OPTIMIZATION OF THE INFEEED FORMULA FOR SPENT COFFEE GROUND EXTRACT ENCAPSULATION USING THE RESPONSE SURFACE METHODOLOGY

OPTIMIZACIJA INFEEED FORMULE ZA KAPSULIRANJE EKSTRAKTA IZ ISKORIŠĆENE MLEVENE KAFE UPOTREBOM RESPONSE SURFACE METHODOLOGY

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ABSTRACT

The purpose of this paper is to optimize the ratio of wall materials (chitosan (CH): maltodextrin (MD), X_1) and spent coffee ground extract (SCGE) to the complex wall material (X_2), i.e. the so-called infeed formula for the SCGE encapsulation by freeze drying. The response surface methodology (RSM), based on the central composite design (CCD), was applied to obtain the infeed formula, providing the best encapsulated SCGE properties (encapsulation yield (EY), hygroscopicity (HG), water solubility index (WSI), total phenolic compounds (TPC) and encapsulation efficiency (EE)). Multiple regression analysis was performed to derive a second-order polynomial equation for predicting properties. The EY ($R^2 = 0.80$), HG ($R^2 = 0.95$), WSI ($R^2 = 0.96$) and TPC ($R^2 = 0.97$) fitted the data well, and was ultimately used to plot a response surface. The plots obtained indicate that the EY and WSI significantly increased during the experimental process, whereas the HG and TPC decreased with the decreasing X_1 and X_2 ($p \leq 0.05$). The equations were used to optimize the infeed formula. The results obtained show that the infeed formula with the CH-to-MD of 30:70 and the SCGE-to-the-wall material of 90:10 would provide the most favorable outcomes (EY (80 %), HG (17g/100 g SCGE db), WSI (88 %) and TPC (5.27 g gallic acid equivalent/100 g SCGE db)).

Key words: chitosan, freeze drying, maltodextrin, total phenolic compounds.

REZIME

Cilj ovog rada je optimizacija odnosa nosećeg matrijala (chitosan (CH): maltodextrin (MD), X_1) i ekstrakta iskorišćene mlevene kafe (SCGE) do kompleknog nosećeg materijala (X_2), to jest takozvane in-feed formule za enkapsulaciju SCGE liofilizacijom. Metoda odzivnih površina, zasnovana na centralnom kompozitnom dizajnu, korišćena je radi dobijanja infeed formule, obezbeđujući najbolje osobine enkapsuliranog SCGE (prinos enkapsulacije (EY), higroskopnost (HG), indeks rastvorljivosti u vodi (WSI), sadržaj ukupnih fenola (TPC) i efikasnost enkapsulacije (EE)). Višestruka regresiona analiza izvedena je da bi se dobio polinom drugog reda radi predviđanja ispitivanih osobina. EY ($R^2 = 0.80$), HG ($R^2 = 0.95$), WSI ($R^2 = 0.96$) i TPC ($R^2 = 0.97$) dobro se poklapaju sa eksperimentalnim podacima i korišćeni su za iscrtavanje odzivnih površina. Dobijeni grafici ukazuju da EY i WSI značajno rastu tokom eksperimentalnog procesa, dok se HG i TPC smanjuju sa smanjenjem X_1 i X_2 ($p \leq 0.05$). Jednačine su korišćene za optimizaciju infeed formule. Dobijeni rezultati pokazuju da infeed formula sa odnosom CH:MD - 30:70 i SCGE:noseći materijal - 90:10 može obezbediti najbolje izlazne rezultate (EY (80 %), HG (17g/100 g SCGE db), WSI (88 %) and TPC (5.27 g gallic acid equivalent/100 g SCGE db)).

Cljučne reči: citozan, sušenje zamrzavanjem, maltodekstrin, sadržaj ukupnih fenola.

INTRODUCTION

Spent coffee ground (SCG) is a by-product of the coffee brewing process, yielding approximately 1.86 kg of SCG per a kilogram of the coffee ground produced (Murthy and Naidu, 2012). Nowadays, coffee consumption is increasing in around the world, including Thailand. Consequently, the amount of SCG is also increasing which is normally disposed for land filled or used as a raw material to produce inexpensive and easily available adsorbent, activated carbon, deodorant, skin exfoliate and scrubber (Kourmentza et al., 2018). Various authors have reported that SCG is a rich source of bioactive compounds (Klangpetch, 2017; Ramalakshmi et al., 2009). Those compounds include caffeine, trigonelline and chlorogenic acid, etc. (Esquivel and Jiménez, 2012), which are sensitive to heat, light, moisture and oxygen (thus limiting their application in the food industry) (Acevedo et al., 2013).

