased internationally (Havea et al., 2009).

Although powdered dairy foods are characterised by their long shelf life, some detrimental changes such as fat oxidation, lactose crystallisation, non-enzymatic browning, and/or particle caking that modify powder physical/functional properties may occur during storage, even in such a strongly dehydrated foods (Kim, Chen, & Pearce, 2009a,b; Chudy, Pikul, & Rudzinska, 2015; Koca, Erbay, & Kaymak-Ertekin, 2015). While oxidation is one of the most important problems causing deterioration of the shelf life for high-fat powders, non-enzymatic browning also has an important effect on carbohydrate-rich powders. The accumulation of fat on the surface of the powder particle greatly influences the physical properties of powders (reconstitution properties, stickiness, flowability, etc.) and it is one of the most important reasons of oxidation for high-fat powders.

The physical and storage properties of high-fat powders may be improved by milk fat encapsulation achieved by enclosing the fat with a continuous thin film of a wall material that helps to protect the fat from the environment and improve its stability. For milk fat

* Corresponding author. Tel.: +90 332 4550000.
E-mail address: zafererbay@yahoo.com (Z. Erbay).

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encapsulation, different kinds and combinations of wall materials, mainly proteins and/or carbohydrates, at different concentrations have been examined in the literature. It has been reported that casein and casein micelles were suitable for bioactive compounds due to their structural characteristics and encapsulation ability (Ranadheera, Liyanaarachchi, Chandrapala, Dissanayake, & Vasiljevic, 2016). The use of sucrose has been recommended in terms of powder physical properties, whereas the use of lactose was preferred with respect to the taste of the powders as the use of sucrose influenced the sweetness of the final product (Konstance & Onwulata, 1995; Onwulata et al., 1994). Danviriyakul et al. (2002) evaluated the physical properties of milk fat powders produced with different wall materials and reported that better results were obtained with the high dextrose equivalent (DE) carbohydrate. The effects of the combined use of different proteins and carbohydrates as wall materials on the emulsion properties prepared from cream were investigated and the most appropriate wall material formulation was determined to be sodium caseinate and high DE maltodextrin with the protein/wall material ratio of 20% for microencapsulated cream powder production (Himmetagaoglu, Erbay, & Cam, 2018).

While the impact and success of microencapsulation mainly depend on the emulsion preparation and the composition of wall materials used (Foerster, Liu, Gengenbach, Woo, & Selomulya, 2017; Keogh & O'Kennedy, 1999), the fundamental technique in converting emulsion into powder is spray drying. Spray drying is the main technique in dairy powder production offering many advantages such as the continuous/automated process design and enhanced product quality (da Silva, Larsen, Hougaard, & Ipsen, 2017; Erbay, Koca, Kaymak-Ertekin, & Ucuncu, 2015; Ranadheera, Evans, Adams, & Baines, 2015) and the effects of spray drying should be evaluated to obtain a high-quality powder (Vignolles et al., 2009). There are some previous studies related to the effects of spray-drying process parameters on the quality of high-fat dairy powders in the literature (Erbay et al., 2015; Kelly, Kelly, & Harrington, 2002; Koca et al., 2015); however, the information about the effects of the spray drying process conditions on the quality parameters of high-fat powders, especially cream powders, is limited. In this study, microencapsulated cream powders were produced at different spray drying conditions and the effects of the main spray drying process conditions that can be adjusted independently such as inlet drying air temperature, feed flow rate, and aspiration rate on the quality parameters of microencapsulated cream powder were investigated.

2. Materials and methods

2.1. Powder production

The emulsions prepared with 25% total solid content while keeping the wall materials to total solid content at 30% to produce the microencapsulated cream powder with 60% fat content. Maltodextrin (dextrose equivalency of 18) and sodium caseinate wall materials in 20% protein per wall material ratio were used in the emulsion as it was previously found to be the most appropriate emulsion according to its lower creaming index, viscosity, and average fat droplet size values (Himmetagaoglu et al., 2018). In addition, sodium citrate (as a stabiliser), sodium phosphate (as an acidulant), calcium carbonate (as an anti-caking agent), and soy lecithin (as an emulsifier) were used in the formulation.

The emulsions were fed to the spray-dryer at 45 °C and dried by a Mini Spray Dryer B-290 (BUCHI, Flawil, Switzerland). Drying variables were: inlet drying temperature (150, 170, and 190 °C), feed flow rate (9.0, 19.5, and 30.0 mL min^{-1}), and aspiration rate (50, 75, and 100%). Process variable ranges were selected based on literature information (Erbay et al., 2015; Kurozawa, Morassi, Vanzo, Park, & Hubinger, 2009; Onwulata et al., 1994) and by preliminary experimentation. During the preliminary experiments, the aim was to obtain powders without scorched particles and to produce a powder with a moisture content lower than 5%. Experimental design and drying conditions used in this study are shown in Table 1. To investigate the impact of different drying conditions, two of the given variables remained constant at the medium-level condition whereas one variable was changed. Six replications were made using the second production set as the conditions were the centre of the experimental design and three replications were made for all other production sets.

While the powders produced were packed in a multilayer plastic package (polyethylene terephthalate/aluminium foil/low density polyethylene) and stored at −18 °C for scanning electron microscopy (SEM) and free fatty acid (FFA) analyses, the remaining samples were kept at room temperature in an air-tight oriented polypropylene package and physical property determinations made within a week after production.

Table 1
Spray drying conditions used in the production of microencapsulated cream powders.

Production set	Inlet temperature (°C)	Feed flow rate (mL min^{-1})	Aspiration rate (%)
1	150	19.5	75
2	170	19.5	75
3	190	19.5	75
4	170	9.0	75
5	170	30.0	75
6	170	19.5	50
7	170	19.5	100

2.2. Composition, water activity, and reconstitution properties

Moisture and fat contents of the microencapsulated cream powder samples were determined by gravimetric (AOAC, 2012) and Teichert's (Eissen, Chaudron, & Werdmuller, 1972) methods, respectively. The water activities of the powders were measured at room temperature using a water activity meter, AquaLab Series 3 TE (DecagonDevices, Pullman, WA, USA).

Wettability of microencapsulated cream powder was evaluated based on the method described by Jinapong, Suphantharika, and Jamnong (2008) with further modifications. Approximately 0.1 g of microencapsulated cream powder sample was poured into 100 mL of distilled water at room temperature and the stopwatch was started. The time was recorded for all powder particles to become completely wetted, which was determined visually. The analyses were replicated five times for each powder sample.

Solubility is one of the most important features for overall reconstitution quality and was measured using the method described by Cano-Chauca, Stringheta, Ramos, and Cal-Vidal (2005) with some modifications. Approximately 0.5 g of microencapsulated cream powder and 40 mL of distilled water were transferred into Falcon tubes and blended by an Ultra-Turrax (IKA, T25, Staufen, Germany). The slurry was centrifuged by a benchtop centrifuge (Universal 320 R, Hettich, Tuttlingen, Germany) at 3000 × *g* for 5 min at room temperature. Ten millilitres of the supernatant were transferred from the tubes to pre-weighed aluminium Petri plates and dried at 102 °C until the remaining weight was constant. According to the weight difference, solubility was calculated in percentage.

