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Microencapsulation of roasted coffee oil by complex coacervation with gelatin/gum arabic enzymatically crosslinked

Camila Carolina Veiga
camilacarolveiga@hotmail.com
Universidade Tecnológica Federal do Paraná, Campus Campo Mourão (UTFPR-CM, via Rosalina Maria dos Santos, 1233, CEP 87301-899, Caixa Postal: 271, Campo Mourão, PR, Brasil.

Rayssa Cristine Simoni
rayssa.simoni@gmail.com
Universidade Tecnológica Federal do Paraná, Campus Campo Mourão (UTFPR-CM, via Rosalina Maria dos Santos, 1233, CEP 87301-899, Caixa Postal: 271, Campo Mourão, PR, Brasil.

Melina Maynara Carvalho de Almeida
melina.almeida88@gmail.com
Universidade Tecnológica Federal do Paraná, Campus Campo Mourão (UTFPR-CM), via Rosalina Maria dos Santos, 1233, CEP 87301-899, Caixa Postal: 271, Campo Mourão, PR, Brasil.

Odinei Hess Gonçalves
odinei@utfpr.edu.br
Universidade Tecnológica Federal do Paraná, Campus Campo Mourão (UTFPR-CM), via Rosalina Maria dos Santos, 1233, CEP 87301-899, Caixa Postal: 271, Campo Mourão, PR, Brasil.

Marianne Ayumi Shirai
marianneshirai@hotmail.com
Universidade Tecnológica Federal do Paraná, Campus Campo Mourão (UTFPR-CM), via Rosalina Maria dos Santos, 1233, CEP 87301-899, Caixa Postal: 271, Campo Mourão, PR, Brasil.

Fernanda Vitória Leimann
fernandaleimann@utfpr.edu.br
Universidade Tecnológica Federal do Paraná, Campus Campo Mourão (UTFPR-CM), via Rosalina Maria dos Santos, 1233, CEP 87301-899, Caixa Postal: 271, Campo Mourão, PR, Brasil.

RESUMO

The roasted coffee oil is a coffee industry byproduct composed by a great number of compounds including the volatiles, responsible by coffee aroma. The main objetive of the present work is the encapsulation of roasted coffee oil in enzimatically crosslinked gelatin and gum Arabic microcapsules. The microcapsules were obtained by complex coacervation. The effect of the roasted coffee oil amount used and the influence of the stirring rate on the average diameter, oil recovery and encapsulation efficiency were evaluated. The microcapsules presented average diameter up to 29.30 ± 1.28 µm with narrow size distributions. The results varied from 78.45 ± 2.47 to 100.70 ± 5.23 % (oil recovery) and 32.50 ± 3.53 to 71.00 ± 4.24 % (encapsulation efficiency). The water sorption isotherm of microcapsules was evaluated at 25 ºC and the results were fitted with the Guggenhein-Anderson-de Boer (GAB) model, presenting a R^2 equal to 0.9892 and "J" shape behavior. Finally, the crosslinking reaction was investigated by Fourier Transform Infrared Spectroscopy (FTIR), showing that the crosslinking reaction promoted by transglutaminase introduced a band in microcapsules spectra located at 1550 cm^{-1} corresponding to the monosubstituted amide.

KEYWORDS: Roasted Coffee Oil; Microencapsulation, Complex Coacervation.

INTRODUCTION

The freshly brewed coffee is appreciated by consumers due to its pleasant aroma. However, this aroma is not stable and changes rapidly after coffee preparation (MONTEIRO et al., 2005). Consisting of a mixture of volatile compounds and obtained from the roasting and pressing the beans (CLARKE; VITZTHUM, 2001; SARRAZIN et al., 2000). Aroma is one of the most important attributes of roasted coffee (DE OLIVEIRA et al., 2005), so it contains properties that can enhance the flavor of coffee-based products.