Encapsulation has been reported to facilitate the protection of bioactive compounds in raw materials from degradation during processing and storage, while concealing unpleasant flavor and

taste (Nedovic et al., 2011). There are various encapsulation methods used in the food industry depending on the type of core and wall materials, size of desired capsules and applications. Freeze drying is the most common of the all encapsulation techniques stated above on account of its ability to preserve the bioactive compounds even though such process is not thermal (Wilkowska et al., 2016). The most common wall materials used in the food industry include carbohydrates (starch, maltodextrins, chitosan, dextran, cyclodextrins), proteins (gluten, casein, gelatin, albumin, whey protein, soy protein) and lipids (wax, paraffin, oils, fats) (Fang and Bhandari, 2010). Proteins usually exhibit favorable emulsion properties. However, some proteins are relatively expensive and potentially allergenic (Shahidi and Han, 1993). Carbohydrates are usually used as wall materials due to their low cost and non-allergic properties. Maltodextrin (MD) is one of the most commonly used wall material featuring high water solubility and film form ease properties (Che Man et al., 1999). MD is also able to protect the core material from oxygen, and thus has been used for the oxygen sensitive, bioactive compound encapsulation (Desai and

Park, 2005). Carbohydrates, particularly hydrolyzed starch, are normally deprived of emulsification properties and have poor flavor refection (Özkan and Bilek, 2014). These problems can be overcome using complex wall materials (containing two or more wall materials), which would be properly modified or used in combination with other encapsulating agents. Chitosan (CH) has attracted much attention due to its biocompatibility, biodegradability, nontoxic nature, low immunogenicity and antibacterial properties. However, CH is normally insoluble in water above pH 6 due to its rigid crystalline structure and requires acids to be protonated (Saloko et al., 2013). Therefore, the complex CH-MD was applied as a wall material for the encapsulation of sensitive core ingredients in order to improve their encapsulation efficiency (Yinbin et al., 2018). However, a variety of factors could affect the capsule properties obtained from the freeze drying process, particularly the type of wall material and the ration of core-to-wall material. Thus far, there have been no reports on the encapsulation of SCGE using the CH-MD complex wall material, particularly in the freeze drying process. The purpose of this paper is to optimize the ratio of wall materials (CH-MD) and SCGE to the complex wall material for encapsulation of SCGE by freeze drying using the response surface methodology (RSM) in order to maximize the bioactive compounds in the encapsulated SCGE.

MATERIAL AND METHOD

Reagents

The Folin-Ciocalteu's phenol reagent and gallic acid standard were purchased from Sigma Aldrich Co. Ltd. Sodium carbonate was purchased from AjaxFinechem (Australia). The encapsulating agents, maltodextrin (MD; food grade DE 10- 12 %) and chitosan (CH; food grade) were purchased from S. Science Equipment Chemical Limited Partnership (Thailand). Spent coffee ground (SCG) was obtained from Inthanin Café, a coffee shop in Burapha University, Chonburi, Thailand. The SCG was dried at 60 °C to a moisture content of 13 %. The dried SCG was stored at -20 °C until further analysis.

Preparation of the aqueous spent coffee ground extract

The dried SCG was mixed with distilled water (DW) at the SCG-to-DW ratio of 1:10. The temperature of the mixture was heated up to the temperature of 90 °C and maintained at the temperature for 5 min while stirred occasionally. Then, the mixture was filtered through filter paper to obtain coffee ground extract (SCGE) (Aprilia et al., 2014). The supernatant was collected and stored at -20 °C until used.

Experimental design

The response surface methodology (RSM) was applied in order to investigate the effect of encapsulation conditions (i.e. the ratio of wall materials (CH: MD) and SCGE to the complex wall material) on the encapsulated SCGE properties (the encapsulation yield (EY, Y_1), hygroscopicity (HG, Y_2), water solubility index (WSI, Y_3), total phenolic compound (TPC, Y_4) and encapsulation efficiency (EE, Y_5)). Central composite design (CCD) was employed to analyze the combined effects of these parameters and to find the region providing the best condition. A summary of the variables and their levels of variation are presented in Table 1. The encapsulated SCGEs were examined relative to the response variables (EY, HG, WSI, and TPC) and encapsulation efficiency (EE). The results obtained were used to determine the coefficients and p -value of each condition in order to obtain a regression equation of the response variables. The regression equation was used to generate a response surface of each response variable, thus the infeed formula optimization for the SCGE encapsulation was obtained.