2.3. Free and surface fat contents

While the free fat extraction was carried out and calculated according to the A/S Niro A 10a method (GEA Niro, 2005), the surface fat was extracted using the methodology described by Kim, Chen, and Pearce (2005) with slight modifications. The content of fat on the microencapsulated cream powder particle surface is defined as the evaporation residue remaining, after the sample has been washed with petroleum ether, filtered and dried. To perform surface fat analysis, approximately 1 g of microencapsulated cream powder was weighed in the filter paper and washed out 4 times with 5 mL of petroleum ether. The filtrate was collected in the pre-weighed aluminium Petri dishes. The dishes were dried in the drying oven at 102 °C until the dishes achieved a constant weight. According to the weight difference, surface fat content (%) was calculated. Free fat and surface fat measurements were made in duplicates.

2.4. Powder densities

The bulk, tapped and particle densities of the microencapsulated cream powders were measured by the methods described in Erbay and Koca (2015). In the particle density determination, a 50 mL metal pycnometer (ZPM3030, Zehntner GmbH, Sissach, Switzerland) was used. The results of the bulk and tapped densities were used in the calculation of Carr Index (CI) and Hausner Ratio (HR) values to evaluate the flowability and cohesiveness of the microencapsulated cream powder samples (Carr, 1965; Hausner, 1967). A HR value lower than 1.25 indicated a free-flowing powder, whereas a value higher than 1.25 referenced poor flowability. The CI results were discussed with the flowability classification explained by Jinapong et al. (2008).

2.5. Scanning electron microscopy

Morphological properties of the microencapsulated cream powders were determined by scanning electron microscopy (SEM; Philips XL30, The Netherlands). Prior to analysis, powders were stuck onto the double-sided adhesive carbon tabs mounted on SEM stubs and coated with gold by using a sputter coater (Polaron SC7610, Quorum Technologies Ltd, East Sussex, UK). Topographic images of the samples were obtained at different magnifications (1000 ×, 2500 ×, and 5000 ×) using an Everhart-Thornley detector.

2.6. Colour evaluation

The colour of the microencapsulated cream powder was quantified by using a colorimeter (Konica Minolta CM-5, Tokyo, Japan). The colour values, L (lightness), a (redness/greenness), and b (yellowness/blueness) for the powder samples were measured and the average value was calculated. For the further examination of the colour values of the microencapsulated cream powders, colour difference (ΔE), chroma, and browning index (BI) values were calculated as follows (Saricoban & Yilmaz, 2010):

$$\Delta E = \sqrt{(L_0 - L)^2 + (a_0 - a)^2 + (b_0 - b)^2} \quad (1)$$

$$Chroma = \sqrt{a^2 + b^2} \quad (2)$$

$$BI = \frac{100 \times \left[\frac{(a + 1.75 \times L)}{(5.645 \times L + a - 3.012 \times b)}\right] - 0.31}{0.17} \quad (3)$$

2.7. Free fatty acid content

The extraction and identification of the free fatty acids (FFAs) in the microencapsulated cream powders were performed with reference to a gas chromatographic method with some modifications (De Jong & Badings, 1990; Deeth, Gerald, & Snow, 1983). For the extraction of FFAs, microencapsulated cream powder samples (1 g) and anhydrous Na_2SO_4 (3 g) were weighed into a glass test tube and 0.3 mL 2.5 M H_2SO_4 added. To quantify the FFAs, 3 mL of solvent (diethyl ether:hexane, 1:1, v/v) and 1 mL of internal standard solution mixture including pentanoic, tetradecanoic, and heptadecanoic acids at a concentration of 0.5 mg mL^{-1} for each were added to the samples. Then, a vortex was used to mix the solution for 1 min, which was then centrifuged at 700 × g for 2 min at room temperature. The supernatant was collected and the extraction procedure was repeated three times to extract the full range of FFAs from the powder. The pooled supernatants were filtered through an aminopropyl solid-phase extraction (SPE) column (Bond Elut, Agilent Technologies, Santa Clara, CA, USA) containing 1 g of deactivated alumina (Sigma–Aldrich Chemie GmbH, Steinheim, Germany) for the isolation of FFAs. Afterward, the alumina was dried, removed and mixed gently with 2 mL of formic acid solution (3%, v/v) using a vortex. Finally, the mixture was centrifuged at 450 × g for 10 min and 0.5 μL of the supernatant solution was injected with a split ratio of 1:10 onto a GC system (7890A, Agilent Technologies, Santa Clara, CA, USA) equipped with a flame ionisation detector (FID). The separation of FFAs was performed with a capillary DB-FFAP column (30 m, 0.25 um, 0.25 mm, Agilent Technologies) and helium used as carrier gas at a constant flow rate of 2 mL min^{-1}. The oven temperature program of GC was started at 90 °C for 1 min. Then, the temperature was raised to 240 °C at a rate of 7 °C min^{-1} and set for 15 min. The oven temperature and the FID temperature was set to 250 °C and 260 °C, respectively, prior to analysis. Both internal and external standard techniques were used in the calculation of FFAs. The standards, with higher than 99.5% purity, were supplied from Sigma–Aldrich. All analyses were made in triplicate and the results were expressed in mg 100 g^{-1} milk fat.

2.8. Statistical analysis

Data were analysed by analysis of variance (ANOVA) and Duncan Post Hoc Test. Statistical significance was set at $P < 0.05$ and SPSS statistical package program (SPSS ver. 13.0 for Windows, SPSS Inc., Chicago, IL, USA) was used in statistical analysis.

3. Results and discussion

3.1. Composition, water activity, free fat content, and reconstitution properties

According to the results, obtained values for the composition, water activity, free fat contents, and reconstitution properties of microencapsulated cream powder were listed in Table 2. While the moisture, fat, and free fat contents of the powders were varied in the ranges 1.01–1.47%, 60.08–62.58%, and 46.84–53.73%, respectively, the water activity and solubility values were measured as 0.163–0.199 and 48.0–53.2%, respectively. These parameters have not shown statistically significant differences against different spray drying conditions ($P > 0.05$).