During the processing of instant coffee its flavor is partially lost due to the processing conditions. Different roasting degrees, coffee bean varieties (arabic or conilon) and extraction processes lead to a great variability on the aroma profile (KOBAYASHI; BENASSI, 2012). Many strategies have been made in order to supplement the lost flavors during soluble coffee production, being this approach a technological challenge that involves the use of roasted coffee oil. The direct incorporation of roasted coffee oil, extracted with supercritical CO_2, to instant coffee was proposed by Oliveira et al. (2009). The authors found that the enrichment of drinks (freeze and spray dryed instant coffee) with the aromatic oil of roasted coffee did not influenced the consumer acceptance with respect to the aroma, but exerts influence with respect to flavour. To limit aroma degradation or loss during processing and storage, it is beneficial to encapsulate volatile ingredients prior to use in foods and beverages (FIGUEIREDO; MIGUEL, 2010).

The complex coacervation technique offers a unique method for the encapsulation of flavours, is a simple, scalable, low cost, solvent-free and reproducible method, being a highly advantageous technique for the fabrication of microcapsules in industry (XIAO et al., 2014). Gelatin, a collagen hydrolysis product positively charged, is widely used in complex coacervation associated with different anionic colloids to neutralize its charge and thereby form a complex. Usually, the negatively charged polysaccharides associated with gelatin are pectin, alginate, gum Arabic, carboxymethylcellulose, etc (PRATA et al., 2007, 2008). A crosslinking agent must be used to stabilize the microcapsules structure. This hardening step can be achieved with the use of aldehydes, such formaldehyde or glutaraldehyde, the most common crosslinking agents used. However they present elevated toxicity levels and thus are undesirable for food processing applications (DONG et al., 2008). Many researches are now exploring new security crosslinking agents such as transglutaminase(Xiao, Liu, Zhu, Zhou, & Niu, 2014a). Transglutaminase (TGase) catalyses the formation of a covalent bond between gamma-carboxylamide groups of peptide or protein-bound glutamine residues and primary amino groups, resulting in inter and intramolecular crosslinks (GONZÁLEZ-PÉREZ; ARELLANO, 2009).

In this work the complex coacervation was applied to microencapsulate roasted coffee oil. Gelatin and gum Arabic were used as wall forming material and TGase was used as crosslinking agent of microcapsules. The effect of the processing paramenters, oil amount and stirring rate, were investigated on the final microcapsules size, size distribution, oil recovery and encapsulation efficiency. The interaction between the encapsulated roasted coffee oil and the microcapsules wall was investigated by Fourier Transform Infrared Spectroscopy and the sorption isotherm describing the relationship between water activity

(aw) and the equilibrium moisture content of the produced microcapsules at 25 ºC were also studied in the present work.

MATERIAL AND METHODS

MATERIALS

Gelatin and gum arabic (P.A., Vetec Química Fina) were used as wall material for microcapsules. The crosslinking agent transglutaminase was kindly supplied by Ajinomoto Interamericana Ind. e Com. Ltda. (Activa TG-S®, São Paulo, SP, Brazil), with nominal activity of 100 U/g of powder, according to the information provided by the industry. Roasted coffe oil was kindly supplied by CIA. Iguaçú de Café Solúvel. Hydrochloridric acid (0.25 mol/L) and sodium hydroxide (0.01 N) (P.A., Vetec Química Fina) aqueous solutions were used to modify the pH of dispersions. Dicloromethane (P.A., Vetec Química Fina) was used in the oil load and encapsulation efficiency determinations.

MICROENCAPSULATION PROCEDURE

Two experimental parameters were studied in this study: the stirring rate and the amount roasted coffee oil added at the formulation. The experiments were carried out in duplicate. The complex coacervation procedure adopted was described by Prata et al. (2008). Aqueous solutions of gelatin (33 mL) and gum Arabic (33 mL) (2.5 wt %) were prepared with water at 50° C and after that mixed in a borosilicate jacketed reactor using a propeller impeller. The temperature was controlled by a thermostatic water bath (Tecnal) at 50°C and water was added (166 ml). Immediately the roasted coffee oil was added (0.8250 g or 0.4125 g, corresponding to 50 and 25 wt % of the total dispersed phase, respectively) and the stirring rate was adjusted to 300 or 500 rpm. This condition was maintained by 30 min. After that the emulsions pH was slowly reduced to pH 4 with the addition of an HCl aqueous solution (0.25 mol/L) and the temperature was reduced gradually during 30 min to 10°C. Finally, the pH was kept equal to 4, the temperature was raised to 25 °C, TGase (0.667 g) was added and the system was maintained under magnetic stirring (100 rpm) during 13 h to complete the crosslinking reaction. The final suspension of the microcapsules was stored under refrigeration (8 ºC).