Table 1. Range of variables at different levels for the central composite design

Infeed formula	Levels				
	-1.414	-1	0	+1	+1.414
X_1 ; ration of wall materials (CH:MD) (%)	0:100	14:86	50:50	86:14	100:0
X_2 ; ration of SCGE to the complex wall material (CH:MD) (%)	0:100	14:86	50:50	86:14	100:0

*CH:chitosan, MD: maltodextrin, SCGE: spent coffee ground extract

Encapsulation procedure

The CH solution (0.2 % w/v) and MD solution (12 % w/v) were prepared in 100 mM acetate buffer (pH 4.0). The SCGE was mixed with CH and MD solutions according to the ratio shown in Table 1. The mixture was homogenized (12000 rpm for 5 min), and then evaporated at 50 °C using a rotary evaporator under reduced pressure to obtain crude extract. The crude extract was then subjected to freeze drying at -100 °C for 24 hrs. The encapsulated SCGE was collected in an airtight container and stored at -20 °C until used (Kaasgaard and Keller, 2010).

Encapsulation yield determination

The EY was calculated and expressed as the percentage of total solid of the mixture before encapsulation per weight of the encapsulated SCGE dry basis (db) (Marcela et al., 2016).

Hygroscopicity determination

The encapsulated SCGE (1.0 g) was weighed in a pre-weighed Petri dish. The Petri dish was placed in an airtight plastic container containing saturated NaCl solution. The container was stored at temperatures of 30 ± 2 °C and 75% RH until equilibrium. The encapsulated SCGE was then subjected to moisture content determination. The HG of the encapsulated SCGE was calculated and expressed as the absorbed moisture per 100 g of the encapsulated SCGE (db) (Cai and Corke, 2000).

Water solubility index determination

The encapsulated SCGE (1.0 g) was dissolved in distilled water (100 mL) and then the mixture was centrifuged at 3000 rpm for 10 min. The supernatant was carefully transferred in a pre-weighed can and dried at 105 °C for 16- 18 hrs in a hot air oven. The WSI was calculated as the percentage of the dried solid per 100 g of the encapsulated SCGE (db) (Anderson et al., 1970).

Total phenolic compound determination

The encapsulated SCGE was dissolved in distilled water to obtain the appropriate concentration. Subsequently, the encapsulated SCGE solution (0.5 mL) was mixed with 2.5 mL of 10 % v/v Folin-Ciocalteu's reagent and 2.0 mL of 7.5 % w/v sodium carbonate. The mixture was stored at ambient temperature for 60 min before the absorbance was determined at a wavelength of 765 nm. The TPC was expressed as the gram gallic acid equivalent (GAE)/ 100 g of the encapsulated SCGE (db)(Singleton et al., 1999).

Encapsulation efficiency determination

The EE was calculated and expressed as the percentage of the TPC differentiation in the encapsulated SCGE and the wall materials used for encapsulation (Marcela et al., 2016).

Statistical analysis

All the experiments were carried out in triplicate and reported as the mean $\pm$ standard deviation (SD). The optimization data were analyzed using the Minitab® 17 software and the Tukey's comparison test at the $p \leq 0.05$ significance level.

RESULTS AND DISCUSSION

Optimization of physicochemical parameters

RSM was applied to determine the effect of infeed formula, i.e. the ratio of wall materials (CH: MD) and SCGE to the complex wall material, on the encapsulated SCGE properties (EY, HG, WSI, TPC and EE). The encapsulated SCGE properties (EY, HG, WSI, TPC and EE) obtained from the infeed formula designed by CCD are shown in Table 2. The multiple regression analysis was employed to derive a second-order polynomial equation to predict the encapsulated SCGE properties. Table 3 shows the regression equations for the response variables with the coefficient of multiple determination (R^2) and coefficient p -values. The regression equation with the coefficient p -value of less than 0.05 and R^2 of more than 0.80 indicates that the regression model fits the data well (Joglekar and May, 1987). In order to plot a response surface, only the statistically significant coefficients of the response variables were selected for optimization: EY ($R^2 = 0.80$), HG ($R^2 = 0.95$), WSI ($R^2 = 0.96$) and TPC ($R^2 = 0.97$).