The moisture content and water activity are critical values contributing to physical and bulk properties of dehydrated foods. High moisture content may cause some detrimental changes such as the decrease in flowability and the increase in caking of powders (Fitzpatrick, Iqbal, Delaney, Twomey, & Keogh, 2004). The

Table 2
Compositional and reconstitution properties of microencapsulated cream powder produced at different spray drying conditions.[a]

Properties	Inlet temperature (°C)			Feed flow rate (mL min^{-1})			Aspiration rate[3] (%)		
	150	170	190	9.0	19.5	30.0	50	75	100
Moisture (%)	1.47 ± 0.08^{a}	1.34 ± 1.09^{a}	1.18 ± 0.54^{a}	1.06 ± 0.18^{a}	1.34 ± 1.09^{a}	1.01 ± 0.03^{a}	1.27 ± 0.29^{a}	1.34 ± 1.09^{a}	1.29 ± 0.36^{a}
Fat (%)	61.17 ± 1.94^{a}	60.08 ± 1.86^{a}	61.33 ± 1.2^{a}	62.33 ± 2.02^{a}	60.08 ± 1.86^{a}	60.83 ± 0.58^{a}	61.25 ± 1.15^{a}	60.08 ± 1.86^{a}	62.58 ± 0.38^{a}
Free Fat (%)	53.73 ± 1.13^{a}	51.83 ± 4.48^{a}	52.58 ± 5.95^{a}	51.15 ± 1.12^{a}	51.83 ± 4.48^{a}	46.84 ± 3.85^{a}	48.95 ± 1.20^{a}	51.83 ± 4.48^{a}	50.59 ± 3.00^{a}
Surface Fat (%)	28.53 ± 0.95^{a}	29.75 ± 0.77^{a}	32.05 ± 0.50^{b}	27.27 ± 0.53^{a}	29.75 ± 0.77^{b}	31.46 ± 0.54^{c}	34.25 ± 1.41^{b}	29.75 ± 0.77^{a}	33.00 ± 0.35^{b}
Water Activity	0.178 ± 0.020^{a}	0.184 ± 0.027^{a}	0.172 ± 0.013^{a}	0.181 ± 0.022^{a}	0.184 ± 0.027^{a}	0.163 ± 0.007^{a}	0.179 ± 0.005^{a}	0.184 ± 0.027^{a}	0.199 ± 0.005^{a}
Wettability (s)	148.9 ± 5.3^{b}	121.2 ± 4.0^{a}	188.4 ± 5.3^{c}	122.6 ± 8.6^{a}	121.2 ± 4.0^{a}	183.2 ± 10.6^{b}	188.0 ± 4.5^{c}	121.2 ± 4.0^{a}	161.9 ± 7.5^{b}
Solubility (%)	52.1 ± 7.3^{a}	51.7 ± 4.6^{a}	53.2 ± 5.6^{a}	48.0 ± 2.2^{a}	51.7 ± 4.6^{a}	51.3 ± 4.7^{a}	52.8 ± 6.1^{a}	51.7 ± 4.6^{a}	49.4 ± 3.9^{a}

[a] For inlet temperature studies the feed flow rate was 19.5 mL min^{-1} and aspiration rate was 75%; for feed flow rate studies the inlet temperature was 170 °C and the aspiration rate was 75%; for aspiration rate studies the inlet temperature was 170 °C and the feed flow rate was 19.5 mL min^{-1}. Values are the mean ± standard deviation of the analysis results; the same superscript letters indicate no significant difference in the property between the powder samples produced at the different conditions ($P > 0.05$).

Table 3
Density properties of microencapsulated cream powder produced at different spray drying conditions.[a]

Properties	Inlet temperature (°C)			Feed flow rate (mL min^{-1})			Aspiration rate (%)		
	150	170	190	9.0	19.5	30.0	50	75	100
Bulk density (kg m^{-3})	257.7 ± 0.7^{b}	285.7 ± 4.8^{c}	237.7 ± 2.9^{a}	244.9 ± 5.4^{a}	285.7 ± 4.8^{c}	258.6 ± 7.9^{b}	268.7 ± 4.6^{b}	285.7 ± 4.8^{c}	236.7 ± 2.0^{a}
Tapped density (kg m^{-3})	472.9 ± 6.3^{b}	499.2 ± 4.2^{c}	458.1 ± 8.7^{a}	455.9 ± 7.3^{a}	499.2 ± 4.2^{b}	462.2 ± 2.9^{a}	468.6 ± 3.4^{b}	499.2 ± 4.2^{c}	454.8 ± 3.3^{a}
HR	1.84 ± 0.02^{b}	1.75 ± 0.02^{a}	1.93 ± 0.05^{c}	1.86 ± 0.02^{b}	1.75 ± 0.02^{a}	1.79 ± 0.06^{a}	1.74 ± 0.04^{a}	1.75 ± 0.02^{a}	1.92 ± 0.03^{b}
CI (%)	45.5 ± 0.6^{b}	42.8 ± 0.8^{a}	48.1 ± 1.5^{c}	46.3 ± 0.5^{b}	42.8 ± 0.8^{a}	44.1 ± 2.0^{a}	42.6 ± 1.2^{a}	42.8 ± 0.8^{a}	48.0 ± 0.8^{b}
Particle density (kg m^{-3})	1120.4 ± 72.9^{a}	1220.0 ± 55.7^{b}	1090 ± 35.7^{a}	1146.7 ± 41.2^{ab}	1220.0 ± 55.7^{b}	1075.4 ± 88.2^{a}	1102.9 ± 68.7^{ab}	1220.0 ± 55.7^{b}	1165.0 ± 33.0^{a}

[a] Abbreviations are: HR, Hausner ratio; CI, Carr index. For inlet temperature studies the feed flow rate was 19.5 mL min^{-1} and aspiration rate was 75%; for feed flow rate studies the inlet temperature was 170 °C and the aspiration rate was 75%; for aspiration rate studies the inlet temperature was 170 °C and the feed flow rate was 19.5 mL min^{-1}. Values are the mean ± standard deviation of the analysis results; the same superscript letters indicate no significant difference in the property between the powder samples produced at the different conditions ($P > 0.05$).

maximum moisture content of cream powder was defined as 5% by the Codex Standard, which is reasonable for the moisture contents of all microencapsulated cream powder samples produced in the present study (CODEX, 2011). Shelf-stable food products need to be formulated at a water activity below 0.3 where microbiological growth and chemical reactivity (e.g., lipid oxidation, nonenzymatic browning, and other enzymatic changes) are at a minimum (Roos, 2007; Tapia, Alzamora, & Chirife, 2007). A suitable water activity for a milk powder of regular composition is approximately 0.23 (Baldwin & Pearce, 2005) thus all the samples under all processing conditions in our study were below this threshold.

Free fat presents non-globular fat or non-encapsulated fat on the particle surface and should be low mainly for required flowability and reconstitution properties (Kelly et al., 2002; Vignolles et al., 2010). Free fat refers to the sum of four different types of fat found in powder particle, namely surface fat, outer layer fat, capillary fat, and dissolution fat (Foster, Bronlund, & Paterson, 2005). However, the fat content on the surface of the powders which does not correlate well with free fat is more important with respect to fat oxidation, caking and wettability properties of powders (Fitzpatrick et al., 2016; Foster et al., 2005; Keogh & O'Kennedy, 1999; Paterson, Zuo, Bronlund, & Chatterjee, 2007). Results of the present study showed that there was not a similar trend in the variation of free fat and surface fat contents of the powders whereas it was detected between surface fat and wettability of microencapsulated cream powders. While raising the inlet drying temperature or feed flow rate caused an increase in surface fat and wetting time of the powders, low surface fat and wetting time was obtained at medium aspiration rates (Table 2).