MICROCAPSULES CHARACTERIZATION

Microcapsules morphology, average size and polydispersity index

The procedure adopted was described by Leimann et al. (2009). Optical observations of the microcapsules were carried out with the aid of an optical microscope (Bioval L-2000A) attached to a digital camera. The final microcapsules suspension was dropped in a microscope slide and observed under a

magnification equal to 100x. The microcapsules average size (Dp) was determined measuring the microcapsules diameter using an image analysis software (SizeMeter 1.1). About 300 microcapsules were measured for each experiment. The polidispersity index (PDI) was determined with the Equation 1 were "σ" (µm) represents the standard deviation of the diameter measurements and "Dp" the average diameter (µm). The procedure was adopted in triplicate for each experimental condition.

$$PDI = \left(\frac{\sigma^2}{Dp^2} \right) \quad \text{Eq. (1)}$$

Oil recovery and encapsulation efficiency

The oil recovery and encapsulation efficiencies analysis were performed in triplicate for each formulation. For the roasted coffee oil recovery determination, the final suspension of microcapsules was placed in a Falcon flask (25 mL), frozen at -90 ºC (Ultra freezer, Liotop) during 5 h and finally freeze dried (Liotop L101) during 3 days. The freeze dried microcapsules were introduced into glass a desiccator, containing silica, at room temperature for 1 week in order to reduce to a minimum the relative humidity. After that, 50 mg of microcapsules were ground in a porcelain mortar to release the oil and 10 mL of dichloromethane were quickly added. The solution was filtered with a nylon filter (0.45 µm, Maxcrom) and diluted with dichloromethane (1:10) and finally its absorbance was measured in a UV-Vis spectrophotometer (UV-Vis Ocean Optics). The maximum wavelength of the roasted coffee oil absorbance spectrum (285 nm) was used as reference (Sibanda et al., 2004). The concentration of the roasted coffee oil (C_{OR}, mg/mL) was determined using a calibration curve previously prepared (in triplicate). The roasted coffee oil recovery (OR) was then calculated with Equation 2 (ZHAO et al., 2010) were C_0 (mg/mL) is the concentration related to the original amount of roasted coffee oil inputted in the system during the microencapsulation procedure.

$$OR(\%) = \frac{C_{OR} \cdot 100}{C_0} \quad \text{Eq. (2)}$$

For the roasted coffee oil encapsulation efficiency (EE) determination 25 mL of the final suspension of microcapsules was vacuum filtered (quantitative filter paper) washed three times with dichloromethane (10 mL each one) to remove the free oil. After that the filter paper with the microcapsules was dried in an oven at 40 ºC during 1 h. Finally, the same procedure adopted to the oil recovery analysis was performed. The roasted coffee oil concentration (C_{EE}, mg/mL) was determined and the encapsulation efficiency was calculated with Equation 3 (Zhao et al., 2010).

$$EE(\%) = \frac{C_{EE} \cdot 100}{C_{OR}} \quad \text{Eq. (3)}$$

INFRARED SPECTROSCOPY

Microcapsules, the pure components (gelatin, gum Arabic, roasted coffee oil and TGase) and the physical mixture of the components were ground and Fourier Transform Infrared (FTIR) spectra were recorded using KBr pellet in a Shimadzu (IR AFFINITY-1). Spectra were obtained between 4000-400 cm^{-1} using 32 accumulations and resolution of 4 cm^{-1}.