Effect of the ratio of wall materials (CH: MD) and SCGE to the complex wall material on the the encapsulated SCGE properties

The 3D response surfaces plots generated regarding to the effect of infeed formula, i.e. the ratio of wall materials (CH: MD) (X_1) and SCGE to the complex wall material (X_2), on the encapsulated SCGE properties (encapsulation yield, (EY) hygroscopicity, (HG) water solubility index (WSI), and total phenolic compounds (TPC)) are shown in Fig 1. The results show that the EY significantly increased as the ratio of wall material (CH: MD) and SCGE to the complex wall material decreased. These results may be accounted for by the fact that at the lower X_1 and X_2 , the higher MD and wall materials in the infeed solution was applied, leading to an increase in the EY value (Fig 1A). The HG is a crucial feature of microencapsulated products as it could promote oxidation of the encapsulated powder (Rodríguez-Hernández et al., 2005). Therefore, the lower HG could help reduce the bioavailability loss of the encapsulated SCGE. The results obtained show that the HG of the encapsulated SCGE significantly decreased as the ratio of wall materials (CH: MD) (X_1) and SCGE to the complex wall material (X_2) of the encapsulated SCGE decreased (Fig 1B) ($p \leq 0.05$). These results may be due to the action of the higher MD in the infeed formula as the X_1 and X_2 decreased. MD is a substance that facilitates the reduction of the water absorption

Table 3. Regression equations, coefficient of multiple determinations (R^2), and coefficient p -values of the response variables for the infeed formula optimization of the SCGE encapsulation by freeze drying

Response variables	Regression equations	R^2	p -value*
EY	$Y_1 = 90.27 - 9.02X_1 - 2.92X_2 - 4.68X_1^2 - 8.37X_2^2$	0.80	0.026
HG	$Y_2 = 17.816 + 4.666X_1 + 0.311X_2 + 3.058X_1^2 + 0.423X_2^2$	0.95	0.001
WSI	$Y_3 = 85.47 - 6.230X_1 - 1.720X_2 - 3.606X_1^2 + 1.402X_2^2$	0.96	0.000
TPC	$Y_4 = 0.590 + 0.565X_1 + 2.422X_2 + 0.302X_1^2 + 1.601X_2^2$	0.97	0.000
EE	$Y_5 = 98.3 + 0.27X_1 + 18.63X_2 + 5.8X_1^2 - 18.2X_2^2$	0.61	0.162

*Significant at $p \leq 0.05$, EY: encapsulation yield, HG: hygroscopicity, WSI: water solubility index, TPC: total phenolic compound and EE: encapsulation efficiency.

ability of materials, thus reducing the HG of the product (Cai and Corke, 2000). In addition, MD influences the balance of the hydrophilic and hydrophobic system by increasing the hydrophobicity of the powder, thus also reducing the HG of the encapsulated SCGE (Farahnaky et al., 2016). In contrast to the HG, the WSI of the encapsulated SCGE increased as the ratio of wall materials (CH: MD), (X_1) and SCGE to the complex wall material (X_2) decreased (Fig 1C). These results are consistent with the results of Vidović et al. (2014), who reported that higher MD provided higher HG and lower WSI of the encapsulated product. WSI determines the amount of soluble components released from the encapsulated powder. Higher WSI of the powder indicates better rehydration abilities of the powder (Cano-Chauca et al., 2005). MD has been reported to provide higher porosity of the encapsulated powder, consequently increasing the surface area to absorb more water during rehydration process (Suriyajunhom and Phongpipatpong, 2017). In addition, the MD with lower dextrose equivalent could also contribute to higher WSI of the powder (Fazaeli et al., 2012). In addition, the results showed that the TPC of the encapsulated SCGE powder exhibited lower than that of the non-encapsulated SCGE powder, which is consistent with those previously reported by Ballesteros et al. (2017). Furthermore, the TPC of the encapsulated SCGE powder significantly increased as the ratio of wall materials (CH: MD) (X_1) and SCGE to the complex wall material (X_2) increased (Fig 1D). Higher values of the X_1 and X_2 indicate higher CH and higher SCGE, respectively. Higher TPC values may be accounted for by the fact that the interaction between gallic acid or other phenolic compounds in the extract, and the CH via hydrogen bonds, consequently increases the TPC of the encapsulated SCGE powder (da Rosa et al., 2013).