Contrary results have been reported in the literature about the effect of inlet spray drying temperature on surface fat (Farkye, 2006; Koca et al., 2015; Vignolles, Jeantet, Lopez, & Schuck, 2007). High drying rates, which mainly stem from the high spray drying inlet temperatures, result in rapid formation of a dried layer on the droplet surface. This leads to the formation of water-impermeable films on the droplet surface and partial pressure of water vapour at the centre of the droplet increases over ambient pressure with a simultaneous increase in temperature that causes bubble formation. It was reported that the powder particle did not show an internal vacuole with less stressful drying conditions (Sadek et al., 2013). Afterward, the droplet inflates and finally irregular/randomly shaped particles are produced. When further high temperature is applied to the droplet with an inflated bubble, a blistered, shrivelled, hollow or inflated puffed particle can be seen and/or particle may collapse. However, the particle does not deflate due to the dry and hard skin, when vapour condenses within the vacuole as the particle moves into cooler regions of the drying chamber. After all, damages such as fissures, breakages or capillaries on the particle surface may occur at high inlet temperature (Alamilla-Beltrán, Chanona-Pérez, Jiménez-Aparicio, & Gutiérrez-Lopez, 2005; Handscomb, Kraft, & Bayly, 2009; Ishwarya & Anandharamakrishnan, 2017; Phisut, 2012; Tonon, Freitas, & Hubinger, 2011). The increase of the surface fat with increased inlet temperature is probably due to the formation of capillaries and vacuoles, which induces larger surface areas that absorb the continuous fat phase (Farkye, 2006; Koca et al., 2015).

The reconstitution properties of powders are generally defined as a combination of wettability, dispersibility, and solubility. Although wettability and dispersibility of powders are mainly affected by the structure and surface properties of the powder particles, solubility is mainly affected by powder composition, especially its protein structure. Wettability is a property identified for powders and expressed as the time required for a powder to absorb water on the surface and get wet. The wettability depends mainly on surface area, surface charge, surface activity of the particles, particle size, contact angle between the powder surface and the penetrating water, powder density, porosity, and presence of moisture-absorbing substances (Kim, Chen, & Pearce, 2002, 2009a,b; Fitzpatrick et al., 2016; Gaiani et al., 2006). During

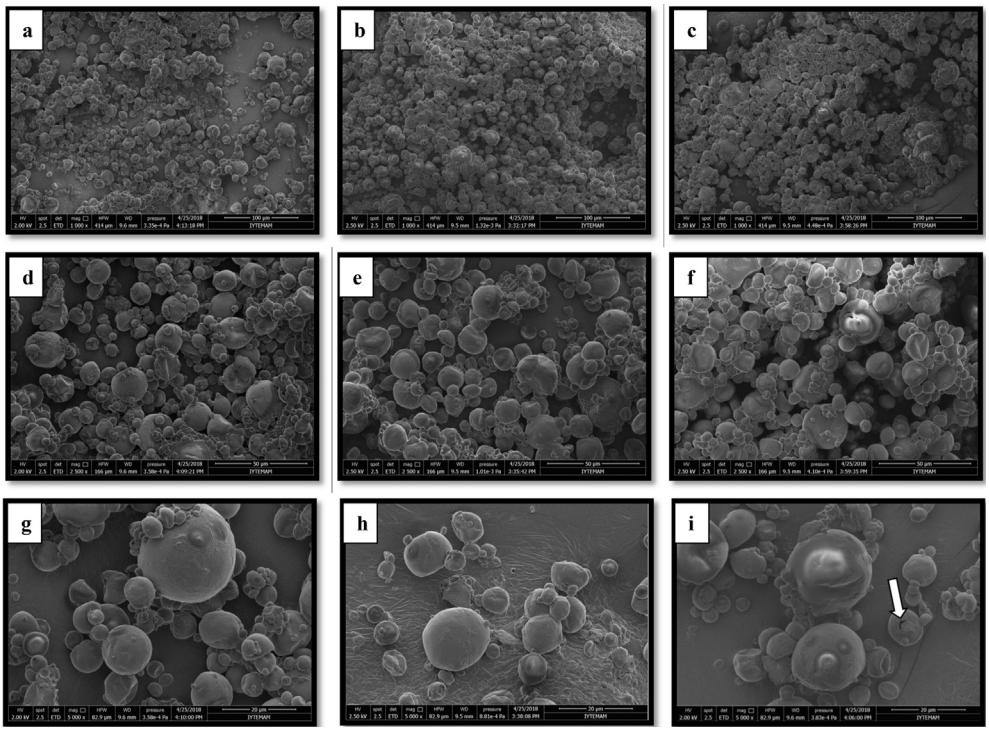

Fig. 1. Scanning electron micrographs of powder particles dried with inlet temperatures of 150 °C (a, d, g), 170 °C (b, e, h) and 190 °C (c, f, i) at a feed flow rate of 19.5 mL min^{-1} and an aspiration rate of 75%. The magnifying ratios were 1000× (a, b, c), 2500× (d, e, f), and 5000× (g, h, i).

