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Microencapsulation of virgin coconut oil by spray drying

Microencapsulação de óleo de coco virgem por spray spray

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ABSTRACT

Microencapsulation and spray drying nanoencapsulation are innovative technologies that has been used in the elaboration of new products. Virgin coconut oil contains a large amount of lauric acid and short chain fatty acids, which are used as antioxidants, antibacterials, antivirus and for increasing human immunity. This work was done aiming at characterizing the nanoencapsulation and microencapsulation of virgin coconut oil by drying in a spray drier. There were evaluated the variables concentration of total solids, oil concentration in relation to solids and drying temperature. A 23experimental design was used. The control parameters were: 20 to 30% of total solids using soy protein isolate with maltodextrin, in a ratio 1:2, concentration of virgin coconut oil of 25 to 50% in relation to total solids, drying temperature of 160 and 180 °C and as emulsifier ester of smite to 0.5%. Emulsions and microcapsules were characterized by stability ofemulsion, microscopy, hygroscopicity, moisture, water activity, apparent density, oil retained in the emulsion and in the

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microcapsule, surface oil, encapsulation efficiency, oil retention and morphology. The criteria for the selection of thebest microencapsulation process were oil retention and low oil content in relation to solids. Morphological analysis showed better spherical volumes with lower cracks or apparent cracks, smooth and smaller sizes (2.5 to 20 μm in diameter) had microcapsules with lower concentrations of total solids at the lowest temperature.

Keywords: Modelling, Atomization, Surface Response, Drying, Novel Products

RESUMO

A microencapsulação e nanoencapsulação por spray drying é uma tecnologia inovadora que tem sido empregue na elaboração de novos produtos. O óleo de coco virgem, contém uma grande quantidade de ácidoláurico e ácidos graxos de cadeia curta, que são usados como antioxidantes, antibacterianos, antivírus e no aumento da imunidade humana. Esse trabalho foi feito com o objetivo de caracterizar a nanoencapsulação e microencapsulação de óleo de coco virgem por meio de secagem por atomização via spray drying. Foram avaliadas as variáveisconcentração de sólidos totais, concentração de óleo em relação aos sólidos e temperatura de secagem, por meio de um delineamento experimental 23. Foram utilizados: 20 a 30% de sólidos totais usando isolado proteico de soja com maltodextrina, em uma proporção 1:2, concentração de óleo de coco virgem de 25 a 50% com relação aos sólidos totais, temperatura de secagem de 160 e 180 °C e como emulsificante éster de sacarose a 0,5%. Foram caracterizadas as emulsões e microcápsulas pelas propriedades: Estabilidade da emulsão, microscopia, higroscopicidade, umidade, atividade de água, densidade aparente, óleo retido na emulsão e na microcápsula, óleo superficial, eficiência de encapsulação, retenção de óleo e morfologia. Os critérios para a seleção do melhor processo de microencapsulação foram a retenção de óleo e o baixo teor de óleo em relação aos sólidos. Asanálises morfológicas mostraram melhores volumes esféricos com menores fissuras ou rachaduras aparentes, lisas e de menor tamanhos (2,5 a 20 μm de diâmetro) tiveram microcápsulas com menores concentrações de sólidos totais na menor temperatura.

Palavras-chave: Modelagem, Atomização, Superfície de Resposta, Secagem, Novos produtos.

1 INTRODUTION

Coconut oil is a product of vegetal origin of the species *Cocos nucifera L*. Research has shown that virgin coconut oil contains a large amount of lauric acid (49%) and myristic acid (20%) and short chain fatty acids, which are used as antioxidant, antibacterial, antivirus, improves digestion, human immunity and other health care functions (Songlin, 2007; Rui, Meiqiu&Qiuyu, 2007; Enig, 2000; Nevin &Rajamohan, 2006)Lauric acid is a medium chain fatty acid, which is transformed into monolaurin in the human body (Enig, 2000; Rajamohan& Nevin, 2006). The lauric acid has the ability to enhance the immune system by activation of the release of an interleukin-2 call substance and act as anti-inflammatory by inhibiting local synthesis of prostaglandins (PGE2) and interleukin-6 are pro-inflammatory substances present in rheumatic frames, arthritis and muscular inflammation (Wallace, et al., 2001; MACHMULLER, et al., 2003).

It is well known that spray drying is commonly used in the food industry. There are many advantages of this process including economy, flexibility and particularly good quality of resultant powder (Re, 1998; Reineccius, 2004). In addition to the properties of the food material, the quality

attributes of the powder could be affected by the spray drying conditions such as the feed flow rate, inlet and outlet temperatures, atomizer speed and inlet air flow rate (Jafari, Assadpoor& Bhandari, 2008), Among those, provided that infeed emulsion is stable over the processing time, it is important to optimize the inlet and outlet temperatures to obtain higher encapsulation efficiency (EE) and encapsulation yield (EY) (Liu, Zhou, Zeng & Ouyang, 2004). Higher inlet temperature, which directly proportional to the drying rate, may destroy heat sensitive components such as unsaturated fatty acids and carotenoids, resulting in low EE. In contrast, if the inlet temperature is too low, the water will not evaporate fully in short time and the spray-dried powder is still wet. Hence, it is easily stuck on the drying chamber wall, resulting in a low EY. In addition, the outlet temperature, which can be considered as the control indicator of the dryer and is controlled by the inlet temperature, atomization pressure and feed flow rate, may result in cracking the microcapsules due to over-heating if it is too high (Liu, et al.,2004; Gharsallaoui, Roudaut, Chambin, Voilley&Saurel, 2007).