Table 2. Infeed formula, the ratio of wall materials (CH: MD) (X_1) and SCGE to the complex wall material (X_2), and the encapsulated SCGE properties (EY, HG, WSI, TPC and EE)

Runs	X_1	X_2	EY (%)	HG (g/100 g SCGE db)	WSI (%)	TPC (g GAE/100 g CGEdb)	EE (%)
1	86:14 (+1)	86:14 (+1)	69.95 ± 0.88	24.07 ± 0.12	77.27 ± 0.83	5.33 ± 0.05	99.81 ± 0.00
2	86:14 (+1)	14: 86 (-1)	71.26 ± 0.98	26.44 ± 0.20	79.27 ± 0.11	0.36 ± 0.02	97.18 ± 0.13
3	14: 86 (-1)	86:14 (+1)	91.18 ± 0.57	18.31 ± 0.06	85.23 ± 0.76	3.42 ± 0.02	99.71 ± 0.00
4	14: 86 (-1)	14: 86 (-1)	80.33 ± 0.84	17.09 ± 0.07	90.45 ± 0.07	0.19 ± 0.00	94.75 ± 0.13
5	100:0 (+1.414)	50:50 (0)	64.11 ± 0.80	31.60 ± 0.04	67.26 ± 0.76	2.22 ± 0.01	97.75 ± 0.01
6	0:100 (-1.414)	50:50 (0)	94.45 ± 0.98	15.90 ± 0.12	89.33 ± 0.72	0.50 ± 0.01	98.01 ± 0.03
7	50:50 (0)	100:0 (+1.414)	60.21 ± 0.77	19.77 ± 0.08	86.11 ± 0.79	7.91 ± 0.13	100.00 ± 0.00
8	50:50 (0)	0:100 (-1.414)	83.45 ± 0.83	17.19 ± 0.13	90.48 ± 0.88	0.01 ± 0.00	0.00 ± 0.00
9	50:50 (0)	50:50 (0)	90.68 ± 0.89	17.86 ± 0.12	85.84 ± 0.39	0.59 ± 0.01	98.30 ± 0.02
10	50:50 (0)	50:50 (0)	89.82 ± 0.73	17.83 ± 0.06	84.59 ± 0.34	0.60 ± 0.00	98.33 ± 0.01
11	50:50 (0)	50:50 (0)	90.10 ± 0.8	17.75 ± 0.20	85.34 ± 0.19	0.58 ± 0.00	98.29 ± 0.01

*Mean ± SD with in the same row, means in a column with different letters are statistically difference ($p \leq 0.05$), EY: encapsulation yield, HG: hygroscopicity, WSI: water solubility index, TPC: total phenolic compound and EE: encapsulation efficiency.

Fig 1. 3D Response surface optimization of the infeed formula for the SCGE encapsulation by freeze drying, the ratio of wall materials (CH: MD) (X_1) and SCGE to the complex wall material; (X_2) on the encapsulated SCGE properties; (A) encapsulation yield, (B) hygroscopicity, (C) water solubility index, and (D) total phenolic compounds

Infeed formula optimization for the SCGE encapsulation

The regression equations (Table 3) were used to optimize the infeed formula for the SCGE encapsulation as shown in Table 4. The best predicted response variables were obtained when the ratio of CH-to-MD was 30: 70 and the ratio of the SCGE-to-the complex wall material was 90: 10. With this optimal formulation, the EY, HG, WSI and TPC would be 80.00 %, 17.00 g/ 100 g SCGE db, 88.05% and 5.27 g GAE/ 100 g SCGE db, respectively.

order polynomial equation to predict the encapsulated SCGE properties. The second-order linear and square models were generated and found to be statistically adequate to describe and predict the responses for the EY ($R^2 = 0.80$), HG ($R^2 = 0.95$), WSI ($R^2 = 0.96$) and TPC ($R^2 = 0.97$). The best encapsulated SCGE properties were obtained when the CH-to-MD ratio was 30: 70 and the SCGE-to-the complex wall material ratio was 90: 10. With this optimal formulation, the EY, HG, WSI and TPC would be 80.00 %, 17.00 g/ 100 g SCGE db, 88.05% and 5.27 g GAE/ 100 g SCGE db, respectively.