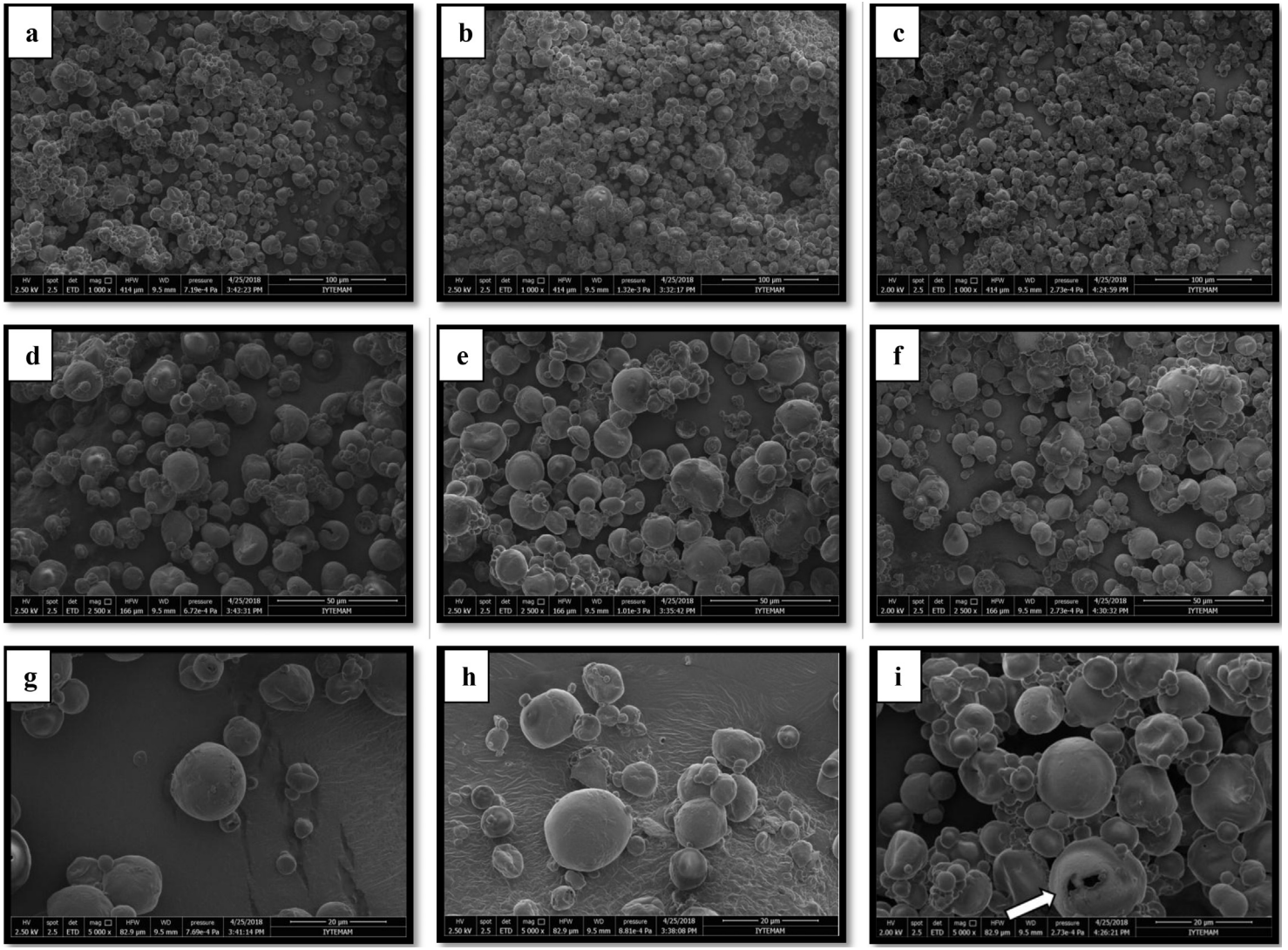

Fig. 2. Scanning electron micrographs of powder particles dried with feed flow rates of 9.0 mL min^{-1} (a, d, g), 19.5 mL min^{-1} (b, e, h), and 30.0 mL min^{-1} (c, f, i) at an inlet temperature of 170 °C and an aspiration rate of 75%. The magnifying ratios were 1000× (a, b, c), 2500× (d, e, f), and 5000× (g, h, i).

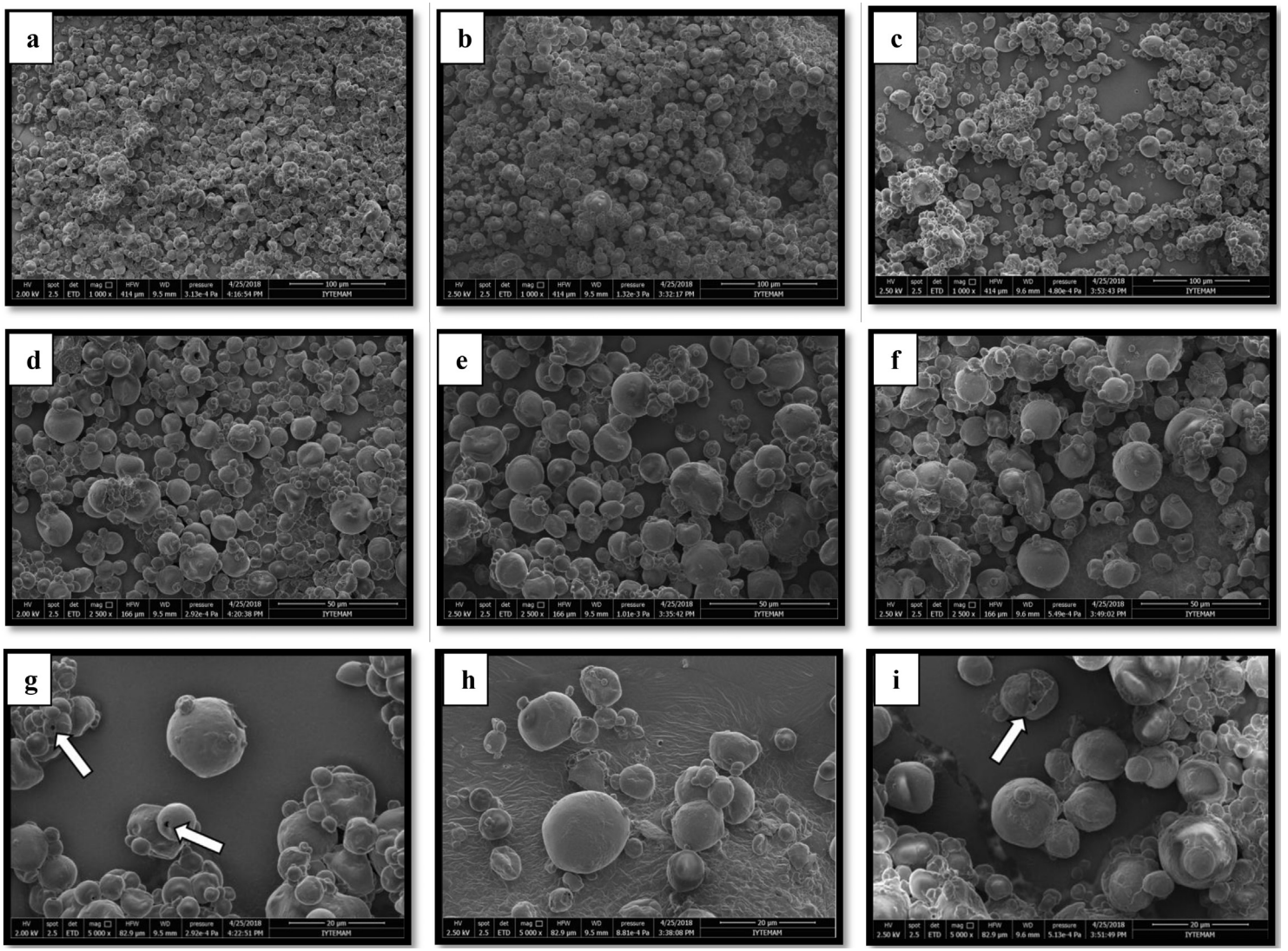

Fig. 3. Scanning electron micrographs of powder particles dried with aspiration rates of 50% (a, d, g), 75% (b, e, h), and 100% (c, f, i) at an inlet temperature of 170 °C and a feed flow rate of 19.5 mL min^{-1}. The magnifying ratios were 1000× (a, b, c), 2500× (d, e, f), and 5000× (g, h, i).