WATER SORPTION ISOTHERM

The microcapsules produced at 350 rpm with 0.4125 g of roasted coffee oil were used to obtain the moisture sorption isotherm. After equilibrate the humidity into a glass desiccator with silica by 1 week the microcapsules were analyzed in a moisture sorption isotherm generator (Aquasorp, Decagon Devices, USA). The Guggenhein-Anderson-de Boer (GAB) model (Equation 4) was used to fit the experimental data, where Xw is the equilibrium moisture content (g water / g dry solid) at a water activity (aw), m_o is the monolayer water content, C is the Guggenheim constant, which represents the sorption heat of the first layer, and K is the sorption heat of the multilayer. The parameters of the GAB model were determined using non-linear regression, with the Statistica 7.0 software (Statsoft, USA).

$$Xw = \frac{m_0.C.K.a_w}{(1-K.a_w).(1-K.a_w+C.K.a_w)} \quad \text{Eq. (4)}$$

STATISTICAL ANALYSIS

The obtained mean results (Dp, PDI, oil recovery % and encapsulation efficiency) were evaluated by the Student's t-test with 5% significance level ($p < 0.05$) with Statistica 7.0 software (Stat-Soft, Tulsa, OK, USA).

RESULTS AND DISCUSSION

MICROCAPSULES MORPHOLOGY, AVERAGE SIZE AND SIZE DISTRIBUTIONS

The microcapsules morphology can be evaluated by the micrographs presented in Figure 1 and the size distributions are presented in Figure 2. It is possible to observe at Figure 1 that for all the experimental conditions microcapsules with well-defined spherical shape were obtained. The size distributions indicated a monomodal behavior for all samples and that the majority of the microcapsules presented diameter between 15 and 45 µm.

The results obtained for each stirring rate (350 and 500 RPM) were analyzed in function of the amount of roasted coffee oil applied the formulation. At Tables 1 and 2 the values of average diameter (Dp), polidispersity indexes (PDI), oil recovery and encapsulation efficiency are presented for the microcapsules

produced at 350 and 500 RPM respectively, as well as the resulting p-value of the t-test average difference analysis.

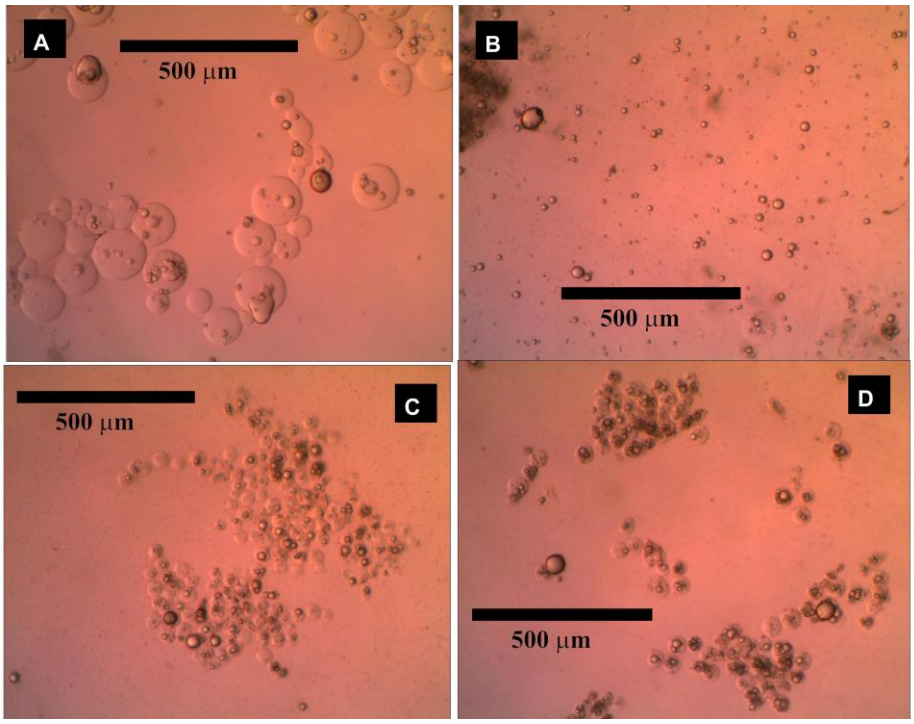

Figure 1 - Microcapsules morphology (100x magnification): (a) Exp. 1: 350 RPM and 0.8250 g oil; (b) Exp 2: 350 RPM and 0.4125 g oil; (c) Exp 3: 500 RPM and 0.4125 g oil; (d) Exp. 4: 500 RPM and 0.8250 g oil.