Table 4. Single and multi-response variables of the encapsulated SCGE as affected by the ratio of wall materials (CH:MD, X_1) and SCGE to the complex wall material (X_2)

Response variables	Variable codes		Variable values		Predicted response variables			
	X_1	X_2	X_1	X_2	EY (%)	HG (g/100 g SCGE db)	WSI (%)	TPC (g GAE/100 g SCGE db)
Single response (EY)	-0.96	-0.19	34/66	43/56	94.87	-	-	-
Single response (HG)	-0.76	-0.36	27/78	37/63	-	15.98	-	-
Single response (WSI)	-0.87	-1.41	30/70	0/100	-	-	93.40	-
Single response (TPC)	1.41	1.41	100/0	100/0	-	-	-	8.62
Multi response (EY+ HG+ WSI+ TPC)	-0.87	1.16	30/70	90/10	80.00	17.00	88.05	5.27

*EY: encapsulation yield, HG: hygroscopicity, WSI: water solubility index, TPC: total phenolic compound

CONCLUSION

The infeed formula for the SCGE encapsulation by freeze drying was successfully optimized using the RSM via CCD. A multiple regression analysis was employed to derive a second-

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REFERENCES

- Acevedo, F., Rubilar, M., Scheuermann, E., Cancino, B., Uquiche, E., Garcés, M., Inostroza, K., Shene, C. (2013). Spent coffee grounds as a renewable source of bioactive compounds. *Journal of Biobased Materials and Bioenergy*, 7(3), 420-428.
- Anderson, R., Conway, H., Peplinski, A. (1970). Gelatinization of corn grits by roll cooking, extrusion cooking and steaming. *Starch-Stärke*, 22(4), 130 -135.
- Aprilia, A. A., Jaisan, C., Punbusayakul, N. (2014). Antimicrobial and antioxidant activities of microwave-assisted extracts from coffee ground residue. *Proceeding of the 1st Joint ACS AGFD- ACS ICSCT Symposium of Agricultural and Food Chemistry*, 292-298.
- Ballesteros, L. F., Ramirez, M. J., Orrego, C. E., Teixeira, J. A., Mussatto, S. I. (2017). Encapsulation of antioxidant phenolic compounds extracted from spent coffee grounds by freeze-drying and spray-drying using different coating materials. *Food Chemistry*, 237, 623-631.
- Cai, Y. Z., Corke, H. (2000). Production and properties of spray-dried amaranthus betacyanin pigments. *Journal of Food Science*, 65(7), 1248-1252.
- Cano-Chauca, M., Stringheta, P., Ramos, A., Cal-Vidal, J. (2005). Effect of the carriers on the microstructure of mango powder obtained by spray drying and its functional characterization. *Innovative Food Science and Emerging Technologies*, 6(4), 420-428.
- Che Man, Y. B., Irwandi, J., Abdullah, W. J. W. (1999). Effect of different types of maltodextrin and drying methods on physico-chemical and sensory properties of encapsulated durian flavour. *Journal of the Science of Food and Agriculture*, 79(8), 1075-1080.
- Da Rosa, C. G., Borges, C. D., Zambiasi, R. C., Nunes, M. R., Benvenuti, E. V., da Luz, S. R., D'Avila, R. F., Rutz, J. K. (2013). Microencapsulation of gallic acid in chitosan, β -cyclodextrin and xanthan. *Industrial crops and products*, 46, 138-146.
- Desai, K. G. H., Park, H. J. (2005). Recent developments in microencapsulation of food ingredients. *Drying Technology*, 23(7), 1361-1394.
- Esquivel, P., Jiménez, V. M. (2012). Functional properties of coffee and coffee by-products. *Food Research International*, 46(2), 488-495.
- Fang, Z., Bhandari, B. (2010). Encapsulation of polyphenols- a review. *Trends in Food Science and Technology*, 21(10), 510-523.
- Farahnaky, A., Mansoori, N., Majzoobi, M., Badii, F. (2016). Physicochemical and sorption isotherm properties of date syrup powder: Antiplasticizing effect of maltodextrin. *Food and bioproducts processing*, 98, 133-141.
- Fazaali, M., Emam-Djomeh, Z., Ashtari, A. K., Omid, M. (2012). Effect of spray drying conditions and feed composition on the physical properties of black mulberry juice powder. *Food and bioproducts processing*, 90(4), 667-675.