Microencapsulation is a technique that has been utilized to protect flavor components from destructive changes and to convert flavor into a free flowing form. It is employed to preserve the stability, bioactivity and bioavailability of active components (Sansone et al., 2011; Schweiggert, Hofmann, Reichel, Schieber, & Carle, 2008). The process allows sensitive ingredients to be blended or homogenized in a solution which contains macromolecules and emulsifiers to form a stable emulsion. Encapsulating agents are used exclusively or in association with other encapsulating agents to achieve an ideal composition (Fernandes, Candido, & Oliveira, 2012). Maltodextrin, gum arabic, pectin and guar gum are examples of encapsulating agents which have been utilized in the encapsulation of bioactive compounds (Ravichandran et al., 2014). Hydrolysed starch may be combined with a surface-active biopolymer, such as gum arabic which has become a popular and common spray drying ingredient due to its emulsifying properties providing excellent volatile retention during the drying process. Combinations of gum arabic and maltodextrin were found to be effective for the encapsulation of oils (Jafari, Assadpoor, He, & Bhandari, 2008). Spray drying is the most widely utilized method of microencapsulation in the food and beverage industry (Gharsallaoui, Roudaut, Chambin, Voilley, &Saurel, 2007). More recent technologies include emulsion electrospraying which has the advantage of encapsulating under milder conditions and can be utilized for thermosensitive bioactives (Gomez-Mascaraque& Lopez-Rubio, 2016).

Microencapsulation by spray drying appears to be an effective way for those compounds in the powder form because of its advantages. Excellent properties of the protection, stabilisation, solubility and controlled release of the bioactive compounds can be obtained (Rocha, Favaro-Trindade & Grosso, 2012). The mechanism therein is to enclose the core material by forming an impermeable membrane (wall matrix) against mechanical stress, temperature, light, and oxygen

diffusion among others (Wang, Tian & Chen, 2011; Desai & Park, 2005). The first step of the microencapsulation process is to select an appropriate wall material, which has excellent functional properties (such as emulsifying, gel and film forming). Whey protein (WP), which has nutritional, physicochemical and functional properties, has been used in foods due to its ability to form thick and flexible films, preventing coalescence; whereas gum Arabic (GA) is a complex blend of natural polysaccharides composed of arabinogalactan, arabinogalactan-protein and glycoprotein. Several studies reported that high stability of encapsulated oil using a blend of WP and GA as the wall material was obtained (Rodea-Gonzalez, Cruz-Olivares, Roman-Guerrero, Rodriguez-Huezo, Vernon-Carter. & Perez-Alonso, 2012; Jimenez, Garria, & Beristain, 2006).

Microencapsulation of oils in powder particles is a technological process addressed to protect polyunsaturated oils against oxidation, to mask or preserve flavors and aromas. Microencapsulation consists of involving a solid, liquid or gaseous component in a wall material, in order to form a particle that may offer protection against oxygen, heat, humidity and light. In addition, it offers the possibility of controlled diffusion of lipophilic functional food ingredients (Charve&Reineccius, 2009). Spray drying is a process widely used for microencapsulation of oils and flavors. However, most of the flavoring compounds which give foods their characteristic aroma are highly volatile with respect to water and hence, they are easily lost during spray-drying operation (Madene, Jacquot, Scher, &Desobry, 2006). Therefore, other technological solutions that minimize the loss of flavor are needed.

Response surface methodology (RSM) is a collection of statistical and mathematical techniques useful for developing, improving and optimizing processes in which a response of interest is influenced by several variables, and the objective is to optimize this response (Bas &Boyaci, 2007). Analyzing the effects of the independent variables, this experimental methodology generates a mathematical model which describes the chemical processes within the experimental range (Myers, Montgomery, & Anderson-Cook, 2009). The most extensive application of RSM is in various stages such as determination of multi-response parameters, their effective levels, selection of experimental design, prediction and verification of model equations, generating response surfaces, contour plots and determination of optimum conditions (Maran, Manikandan, Nivetha& Dinesh, 2013).

The objective of this work was to study and characterize the physical, physicochemical and morphological properties of microcapsules of virgin coconut oil through spray drying through spray drying

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2 MATERIAL AND METHODS

Virgin coconut oil (COPRA food industries) was used for the microencapsulation. The wall materials were: maltodextrin (MD) DE-18, and soybean protein isolate (SPI) (BREMIL industries) in combination with sucrose ester emulsifier (SE) (Chemical GALENA). The virgin coconut oil was analyzed in relation to the composition of fatty acids by gas chromatography analysis coupled with mass spectrometry to check the active content of lauric and myristic acid.

2.1 EXPERIMENTAL DESIGN

The factorial design 2^3 with 3 central points was used, the variables were drying temperatures of 160 and 180 ° C, the total solids content of 20 and 30%, and the coconut oil concentration of 25% and 50% with respect to the total solids as presented in Table 1. The parameters set were the ratio of IPS: MD of 1: 2 and ES of 0.5%.

Table 1. DCCR 2^3 experimental design

Variable	Codifiedlevels		
	-1	0	+1
	DecodifiedVarables		
Total solids (%)	20	25	30
CoconutOil (%) *	25	37,5	50
Temperature (°C)	160	170	180

* In relation to total solids.