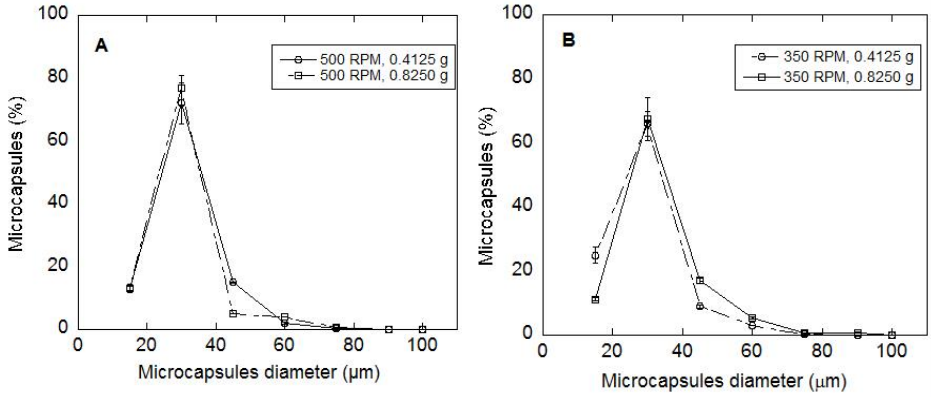

Figure 2- Microcapsules size distribution: (A) 500 RPM - (○) 0.4125 g roasted coffee oil and (□) 0.8250 g roasted coffee oil; (B) 350 RPM - (○) 0.4125 g roasted coffee oil and (□) 0.8250 g roasted coffee oil.

The average diameters obtained presented similar values for experimental conditions and it was not detected significant differences between the treatments (amount of roasted coffee oil). Bachtsi et al. (1996) stated that there is a limiting value for the average diameter that occurs when the stirring rate or

variations in other conditions from the dispersion system no longer affects it. Probably for the experimental values studied this limiting value was reached.

In the case of PDI significant differences were detected for both stirring rates in function of the roasted coffee oil amount. According to Bachtsi et al. (1996) the scattering in caused by coalescence and breakage mechanisms. In general the size of the droplets in a liquid-liquid dispersion is determined by the balance between the turbulent forces which tend to break drops and the interfacial tension and viscous forces that tend to keep the beads together, or coalesce. Coalescence occurs when the energy of adhesion between two drops is equal to or greater than the turbulent energy (SHINNAR; CHURCH, 1960). It is apparent that with increasing the stirring rate the size distribution shifts to smaller diameters as the turbulent kinetic energy associated with the breaking of the drops increases.

The narrower size distribution was obtained when the smallest amount of oil was added and the stirring rate was increased (Figure 2, a with 0.4125 g of roasted coffee oil). The wider size distribution obtained is related to the experiment conducted at 350 RPM with 0.8250 g of roasted coffee oil. In this case most of microcapsules presented diameter between 15 and 45 µm as well as a few particles up to 90 µm (Figure 2, b). The experimental conditions that leaded to the production of the largest number of microcapsules with diameter smaller than 15 µm were 350 RPM and 0.4125 g of roasted coffee oil.

ROASTED COFFEE OIL RECOVERY AND ENCAPSULATION EFFICIENCY

The roasted coffee oil recovery and encapsulation efficiency results are shown in Table 1.

Table 1 – Average diameters (Dp), polydispersity indexes (PDI), oil recovery, encapsulation efficiency and p-value (t-test, 95% significance level) of the microcapsules produced at 350 and 500 RPM.