Table 4
Colour properties of microencapsulated cream powder produced at different spray drying conditions.[a]

Properties	Inlet temperature (°C)			Feed flow rate (mL min^{-1})			Aspiration rate (%)		
	150	170	190	9.0	19.5	30.0	50	75	100
L	95.96 ± 0.88^{a}	94.69 ± 0.95^{a}	96.18 ± 0.65^{a}	96.10 ± 1.38^{a}	94.69 ± 0.95^{a}	95.44 ± 1.00^{a}	95.80 ± 0.77^{a}	94.69 ± 0.95^{a}	95.36 ± 0.73^{a}
a	−0.17 ± 0.12^{a}	−0.15 ± 0.06^{a}	−0.24 ± 0.09^{a}	−0.24 ± 0.07^{a}	−0.15 ± 0.06^{a}	−0.19 ± 0.12^{a}	−0.14 ± 0.09^{b}	−0.15 ± 0.06^{b}	−0.27 ± 0.04^{a}
b	6.96 ± 1.05^{a}	7.85 ± 0.63^{a}	6.90 ± 0.74^{a}	7.12 ± 0.92^{a}	7.85 ± 0.63^{a}	7.07 ± 0.63^{a}	7.24 ± 0.69^{a}	7.85 ± 0.63^{a}	6.97 ± 0.70^{a}
Chroma	6.96 ± 1.05^{a}	7.85 ± 0.63^{a}	6.91 ± 0.73^{a}	7.12 ± 0.92^{a}	7.85 ± 0.63^{a}	7.07 ± 0.63^{a}	7.24 ± 0.69^{a}	7.85 ± 0.63^{a}	6.97 ± 0.71^{a}
BI	7.23 ± 1.32^{a}	8.34 ± 0.80^{a}	7.09 ± 0.93^{a}	7.34 ± 1.16^{a}	8.34 ± 0.80^{a}	7.37 ± 0.87^{a}	7.57 ± 0.90^{a}	8.34 ± 0.80^{a}	7.19 ± 0.71^{a}
ΔE-cream	55.96 ± 1.14^{a}	54.48 ± 1.04^{a}	56.19 ± 0.83^{a}	56.05 ± 1.54^{a}	54.48 ± 1.04^{a}	55.43 ± 11.4^{a}	55.72 ± 0.93^{a}	54.48 ± 1.04^{a}	55.39 ± 0.59^{a}
ΔE-emulsion	78.82 ± 0.92^{a}	77.50 ± 0.97^{a}	79.04 ± 0.68^{a}	78.95 ± 1.41^{a}	77.50 ± 0.97^{a}	78.29 ± 1.03^{a}	78.64 ± 0.80^{a}	77.50 ± 0.97^{a}	78.22 ± 0.71^{a}

[a] Abbreviations are: *L*, lightness; *a*, redness/greenness; *b*, yellowness/blueness; ΔE-cream, colour difference with cream as reference; ΔE-emulsion, colour difference with emulsion as reference; BI: Browning Index. For inlet temperature studies the feed flow rate was 19.5 mL min^{-1} and aspiration rate was 75%; for feed flow rate studies the inlet temperature was 170 °C and the aspiration rate was 75%; for aspiration rate studies the inlet temperature was 170 °C and the feed flow rate was 19.5 mL min^{-1}. Values are the mean ± standard deviation of the analysis results; the same superscript letters indicate no significant difference in the property between the powder samples produced at the different conditions ($P > 0.05$).

wetting, the surface composition of the powder plays an important role. Generally, hydrophobic components (e.g., fat) covering the surface contribute to poor wetting properties (Kim et al., 2002), which was confirmed in our study. In most cases, large particles form large pores, small values of the contact angle, and high porosity resulting in faster wetting (Freudig, Hogekamp, & Schubert, 1999; Kelly et al., 2002; Kim et al., 2002). Moreover, wettability and the bulk density of the powders appeared to be related (Tables 2 and 3). Powders with lower densities and lower wettability had a tendency to float. This problem can be alleviated by creating a vortex in an agitation tank which substantially improves the wettability of powders with very poor wettability properties by sinking them rapidly beneath the water surface (Fitzpatrick et al., 2016).

3.2. Powder densities

The physical properties such as compressibility, flowability, and bulk density of food powders are related. The measurement of these physical properties is important because they inherently affect the powder behaviour during storage, handling, transportation, processing, and packaging (Teunou, Fitzpatrick, & Synnott, 1999). Bulk density is defined as the weight per unit volume and is expressed as kg m^{-3} and lower bulk densities result in a higher volume of the package; therefore, lower bulk densities are not desired. Generally, an increase in the fat content of powders causes a decrease in bulk density. Moreover, high bulk density often indicates a lesser empty space available between particles to be occupied by air. Therefore, high bulk densities enhance the stability of the powder and improve protection from oxidation (Goula & Adamopoulos, 2008; Kurozawa et al., 2009).

Average bulk densities of food powders are in the range of 300–800 kg m^{-3} and skim milk powders have 400–450 kg m^{-3} bulk densities (Kelly et al., 2002). However, high-fat dairy powders have lower bulk densities usually in the range of 210–350 kg m^{-3} (Erbay & Koca, 2015; Erbay et al., 2015; Fitzpatrick et al., 2016). Additionally, tapped densities of high-fat dairy powders were reported to be in the range of 400–500 kg m^{-3} (Erbay et al., 2015; Fitzpatrick et al., 2016). While the bulk densities of the microencapsulated cream powders in our study were obtained between 236.7 kg m^{-3} and 285.7 kg m^{-3}, the tapped densities were between 454.8 kg m^{-3} and 499.2 kg m^{-3} (Table 3). All spray drying conditions significantly influenced and moderate drying conditions resulting in the highest bulk and tapped densities ($P < 0.05$). The lowest bulk and tapped densities were obtained for the highest inlet temperature and aspiration rates. Both high inlet air temperatures and aspiration rates led to the formation of air bubbles and vacuole due to rapid drying of the particles as explained above and it lowered powder density.

Carr Index (CI) and Hausner Ratio (HR), which are indices for powder flowability and cohesiveness, are reported in Table 3. All powders showed high cohesiveness and poor flowability according to the HR values ranging between 1.74 and 1.93. The CI values ranged from 42.6% to 48.1% (Table 3). With regard to the CI values, powders with CI values > 45 showed very poor flowing, whereas the CI values between 35 and 45 represented poor flowing. According to the classification of powder flowability, the CI values < 15 indicate very good flowability whereas CI values between 15 and 20 or 20 and 35, represent good or fair flowability, respectively (Jinapong et al., 2008). The decrease in inlet temperature and aspiration rate and the increase in the feed flow rate resulted in better flowability properties for the microencapsulated powders (Table 3).

3.3. Powder morphology

Scanning electron micrographs of powder particles dried under different conditions are presented in Figs. 1–3. Some cracks and deformities observed in SEM micrographs are marked with arrows in these figures. The number of the surface imperfections (damaged, cracked or fractured powder particles) for the microencapsulated cream powder was not high. Bigger particles with regular spherical shapes generally show higher flowability because of their reduced specific surface area, contact area, friction force, and adhesion of particles (Meena et al., 2018). Therefore, the flow properties of powder particles are negatively influenced by the presence of the dents; process conditions that minimise or eliminate the formation of dents should be chosen (Rosenberg, Kopelman, & Talmon, 1985).