2.2 EMULSION PREPARATION

To prepare the emulsions, wall materials were dissolved in distilled water at 25 °C until complete dissolution. The virgin coconut oil was added to the hydrated wall material in concentrations of 25% and 50% relative to total solids, respectively. The emulsion was stirred using a T18 Digital Ultra Turrax homogenizer (IKA trademark) operating at a speed of 17000 rpm for 12 minutes. Optical microscopy of the emulsion drops was held in an optical microscope. The samples were placed on slides, covered with cover slip and viewed with 100X amplification. The stability of the emulsion was tested after its preparation measuring every 10 minutes during the first hour, then every 1 hour. The stability was measured by the height of the upper phase after 12 hours by the emulsion creaming index (ECI) described by Equation (1).

$$ICE = (H / Ho)100 \quad (1)$$

Where: Ho is the initial height of the lower phase and H represents the height of the upper phase after 12 hours.

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2.3 SPRAY DRYING

Spray drying was performed on a pilot scale atomizer (LABMAQ Spray Dryer MSD 3.0) with the following parameters: nozzle diameter 0,70 mm, air pressure at nozzle 5.25 bar, nozzle air flow 583m³/s, air flow rate 500 m³/s, feed pump 0,42 m³/s

2.4 EMULSIONS AND POWDERS CHARACTERIZATION

The density of emulsions (20 °C) was measured by pycnometry. Their apparent viscosity μ (Pa·s) and their rheological behaviour at 25 °C were measured using a rotational rheometer with coaxial cylinders (Brookfield DV-II PRO). Apparent viscosity was deduced from shear stress values τ (Pa) measured for different velocity gradients γ (s−1) imposed between the rotor and the stator (Newton's law $\mu = \tau \cdot \gamma^{-1}$).

The size and size distribution of emulsions and powders weremeasured by laser granulometry (Mastersizer 2000, Malvern, GB). Forthe emulsions, the analysis was performed in wet mode, dispersing afew drops of emulsion in deionized water (Hydro 2000). Powderswere analyzed in drymode (Scirocco 2000) with a compressed air pressureof 1 bar ensuring the dispersion of the particles.

Powder was also characterized by its hygroscopicity, water activity, sorption isotherms, moisture, total amount of oil in the microcapsule, surface oil, encapsulation efficiency, oil retention and morphology.

Oil retention of the microcapsules was performed according to the method described by Huynh et al., (2008). In this method, microcapsules encapsulating efficiency (MEE) is determined by the relation between the total amount of oil and the amount of oil present on the surface of the microcapsules which was determined by the method described by Bae & Lee (2008). So, MEE is given by equation (2).

$$EEM = ((To-So) / To)\ 100 \quad (2)$$

Where To is the total amount of oil and So is the amount of oil on the surface of microcapsules. Water activity aw wasmeasuredat 20 °C with an aw-meter (Decagon Agualab 4 TE Duo). For sorption isothermsX = f (aw), powder sampleswere first storedat 20 °C for severalweeks in a dry atmosphere (P_2O_5) to be dehydrated.Theywere then placed at 20 °C under different relative humidities fixedby saturated salt solutions: LiCl (11%), MgCl2 (33%), K2CO3 (43%),Mg(NO3)2 (53%) and NaCl (75%) until equilibrium (constant weight).Thewater content for each relative humidity, equal to thewater activityof the powder at equilibrium, was then measured by oven drying(105 °C).

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2.5 STATISTICAL ANALYSIS

For statistical analysis we used the response surface methodology, analysis of variance (ANOVA) and regression analysis.

3 RESULTS AND DISCUSSION

3.1 CHARACTERIZATION OF THE MATERIAL

The fatty acid composition of coconut oil is shown in Table 2. The results show coconut oil is an oil with high content of fatty acids, of which the major portion of saturated acids. The fatty acid which appears in a higher proportion is lauric acid (40.4%), followed by myristic acid (20.1%) and palmitic acid (13.6%). These results are in agreement with the manufacturer (COPRA) being small the variations compared with the literature data (Lee et al., 1998; Pehowich, et al.,2000; Dileesh, et al. 2013). which may result from these different conditions of crop seeds and oil extraction method.

Table 2 - Composition of virgin coconut oil fatty acids and its relation with manufacturer data.

Fatty acid	manufacturer	Analysed sample
Caprylic acid (C8: 0)	5.0	4.7
Capric acid (C10: 0)	5.0	5.2
Lauric acid (C12: 0)	40.0	40.4
Myristic acid (C14: 0)	21.0	20.1
Palmitic acid (C16: 0)	14.0	13.6
Stearic acid (C18: 0)	4.0	3.3
Oleic acid (C18: 1)	14.0	13.2
Linoleic acid (C18: 2)	6.0	5.5
Caproic acid (C6: 0)	0.4	0.4

Emulsions must remain stable during the time of microencapsulation process by spray drying, which has a duration of approximately 1 hour. For this period, all the emulsions were stable and no phase separation was observed, confirming the sucrose ester as an excellent emulsifier.

It was found that the total solids, the concentration of oil in relation to the solids as well the interaction between them were not statistically significant (ANOVA at 95% confidence). Thus, all of the emulsions were stable during the observation period. However, it may be noted that the greater the amount of isolated soybean protein, there was a slight instability in the emulsion within 12 hours. This slight phase separation was characterized by the presence of an oil surrounded by foam collar. There are some studies in the literature on the ability of proteins to form foam (Lam & Nickerson, 2013; Oboroceanu et al.,2014; Sadahira, et. al., 2014). and this may be related to the incorporation of air during homogenization, where frictional forces are primarily responsible for the event in question (Bals &Kulozik, 2003).