Roasted coffee oil (g)	Dp (µm)	PDI (-)	Oil Recovery (%)	Encapsulation Efficiency (%)
		350 RPM		
0.4125	29.30±1.28	1.53±0.06	78.45±2.47	71.00±4.24
0.8250	33.10±0.46	2.08±0.14	100.70±5.23	39.20±8.20
p-value	0.0586	0.0383	0.0322	0.0396
		500 RPM		
0.4125	30.74±1.33	1.19±0.03	84.80±3.96	47.40±0.85
0.8250	29.82±0.68	1.52±0.07	85.60±4.81	32.50±3.53
p-value	0.4763	0.0272	0.8726	0.0285

Valores representam a média ± desvio padrão (n=3). Médias seguidas de letras minúsculas diferentes nas colunas diferem pelo teste de Tukey a 5% de probabilidade.

All recovery values obtained were satisfactory. Freiberger et al. (2015) encapsulated roasted coffee oil in PLLA nanocapsules and also obtained recovery values above 80% using the miniemulsion/solvent evaporation technique. A significant difference was detected for 350 RPM showing a higher oil recovery (%) when 0.8250 g of roasted coffee oil were added.

In the case of encapsulation efficiency there were detected significant differences for both cases (350 and 500 RPM) in function of the roasted coffee oil amount. The higher percentages were obtained for the lower amount of oil. The oil recovery represents the amount of oil that kept on the system after the encapsulation procedure, inside or at their surface and outside the microcapsules (free). However, the encapsulation efficiency depends of the relation between the amount of polymer available to encapsulate the oil and the surface area of the oil droplets generated during the dispersion. As the number of microcapsules produced in the experiments with higher amount of oil is higher (considering that there was not detected significant difference between Dp) a greater amount of polymer is necessary to cover their total surface. If the gelatin and gum Arabic amount used in the experiment is maintained constant and at this condition (higher total surface area) this amount is not enough to cover the surface of all droplets the encapsulation efficiency is reduced. Maji and Hussain (2009) also applied the complex coacervation technique to encapsulate *Limonella Zanthoxylum* oil in gelatin and chitosan and obtained maximum encapsulation efficiency of 60 % which is in agreement with the results obtained in the present work.

When the increase on stirring rate from 350 to 500 RPM is evaluated a decrease in the encapsulation efficiency can be observed for both roasted coffee oil amounts evaluated. Tayade and Kale (2004) also observed that the increase in the stirring rate promoted a reduction in the encapsulation efficiency as well as in the recovery of ibuprofen in gelatin micropellets.

INFRARED SPECTROSCOPY

At the FTIR spectra of roasted coffee oil (Figure 3 (A)) it is possible to observe bands related to pyrroles (ring-stretching vibrations), furans and pyridines. The bands located at 1380 and 1470 cm^{-1} are related to the ring stretching vibration of pyrrole (HUANG; KALIAGUINE, 1992). The bands at 3100 to 2750 cm^{-1} region are due to asymmetrical C-H stretching (2931 cm^{-1}), symmetrical C-H stretching (2860 cm^{-1}), and methylene asymmetrical stretching band (weak shoulder at 3010 cm^{-1}). The 1800–800 cm^{-1} region presents absorbance bands due to C=O stretching (ester, aldehydes, and ketones), C-H bending (methylene scissoring), C-O (esters and alcohol) and CH_2 stretching/bending (INNAWONG et al., 2004).

For gum Arabic the C-O stretch (at 1049 and 1426 cm^{-1}), C-O stretch and N-H bending (1612 cm^{-1}), and O-H stretch (3000-3600 cm^{-1}) can be observed (WU; CHEN, 2010). At gelatin spectra it is possible to observe the amide A, I, II and III bands at 3452, 1650, 1553 and 1237 cm^{-1}, respectively (MUYONGA et al., 2004).

At Figure 3 (B) it is possible to observer that the major difference between the spectra of the physical mixture and the microcapsules is located at 1550 cm^{-1}, corresponding to the monosubstituted amide. The band had its intensity

increased due to the crosslinking reaction promoted by TGase (MACEDO; SATO, 2005).