Generally, the microencapsulated cream powder particles seemed to be spherical. Spherical powder particles should be preferred in terms of improved flowability, increased bulk densities (improved packaging properties), and enhanced retention of aroma compounds (decreased surface-to-volume ratios) (Reineccius, 2004; Vignolles et al., 2007). However, the particle size distribution of microencapsulated cream powder was not uniform and an increase in the number of small-sized particles caused a rise in the caking tendency of the particles. Reduction in the particle size increases the surface area of the powder which causes an increase in the surface fat content (Park & Drake, 2014). Increase in surface fat maximises the contact area between particles and causes the creation of interparticle liquid bridges which solidify due to fat crystallisation (Fitzpatrick et al., 2007, 2004; Thomas, Scher, Desorby-Banon, & Desorby, 2004). As a result of this, caking and agglomeration of powder starts creating an important problem during storage (Ceylan Sahin, Erbay, & Koca, 2018; Erbay & Koca, 2015). In particular, the surfaces of large particles in the microencapsulated cream powders

Table 5
Free fatty acid composition and content of microencapsulated cream powder produced at different spray drying conditions.[a]

Fatty acid (mg 100 g^{-1} milk fat)	Inlet temperature (°C)			Feed flow rate (mL min^{-1})			Aspiration rate (%)		
	150	170	190	9.0	19.5	30.0	50	75	100
Butyric acid ($C_{4:0}$)	3.10 ± 0.05^{ab}	3.52 ± 0.66^{b}	2.62 ± 0.23^{a}	2.83 ± 0.43^{a}	3.52 ± 0.66^{a}	2.95 ± 0.22^{a}	2.56 ± 0.49^{a}	3.52 ± 0.66^{a}	2.75 ± 0.22^{a}
Caproic acid ($C_{6:0}$)	1.92 ± 0.12^{a}	2.01 ± 0.46^{a}	1.38 ± 0.17^{a}	1.87 ± 0.14^{a}	2.01 ± 0.46^{a}	1.46 ± 0.09^{a}	1.49 ± 0.20^{a}	2.01 ± 0.46^{a}	1.47 ± 0.12^{a}
Caprylic acid ($C_{8:0}$)	2.23 ± 0.12^{b}	2.03 ± 0.18^{ab}	1.82 ± 0.10^{a}	2.15 ± 0.15^{b}	2.03 ± 0.18^{ab}	1.80 ± 0.19^{a}	1.80 ± 0.17^{a}	2.03 ± 0.18^{a}	1.78 ± 0.23^{a}
Capric acid ($C_{10:0}$)	6.06 ± 0.83^{a}	5.12 ± 0.31^{a}	5.20 ± 0.60^{a}	5.59 ± 0.15^{b}	5.12 ± 0.31^{ab}	4.70 ± 0.74^{a}	4.58 ± 0.19^{ab}	5.12 ± 0.31^{b}	4.11 ± 0.47^{a}
Lauric acid ($C_{12:0}$)	8.45 ± 0.45^{c}	7.58 ± 0.48^{b}	6.48 ± 0.32^{a}	7.82 ± 0.32^{b}	7.58 ± 0.48^{ab}	6.86 ± 0.48^{a}	6.93 ± 0.55^{a}	7.58 ± 0.48^{a}	6.59 ± 0.83^{a}
Myristic acid ($C_{14:0}$)	24.63 ± 1.61^{a}	24.47 ± 1.94^{a}	23.18 ± 1.64^{a}	25.08 ± 1.93^{b}	24.47 ± 1.94^{b}	19.99 ± 0.84^{a}	23.41 ± 2.20^{a}	24.47 ± 1.94^{a}	22.51 ± 1.20^{a}
Palmitic acid ($C_{16:0}$)	129.1 ± 11.3^{a}	125.3 ± 16.6^{a}	124.3 ± 4.6^{a}	130.1 ± 9.0^{a}	125.3 ± 16.6^{a}	108.4 ± 13.1^{a}	113.4 ± 3.8^{a}	125.3 ± 16.6^{a}	105.1 ± 9.7^{a}
Stearic acid ($C_{18:0}$)	62.88 ± 1.48^{a}	65.58 ± 8.43^{a}	62.87 ± 1.79^{a}	54.30 ± 5.06^{ab}	65.58 ± 8.43^{b}	49.55 ± 1.09^{a}	72.53 ± 9.91^{b}	65.58 ± 8.43^{ab}	56.01 ± 5.78^{a}
Oleic acid ($C_{18:1}$)	116.3 ± 15.4^{a}	123.7 ± 20.8^{a}	120.1 ± 14.6^{a}	112.2 ± 4.6^{ab}	123.7 ± 20.8^{b}	88.3 ± 2.6^{a}	123.2 ± 15.2^{a}	123.7 ± 20.8^{a}	103.9 ± 1.3^{a}
Linoleic acid ($C_{18:2}$)	12.89 ± 1.24^{a}	12.27 ± 0.53^{a}	12.20 ± 0.48^{a}	12.09 ± 0.73^{b}	12.27 ± 0.53^{b}	10.02 ± 0.38^{a}	13.11 ± 1.44^{a}	12.27 ± 0.53^{a}	11.71 ± 1.81^{a}
Linolenic acid ($C_{18:3}$)	2.05 ± 0.12^{b}	1.84 ± 0.19^{ab}	1.69 ± 0.19^{a}	1.90 ± 0.25^{a}	1.84 ± 0.19^{a}	1.76 ± 0.17^{a}	2.25 ± 0.40^{c}	1.84 ± 0.19^{b}	1.39 ± 0.13^{a}
VFFA	13.32 ± 0.63^{b}	12.68 ± 1.09^{b}	11.01 ± 0.76^{a}	12.44 ± 0.47^{b}	12.68 ± 1.09^{b}	10.91 ± 0.62^{a}	10.42 ± 0.41^{a}	12.68 ± 1.09^{b}	10.11 ± 0.54^{a}
MLCFFA	356.3 ± 22.7^{a}	360.8 ± 43.6^{a}	350.8 ± 11.0^{a}	343.5 ± 4.2^{b}	360.8 ± 43.6^{b}	284.9 ± 16.8^{a}	354.9 ± 24.2^{a}	360.8 ± 43.6^{a}	307.2 ± 17.1^{a}
TFFA	369.7 ± 23.0^{a}	373.5 ± 43.8^{a}	361.8 ± 10.8^{a}	355.9 ± 3.7^{b}	373.5 ± 43.8^{b}	295.8 ± 17.4^{a}	365.3 ± 24.2^{a}	373.5 ± 43.8^{a}	317.3 ± 16.7^{a}

[a] Abbreviations are: VFFA, total volatile free fatty acids ($C_{4:0}$–$C_{10:0}$); MLCFFA, total medium- and long-chain fatty acids ($C_{12:0}$–$C_{18:3}$); TFFA, total free fatty acids. For inlet temperature studies the feed flow rate was 19.5 mL min^{-1} and aspiration rate was 75%; for feed flow rate studies the inlet temperature was 170 °C and the aspiration rate was 75%; for aspiration rate studies the inlet temperature was 170 °C and the feed flow rate was 19.5 mL min^{-1}. Values are the mean ± standard deviation of the analysis results; the same superscript letters indicate no significant difference in the property between the powder samples produced at the different conditions ($P > 0.05$).

were not smooth, generally wrinkled which indicated that the amount of lactose was dominant on the surface while the surface content of fat was lower (Thomas et al., 2004).