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Figure 1 shows some microphotographs of the emulsions, it is noted that for all the samples homogenized during 12 minutes, the beads of emulsion proved to have homogeneous sizes ranging from 10 to 100 micrometers. In the emulsions obtained with 30% and 20% of total solids and 50% of coconut oil we observed few drops with sizes larger than that having 25% of coconut oil. This may be explained by the increase in oil content, which reduces concentration, and consequent availability, of emulsifier in the emulsion, preventing to completely cover of the drops, causing an increase in the coalescence rate and hence in the droplet size (Jafari & Bhandari, 2007).

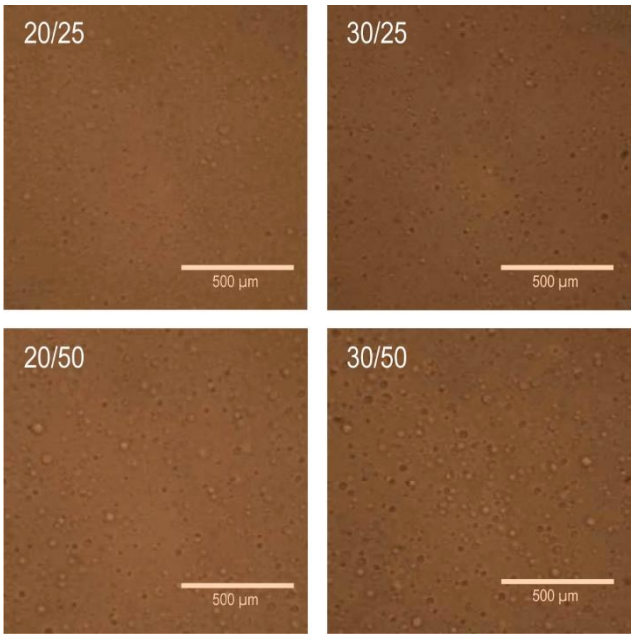


Figure 1 – Optical microscopy of the homogenized emulsions (100X amplified)

The results of the statistical analysis, applied to the experimental data of oil content in the emulsion, obtained during the homogenization, are presented in Table 3. It is verified that the oil concentration in relation to the solid was statistically significant (ANOVA at 95% confidence) and showed an effect on the retention of oil in the emulsion, that is, an increase in these variables caused a decrease in oil retention; were not significant with respect to the total solids and the interactions between the oil concentration and total solids.

Analyzing the response surfaces generated, it was observed that high oil content (25-50%) compared to solid resulted in lower total oil retention in the emulsion during homogenization. The efficiency of emulsification depends mainly on emulsifying properties of the matrix and its ability to form films in the emulsion interfaces (Baranauskiene et al., 2007).

3.2 CHARACTERIZATION OF THE MICROCAPSULES

Microcapsules hygroscopicity values are shown in Table 4. The microcapsules hading the highest hygroscopicity values are those that were microencapsulated at the temperature of 180 °C for

all samples, while the samples produced at 160 °C showed lower values. For samples 30/50/180 and 30/50/160 hygroscopicity decreases with increasing the amount of maltodextrin in relation to 20/50/180 and 20/50/160 samples, respectively, these results can be due to the fact that maltodextrin is a material low hygroscopicity (Tonon, Hubinger&Brabet, 2008).

The variables oil concentration and temperature showed statistically significant differences (ANOVA at 95% confidence) while total solids variable and all interactions between variables showed no statistically significant differences. In response surfaces generated for hygroscopicity, the concentration of oil in relation to solids and temperature were the variables that most influenced the hygroscopicity response, with the highest values hac been obtained when using high concentrations of oil and temperature.

Water activity valuesof the oil microcapsules are shown in Table 4. Total solids, oil concentration, temperature and the interation total solids concentration X Oil concnetration showed differenes statistically significants (ANOVA 95% confidence). The interations total solids X temperature and concentration of oil X temperature did not show statistically significant differences (ANOVA 95% confidence). The response surface and response contour curve generated for water activity indicate that it increases with the concentration of total solids and decreases with the temperature and concentration of coconut oil.

Table 4 – Results of hygroscopicity, water activity, moisture and bulk density of coconut oil microcapsules

Samples	Higroscopicity (g/100g)	Water Activity (Aw)	Moisture Content (%)	Bulk Density (g/cm3)
30/50/180	16,547±0,468	0,162±0,005	2,611±0,020	0,524±0,011
30/50/160	15,537±0,284	0,184±0,002	2,988±0,097	0,544±0,011
30/25/180	16,033±0,225	0,175±0,005	2.158±0,168	0,518±0,009
30/25/160	15,223±0,287	0,193±0,008	2.430±0,026	0,553±0,028
20/50/180	16,536±0,105	0,133±0,005	2.146±0,162	0,450±0,017
20/50/160	15,320±0,265	0,144±0,005	2.276±0,135	0,485±0,010
20/25/180	16,150±0,157	0,174±0,007	2.115±0,126	0,450±0,016
20/25/160	15,210±0,110	0,176±0,006	2.250±0,067	0,484±0,007
25/37,5/170	15,883±0,125	0,168±0,007	2,461±0,221	0,475±0,005

The water activity (Aw) values founded can be considered very low, varying from 0.133 to 0.184. The value of water activity is strongly related to food preservation. Water-rich foods with Aw values above 0.90 may form dilute solutions with components of food that will serve as a substrate for microorganisms growing. At lower Aw values varying from 0.40 to 0.80 there is a possibility of fast chemical and enzymatic reactions due to the increasing concentrations of the reactants, whereas in foods with Aw below 0.60 there is little or no microorganism growth.