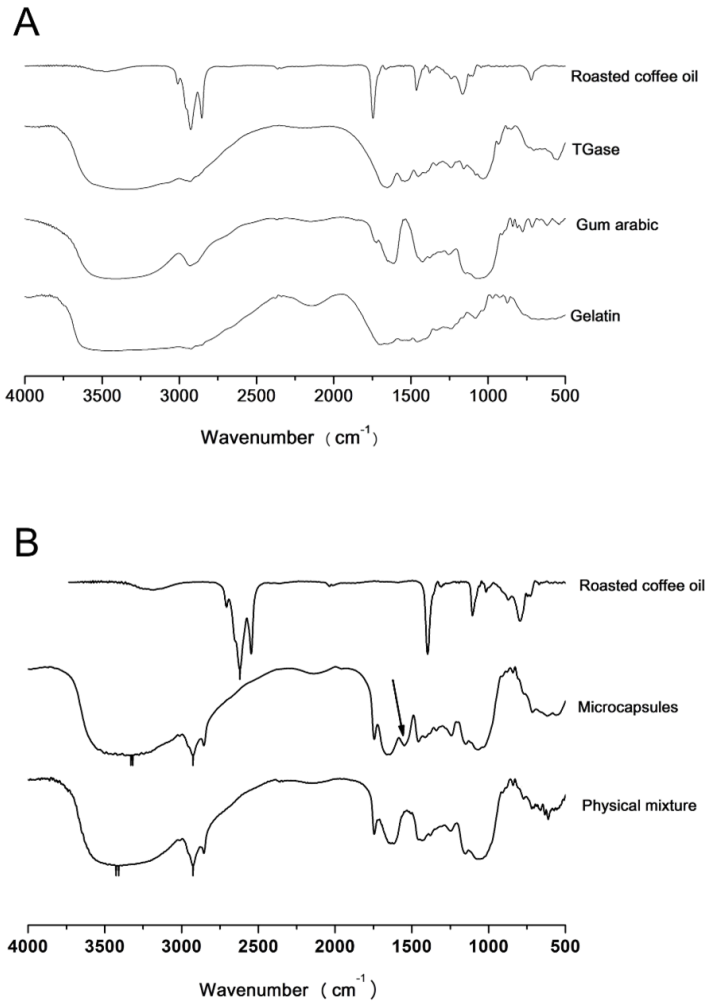

Figure 3 – FTIR spectra: (A) roasted coffee oil; TGase; gum Arabic; gelatin, and (B) roasted coffee oil; physical mixture of the components used for microcapsules preparation; microcapsules.

Gelatin presents both lysine and glutamine residual groups and the crosslinking reaction produces a monosubstituted amide (CORTESI et al., 1999; de CARVALHO; GROSSO, 2004; FUCHSBAUER et al., 1996). In this way it is possible to state that TGase acted as a crosslinking agent to microcapsules formation.

WATER SORPTION ISOTHERM

The water sorption isotherm (25 ºC) fitted to the GAB model is presented at Figure 4 and the parameters are listed in Table 2.

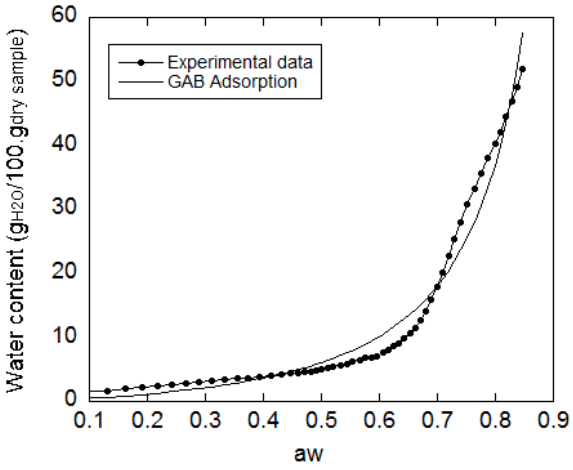

Figure 4- Water sorption isotherm of roasted coffee oil microencapsulated in crosslinked gelatin/gum Arabic.

Table 2. Parameters for the fit of the GAB model to the isotherm of the roasted coffee oil microcapsules and the coefficient of determination (R^2).