For low and medium inlet air temperatures (150 and 170 °C), powder particles were larger and more spherical with fewer surface imperfections. It was reported that surface fat content is highly correlated with the surface area to volume ratio of powders and increase in this along with the surface imperfections may enhance the surface fat content (Vignolles et al., 2007). The results presented in Table 2 showed that the high inlet temperature caused an increase in the surface fat and a closer look at the particles showed that the surfaces of the powder particles produced at high temperatures were smooth which indicated the higher amounts of surface fat content. Slow film formation during drying of atomised droplets enhances the occurrence of the surface imperfections (e.g., cracks, collapses, wrinkles) (Ré, 1998). On the other hand, high drying rates occur on droplet surfaces cause a shrinkage and the level of it increases at higher temperatures (Phisut, 2012; Ré, 1998). As mentioned before, the rise in the inlet spray drying temperature may promote the formation of capillaries and vacuoles which results in the unprotected fat and causes an increase in the surface fat content (Farkye, 2006; Koca et al., 2015; Vignolles et al., 2007).

At the highest feed flow rate (30.0 mL min^{-1}), the number of irregularly shaped particles with shrinkages, collapses, cracks, and fractures increased. Agglomeration was initiated, especially for small particles produced at high feed flow rate conditions (Fig. 2). This was probably caused by the high level of surface fat that causes the creation of inter-particle liquid bridges and accelerates the agglomeration process. Increasing the feed flow rate resulted in higher drying rate and similar effects with the effects obtained at high inlet drying temperatures were observed.

The scanning electron micrographs of the microencapsulated cream powder particles dried at different aspiration rates were illustrated in Fig. 3. The figure showed that the particles obtained at an aspiration rate of 75% were more spherical with less agglomeration. Decreasing aspiration rate can be used to reduce the outlet temperature; however, it results in longer drying time which causes surface wrinkling and indentations. On the other hand, increase in aspiration rate leads to a rise in the drying rate which causes the formation of vacuoles and capillaries as explained before. Increased formation of vacuoles and capillaries results in the leakage of the unprotected fat found in the powder core. As shown in the scanning electron micrographs, powder particles were tended to cluster together and showed surface imperfections especially at the highest level of aspiration rate. This may be the result of increasing surface fat since it can be located at the contact ends of agglomerated particles (Vignolles et al., 2007).

The results of SEM analysis showed that the undesirable powder bulk and particle structures were obtained at the high inlet drying temperature with high feed flow rate and at high or low aspiration rate spray drying conditions. The results of the surface fat content, bulk density, and wettability analyses were generally in line with the SEM results.

3.4. Colour evaluation

The results of objective colour measurements are shown in Table 4; spray drying conditions did not have a significant effect on the colour properties of the microencapsulated cream powder ($P > 0.05$). While *a* values were measured between −0.27 and −0.15, *L*, and *b* values were in the range of 94.69–96.18, and 6.90 to 7.85, respectively. The colour difference (ΔE) with respect to cream and emulsion were calculated in the range of

54.48–56.19 and 77.50 to 79.04, respectively. The chroma values ranged from 6.91 to 7.85, whereas the Browning Index (BI) values were between 7.09 and 8.34 (Table 4).

3.5. Free fatty acid contents

The variations of FFA contents of the microencapsulated cream powders with the spray drying conditions are represented in Table 5. It is obvious that the amounts of medium- and long-chain fatty acids (MLCFFAs; $C_{12:0}$–$C_{18:3}$) were very high compared with the total volatile free fatty acids (VFFAs; $C_{4:0}$–$C_{10:0}$) contents in the microencapsulated cream powders. Approximately 96.5% of the total free fatty acids (TFFAs) in the microencapsulated cream powder were MLCFFAs. Palmitic acid was determined as the most abundant FFA in the microencapsulated cream powders followed by oleic and stearic acids. These three FFAs constituted 84% of TFFAs.

According to the results in Table 5, TFFA content did not show statistically significant difference against different spray drying inlet temperature conditions ($P > 0.05$). On the other hand, there was a remarkable increase in VFFA content as the inlet drying temperature decreased ($P < 0.05$). Moreover, the rise in the amount of butyric, caprylic, lauric, and linolenic acids in the powders with the decrease in inlet temperature was found to be important ($P < 0.05$). With respect to the FFA content at different feed flow rates, a significant variation was observed ($P < 0.05$). All FFAs, except butyric, caproic, palmitic, and linolenic acids, showed a decrease at the highest feed flow rate (30.0 mL min^{-1}). As a result, VFFA, MLCFFA, and TFFA values decreased as the feed flow rate increased. Regarding changes in the FFA content with aspiration rate, while TFFA content did not vary ($P > 0.05$), VFFA content and capric, stearic, and linolenic acids showed a statistically significant difference at different aspiration rates ($P < 0.05$). The highest VFFA content was obtained at the medium aspiration rate.

The variation of FFAs during processing of dairy products is not clear in the scientific literature, i.e., Pereira, Martins, and Vicente (2008) detected no significant variation in FFAs whereas Evers, Morris, Conaghan, and Palfreyman (2000) reported a decrease during pasteurisation according to the data obtained from a whole milk powder production plant. While a notable decrease in volatile compounds during cheese powder production was reported (Varming, Beck, Petersen, & Ardö, 2011), the decrease of the FFAs during Cheddar cheese powder production was minimised by decreasing inlet temperature and atomisation pressure (Amundson, Ishino, & Lindsay, 1980). In contrast, an increase in VFFA content during the production of milk powder was detected and attributed to enhanced lipase activity stemmed from the thermal effect of spray drying (Kim, Chang, & Kwak, 2010).

The VFFA values in our study increased as the inlet spray drying temperature and feed flow rate decreased likely due to the rise in the drying rate of the particle. The rapid drying may cause some deformations (formation of vacuoles and capillaries) on the particle structure and produce a porous powder particle with an enhanced surface area, which may enhance the effect of heat on sensitive, especially volatile, compounds (Reineccius, 2004; Souza et al., 2011). The rise in the rate of film formation would enhance the volatile retention by trapping and protecting the volatiles in the particle core until the inlet temperature was high enough to cause bubble formation and bursting out to the surface causing the enhanced specific surface area (Reineccius, 2004). The variations of surface fat and VFFA content of the microencapsulated cream powders with the spray drying processing conditions appeared to be inversely related.

4. Conclusions

In this study, the effects of the spray drying process conditions on the main physical and chemical properties of microencapsulated cream powders were investigated. High inlet drying temperatures (190 °C) with high feed flow rate (30 mL min^{-1}) resulted in powders with undesirable physical properties such as high surface fat, long wetting time, low bulk and tapped densities. Spray drying at a medium level of aspiration rate (75%) improved the powder physical properties. Results of the present study showed that the high drying rates resulted in a decrease in the quality parameters of microencapsulated cream powders. Further studies should be carried out to obtain the optimum spray drying conditions in the production of microencapsulated cream powder due to the physical and chemical quality properties and to investigate the variations in the quality parameters of microencapsulated cream powders during storage.

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