In regions where Aw is less than 0.30, as in the present work, the primary absorption zone is reached, where the water molecules may be bound to primary absorption points (eg-COOH) and also bind to other water molecules through hydrogen bonding. This water is tightly bound to the food forming the so-called monolayer (single layer with limited thickness of few Angstroms). This water is not usable for dissolving food components, which leads the reactions wich have velocities tending to zero, except for lipid oxidation which is considerably faster (Bobbio&Bobbio, 1992).

The moisture content of the samples presented in Table 4 ranged from 2.12% to 2.99% for samples 20/25/180 and 30/50/160, respectively. Similar results were observed by HOGAN et al. (2001). These authors reported moisture values of 1 to 3% in microcapsules of soybean oil in spray dryer drying. The moisture content of the microcapsules is strongly determined by the relative humidity of the air in the dryer.

The results of the statistical treatment (95% confidence interval ANOVA) for the response variable moisture as a function of the concentration of total solids, coconut oil and temperature, indicates that there is a significant difference in the total solids concentration, in increasing the moisture content of the microcapsules. The response and contour curves of the moisture response generated by the proposed model indicate that moisture increases more when the total solids concentration increases than when the variables coconut oil concentration and temperature increase. However, the increase in coconut oil concentration and the decrease in temperature favors the increase in moisture of the microcapsules. The variables coconut oil concentratioemtemperatura were no significnt effect in the model.

HOGAN et al. (2001) reported that the moisture content was not affected by total solids concentration and oil concentration with respect to total solids. CARNEIRO (2011) mentions that there was a significant difference between all combinations of wall materials in the moisture analysis of the linseed oil microcapsules. According to (BAIK et al., 2004) one of the most important factors that determines the stability of microcapsules is the presence of moisture

The bulk density of the coconut oil microcapsules was in the range of 0.450 to 0.544 g/cm^3 for all samples as shown in Table 4. KIM & MORR (1996) reported bulk density values for orange oil

microcapsules in the range of 0.21 g/cm^3 to 0.46 g/cm^3 for particles with protein isolate from whey and gum arabic as wall material, respectively.

The results of the statistical treatment (95% confidence intervals ANOVA) for the "bulk density" response variable as a function of the concentration of total solids, coconut oil and temperature factors indicate that there is a significant difference in the total solids concentration, increase in bulk density. The response surface and the contour curve of the moisture response generated through the proposed model indicate that the bulk density increases significantly as the total solids concentration increases. Variables coconut oil concentration and temperature were not significant in the model. This result may be due to these samples containing higher concentration of soy protein isolate and maltodextrin.

The decrease in coconut oil concentration and decrease in temperature favored the increase of the apparent density in the microcapsules. Similar results were observed by BAE and LEE (2008) for microencapsulation of avocado oil. Apparent densities increased gradually with increasing proportion of maltodextrin (total solids). The authors affirmed that this result is related to the fact that the particles containing more maltodextrin present a high degree of agglomeration and structural collapse that can generate decrease of the volume of particles.

Table 5 shows the amount of total oil in the microcapsules that were in the range of 65.24 to 89.50 for the samples 20/25/180 and 30/25/180, respectively. The results of the statistical analysis (ANOVA at 95% for the response variable "total oil content" as a function of the factors total solids concentration, coconut oil and temperature indicated that there is a significant difference in coconut oil concentration, which led to an increase in the oil content of microcapsules. The response surface and contour curve of the "oil content" response generated through the proposed model indicate that the total oil content increases as the coconut oil concentration increases. It was also observed that the variables Total Solids and temperature were not significant in the model.

The decrease in the coconut oil concentration and the increase in drying temperature favors the increase of oil content in the microcapsules, as well as increase of total solids and the drying temperature increases the total oil content which can be explained by the higher retention (RÉ, 1998), which reduces the mobility of the compounds to form a protective barrier around the particles (CHARVE & REINECCIUS, 2009).

The results of surface oil content in the microcapsules (Table 5) ranged from 62.67 to 96.16% for samples of 30/25/180 and 20/50/160, respectively. These results are related to the solids content, the concentration of oil placed in the emulsions and the drying conditions. The results of the statistical treatment (95% confidence intervals ANOVA) for the "surface oil" response variable as a function of the concentration of total solids, coconut oil, indicate that increase in coconut oil concentration

and temperature leads to an increase in oil of the coconut oil microcapsules. The response surfaces and the contour curve of the "surface oil" response generated by the proposed model confirm this increase. It was also observed that the variables total solids and temperature were not significnt in the model.

According to BAE and LEE (2008), working with microscopes of avocado oil in matrices constituted by mixtures of whey protein isolate: and maltodextrin in a ratio of 10:90 and 50:50, did not observe correlation between oil content and the concentrations of protein isolate whey and maltodextrin. These authors reported that the high levels of surface oil observed were probably due to several factors such as: agglomeration and collapse of the particles, which could have altered the structural integrity and morphology of the structures, resulting in the release of the encapsulated oil by the compression of the and the high volume of vacuums that increase the surface oil content (KEOGH et al., 2001).