Parameter	Value
C	0.0132
K	0.9166
m_0 (gwater /100 g_{dry} solid)	2.8460
R^2	0.9892

It is possible to observe that the microcapsules presented an increase in water content with increasing water activity. Most of food water sorption isotherms have a sigmoidal shape (LEWICKI, 1997), however the obtained result indicated a "J" shape (type III according to Brunauer et al., 1940) for the roasted coffee oil microencapsulated. This type of sorption isotherm is observed frequently in the case of solids soluble in water e.g., sugars (BLAHOVEC; YANNIOTIS, 2009). This can be a good result in terms of the application of the microcapsules. During the storage conditions, where the water activity is maintained at low levels the water adsorption is low. Rocha-Selmi et al. (2013) obtained the same behavior for the water sorption isotherm of aspartame microencapsulated in crosslinked gelatin/gum Arabic by complex coacervation. The GAB model represented well the experimental data with correlation coefficient of 0.98. The monolayer moisture content (mo) indicates the maximum amount of water that can be adsorbed in a single layer per gram of dry solid and it is a measure of number sorbing sites (MALI et al., 2005). The parameter K is related to the sorption heat of the multilayer, and when k = 1 it assumed that there is no interaction of the water vapor in the multilayer or no variation in the energy of sorption in multilayer, which occur in a homogeneous solids (BRANDELERO et al., 2013). Finally the parameter C is associated with the sorption heat of the monolayer, and the value obtained was lower than reported by Rocha-Selmi et al. (2013).

CONCLUSIONS

Roasted coffee oil was successfully microencapsulated by complex coacervation in gelatin/gum Arabic crosslinked microcapsules. Oil recovery, microencapsulation efficiency, average diameter and size distributions were evaluated as a function of the production parameters, stirring rate and amount of roasted coffee oil. An increase in the amount of roasted coffee oil led to lower values of encapsulation efficiency due to a wll thinning effect. Oil recovery, encapsulation efficiency and size distribution were also affected. FTIR analysis confirmed the crosslinking of the polymeric wall of the microcapsules due to the presence of monosubstituted amide absorption bands in the microcapsules. Water sorption isotherm demonstrated that the produced microcapsules has potential to be applied in instant coffee formulations.

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Microencapsulação de Óleo de Café Torrado por Coacervação Complexa com Gelatina/Goma Arábica Enzimaticamente Reticulada

RESUMO

O óleo de café torrado é um subproduto industrial composto por um grande número de compostos incluindo os voláteis, responsáveis prlo aroma do café. O principal objetivo deste trabalho é a encapsulação do óleo de café torrado em microcápsulas de gelatina/goma arábica enzimaticamente reticuladas. As microcápsulas foram obtidas por coacervação complexa. A influência da quantidade de óleo torrado e da taxa de agitação no diâmetro médio, recuperação de óleo e eficiência de encapsulação foram avaliadas. As microcápsulas apresentaram diâmetro médio de até 29,30 ± 1,28 µm com distribuição de tamanhos estreita. Os resultados de recuperação de óleo e eficiência de encapsulação variaram de 78,45 ± 2,47 até 100,70 ± 5,23 % e de 32,50 ± 3,53 até 71,00 ± 4,24 % respectivamente. A isoterma de sorção de água das microcápsulas foi avaliada a 25 ºC e os resultados foram ajustados ao modelo de Guggenhein-Anderson-de Boer (GAB) apresentado R^2 igual a 0,9892 e comportamento tipo "J". Finalmente a reação de reticulação foi investigada com Espectroscopia de Infravermelho com Transformada de Fourier, mostrando que a reação de reticulação promovida pela transglutaminase introduziu uma banda no espectro localizada em 1550 cm^{-1} correspondendo a amida monosubstituída

PALAVRAS-CHAVE: Óleo de Café Torrado; Microencapsulação, Coacervação Complexa..

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Correspondência:
Fernanda Vitória Leimann
Programa de Pós Graduação e Tecnologia de Alimentos - Universidade Tecnológica Federal do Paraná Campus Campo Mourão - Via Rosalina Maria dos Santos, 1233 CEP 87301-899 Caixa Postal: 271 – Campo Mourão - PR, Brasil.