- Joglekar, A., May, A. (1987). Product excellence through design of experiments. *Cereal Foods World*, 32(12), 857-868.
- Kaasgaard, T., Keller, D. (2010). Chitosan coating improves retention and redispersibility of freeze-dried flavor oil emulsions. *Journal of Agricultural and Food Chemistry*, 58(4), 2446-2454.
- Klangpetch, W. (2017). Evaluation of antioxidant, antipathogenic and probiotic growth stimulatory activities of spent coffee ground polyphenol extracts. *International Food Research Journal*, 24(5).
- Kourmentza, C., Economou, C. N., Tsafrakidou, P., Kornaros, M. (2018). Spent coffee grounds make much more than waste: Exploring recent advances and future exploitation strategies for the valorization of an emerging food waste stream. *Journal of Cleaner Production*, 172, 980-992.
- Marcela, F., Lucía, C., Esther, F., Elena, M. (2016). Microencapsulation of L-ascorbic acid by spray drying using sodium alginate as wall material. *Journal of Encapsulation and Adsorption Sciences*, 6, 1-8.
- Murthy, P. S., Naidu, M. M. (2012). Sustainable management of coffee industry by-products and value addition-A review. *Resources, Conservation and Recycling*, 66, 45-58.
- Nedovic, V., Kalusevic, A., Manojlovic, V., Levic, S., Bugarski, B. (2011). An overview of encapsulation technologies for food applications. *Procedia Food Science*, 1, 1806-1815.
- Özkan, G., Bilek, S. E. (2014). Microencapsulation of natural food colourants. *International Journal of Nutrition and Food Sciences*, 3(3), 145-156.
- Ramalakshmi, K., Rao, L. J. M., Takano-Ishikawa, Y., Goto, M. (2009). Bioactivities of low-grade green coffee and spent coffee in different in vitro model systems. *Food Chemistry*, 115(1), 79-85.
- Rodríguez-Hernández, G., Gonzalez-Garcia, R., Grajales-Lagunes, A., Ruiz-Cabrera, M., Abud-Archila, M. (2005). Spray-drying of cactus pear juice (*Opuntia streptacantha*): effect on the physicochemical properties of powder and reconstituted product. *Drying Technology*, 23(4), 955-973.
- Saloko, S., Darmadji, P., Setiaji, B., Pranoto, Y., Anal, A. (2013). Encapsulation of coconut shell liquid smoke in chitosan-maltodextrin based nanoparticles. *International Food Research Journal*, 20(3).
- Shahidi, F., Han, X. Q. (1993). Encapsulation of food ingredients. *Critical Reviews in Food Science and Nutrition*, 33(6), 501-547.
- Singleton, V. L., Orthofer, R., Lamuela-Raventós, R. M. (1999). Analysis of total phenols and other oxidation substrates and antioxidants by means of folin-ciocalteu reagent. In *Methods in Enzymology* (Vol. 299, pp. 152-178): Elsevier.
- Suriyajunhom, P., Phongpipatpong, M. (2017). Effect of drying temperature, maltodextrin and gum arabic on physiochemical characteristics of Gac powder produced by double drum drying. *The 18th TSAE National Conference and the 10th TSAE International Conference*, 243-249.
- Vidović, S. S., Vladić, J. Z., Vaštag, Ž. G., Zeković, Z. P., Popović, L. M. (2014). Maltodextrin as a carrier of health benefit compounds in *Satureja montana* dry powder extract obtained by spray drying technique. *Powder Technology*, 258, 209-215.
- Wilkowska, A., Ambroziak, W., Czyżowska, A., Adamiec, J. (2016). Effect of microencapsulation by spray-drying and freeze-drying technique on the antioxidant properties of blueberry (*Vaccinium myrtillus*) juice polyphenolic compounds. *Polish Journal of Food and Nutrition Sciences*, 66(1), 11-16.
- Yinbin, L., Wu, L., Weng, M., Tang, B., Lai, P., Chen, J., (2018). Effect of different encapsulating agent combinations on physicochemical properties and stability of microcapsules loaded with phenolics of plum (*Prunus salicina* lindl.). *Powder Technology*, 340, 459-464.

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