Table 5. Results of Oil content, surface oil, retention and encapsulation efficiency of coconut oil microcapsules

Samples	Total oil content in powder (%)	Surface Oil (%)	Oil retention (%)	Encapsulation Efficiency (%)
30/50/180	75,36±1,03	94,99±2,04	54,36±0,80	60,06±1,49
30/50/160	67,08±2,06	96,16±1,32	58,95±1,62	63,00±2,38
30/25/180	89,50±2,07	62,67±4,39	74,85±2,13	75,43±1,26
30/25/160	86,59±1,55	81,50±2,61	75,44±1,39	74,81±2,99
20/50/180	73,29±0,68	82,45±2,52	64,33±3,89	68,37±1,44
20/50/160	65,24±4,56	96,05±4,95	57,99±2,04	66,73±2,60
20/25/180	81,74±1,54	71,73±3,69	74,45±0,95	78,79±1,18
20/25/160	80,10±0,40	80,11±2,19	76,10±2,20	81,29±2,09
25/37,5/170	73,66±1,49	85,62±1,02	67,07±2,10	73,10±2,42

The oil retention in the coconut microcapsules was in the range of 54.36 to 75.44% (Table 5). The results of the ANOVA (95% confidence) for the oil retention response variable as a function of the concentration of total solids, coconut oil and temperature indicated that there is a significant difference in the concentration of coconut oil, the retention of oil in coconut oil microcapsules. In Figure 2 the response surfaces and contour curves of the oil retention response generated by the proposed model are shown, indicating that the oil retention increases as the coconut oil concentration

decreases. It was also observed that the concentrations of total solids and temperature were not significant

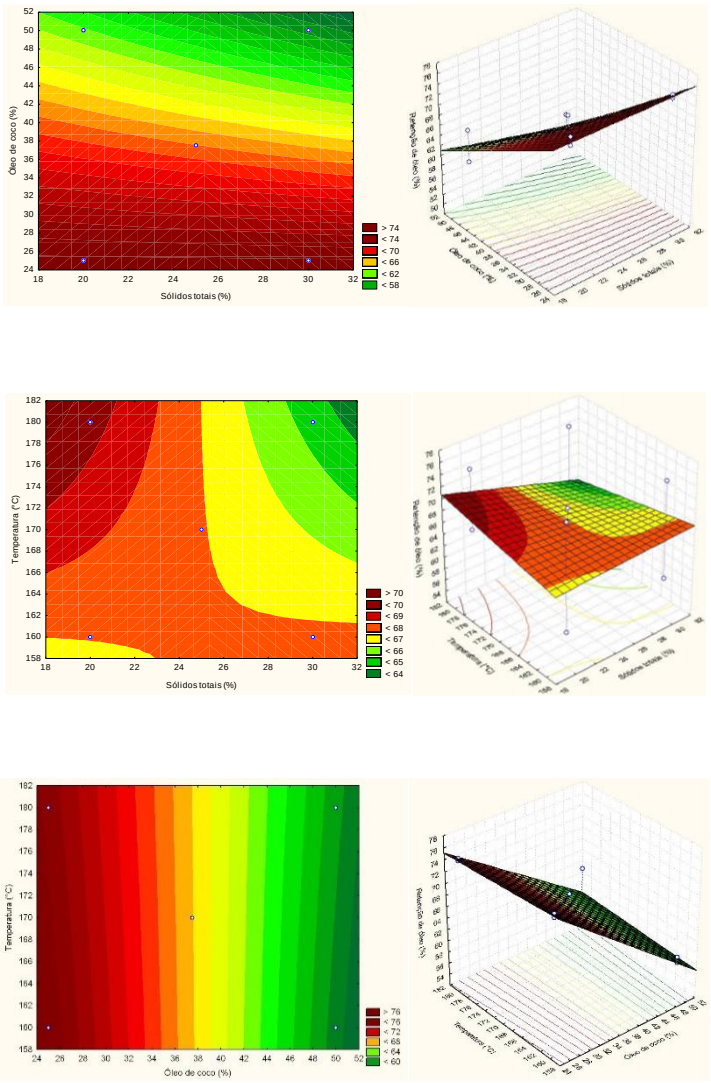


Figure 2. Surface response surface and contour curve of the microcapsule oil retention response.

The encapsulation efficiency (EEM) (Table 5) ranged from 60.06% to 81.29%, the smallest value being the 30/50/180 sample and the highest value for the 20/25/160 combination. The results of the statistical analysis (95% confidence interval ANOVA) for the response variable "encapsulation efficiency" as a function of the concentration of total solids, coconut oil and temperature factors indicate that there are significant differences in the concentration of total solids and oil of coconut, which have the effect on the encapsulation efficiency of virgin coconut oil microcapsules. Figure 3 shows the response surfaces and contour curves for the oil retention response variable generated by the proposed model, indicating that the encapsulation efficiency increases as the coconut oil concentration and the total solids decrease. It was also observed that the temperature variable was not significant when used in the model. Thus, it is possible to conclude that the use of lower concentration

of maltodextrin and soybean protein isolate had the effect of the higher total wall combinations of total solids and coconut oil concentration as wall material.

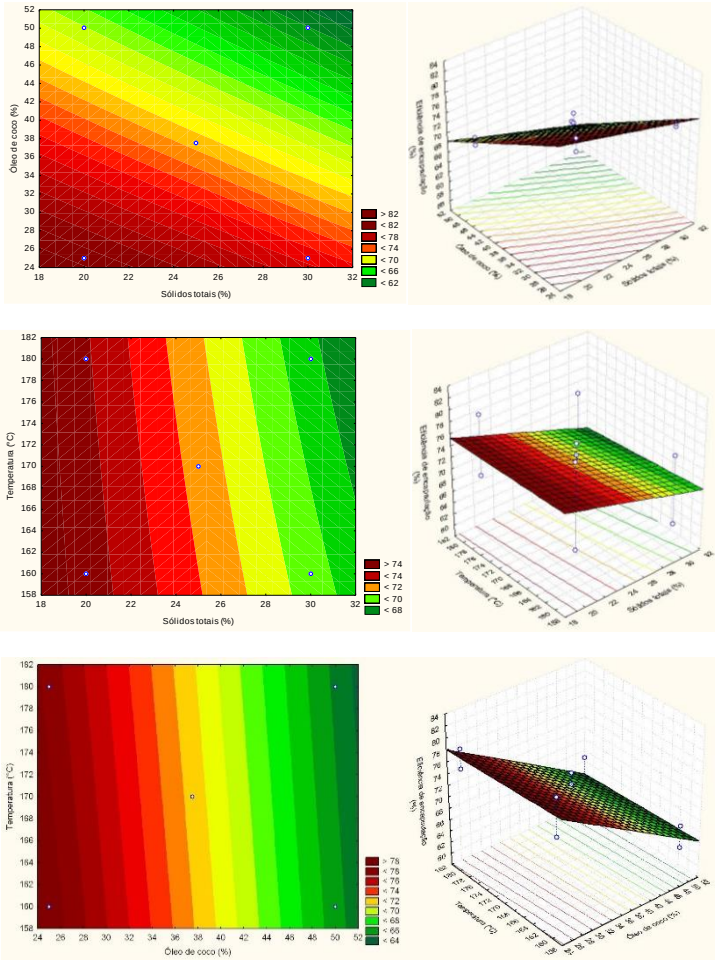
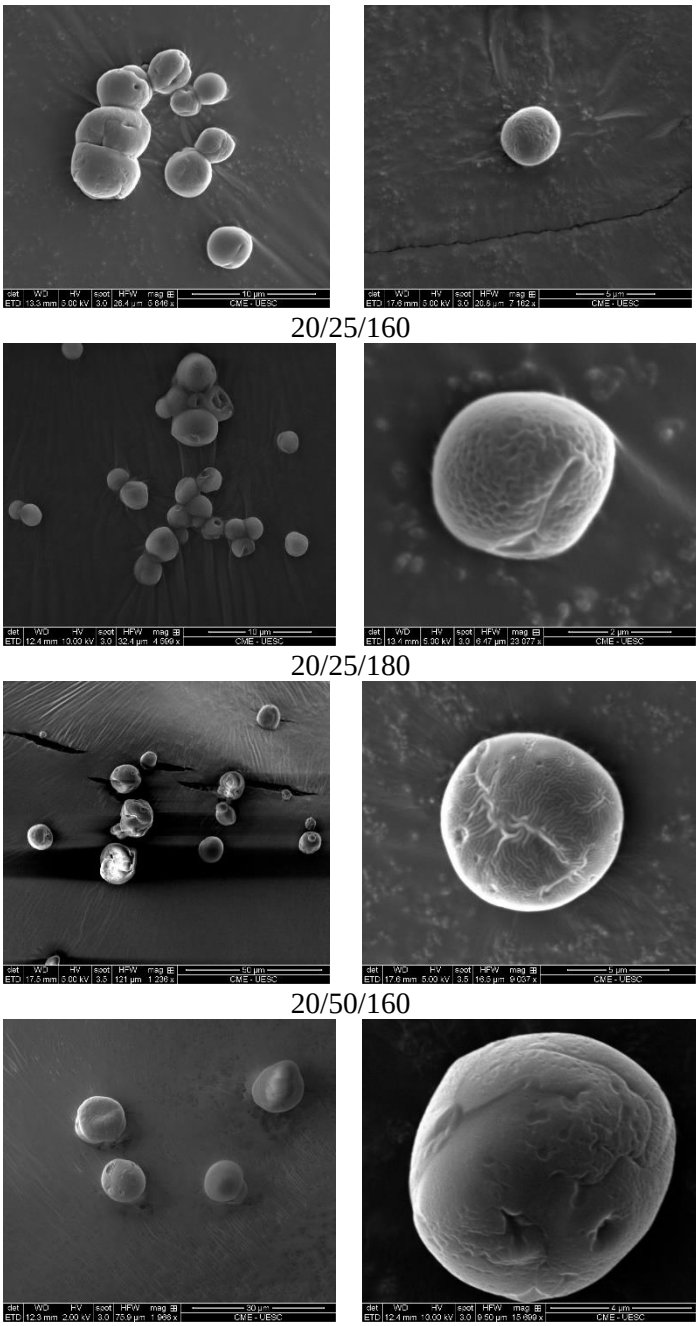


Figure 3. Surface surfaces of response and contour curve for response variable efficiency of encapsulation in microcapsules of coconut oil.

Similar effects are observed by CARMONA et. al (2013) for orange microcapsules. These authors stated that high concentrations of oil in relation to solids decreased the efficiency of encapsulation, due to the higher oil content on the surface of the particles. Such a result can be explained by the fact that at a same concentration of total solids the increase in oil content decreases the concentration of the encapsulating agent to form the matrix on the surface of the core prior to the formation of the crust around the dried droplets.

Figures 4 and 5 illustrate the morphological differences between the intact particles obtained at different ratios and total solids concentrations of 20 and 30%. The microparticles showed spherical volume without cracks or apparent cracks, which is a positive point, since this characteristic represents less permeability of the capsules to gases, increases the protection and retention of the active material. In addition, the microspheres showed variety in size between 2.5 and 50 μm, being

classified as microparticles or microcapsules. Similar morphological characteristics were found by TONON et al. (2011) and by TRINDADE and GROSSO (2000), in microencapsulation by spray drying of linseed oil and ascorbic acid, respectively.



20/25/160

20/25/180

20/50/160

20/50/180

Figure 4. Scanning electron microscopy of microcapsules of virgin coconut oil at a total solidsconcentration of 20%

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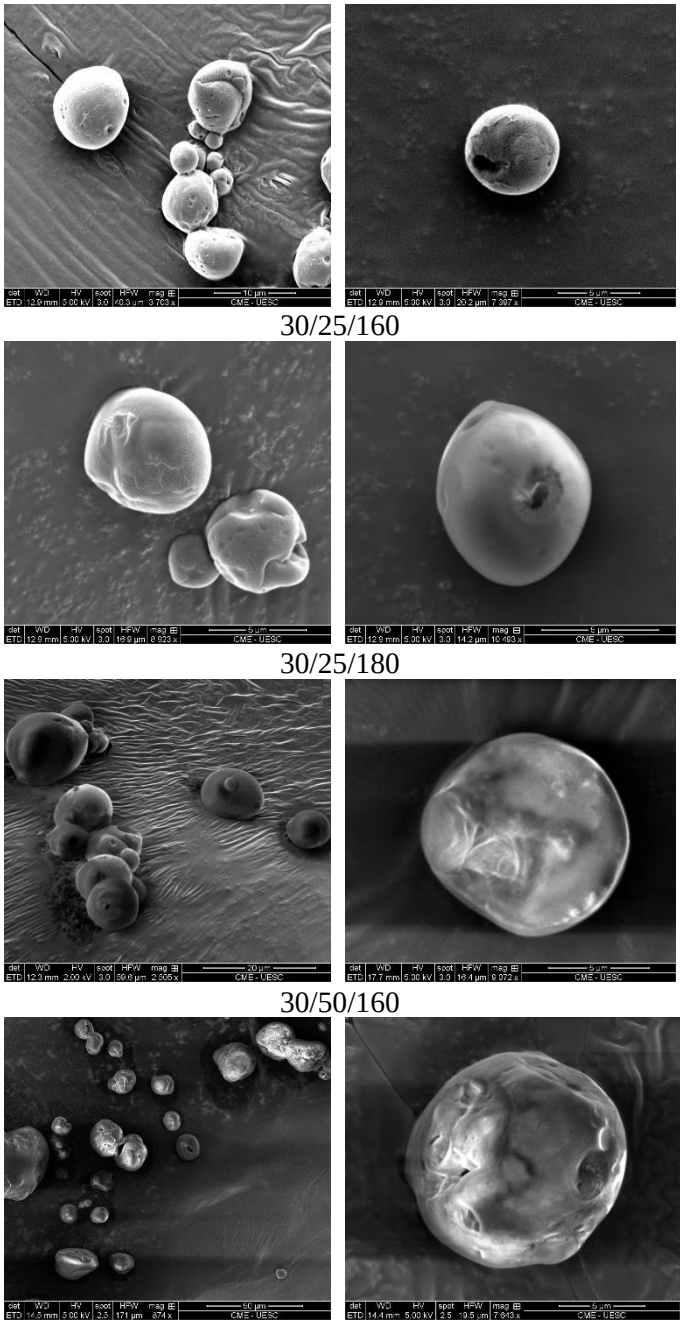

Figure5. Scanning electron microscopy of microcapsules of virgin coconut oil at a total solidsconcentration of 30%.

It was observed that the blends of different wall materials had little influence on the morphology of the microparticles. This difference is most evident when comparing the images of the combination of lower concentration of coco nut oil with relatively smaller sizes than higher concentrations of coconut oil. It can also be observed that for all the samples with smooth surface, it also observed agglomerations for images with greater magnification of 500 times approximately.

BAE and LEE (2008), studding the microencapsulation of avocado oil with mixtures of protein isolate of serum and maltodextrin, observed surface roughness of the particles and high degree of agglomeration.

4 CONCLUSIONS

For all samples the emulsions were stable for 1 hour and no phase separation was observed, confirming that the sucrose ester is an excellent emulsifier.

Emulsion droplets showed homogeneous sizes. In the emulsions obtained with 30% and 20% of total solids, with 25% coconut oil were observed some drops smaller than 50% virgin coconut oil. The variables that had statistical significance in the efficiency of microencapsulation and oil retention were the total solids content and the oil content in relation to the solids.

Microparticles with spherical volumes of 2.5 to 50 μm in diameter, without cracks, smooth and without cracking were obtained with total solids concentrations of 20%, coconut oil concentration of 25% and temperature between 160 and 180 ° W.

The obtained microcapsules of virgin coconut oil by spraydrying showed to be quick and practical to produce using as wall material the soy protein isolate, maltodextrin and sucrose ester emulsifier, the indicated proportions being 20/25 at 160 ºC, followed by 20/25 at 180 ºC.

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