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Optimization of carriers (maltodextrin, arabic gum) for spray-drying of *Pouzolzia zeylanica* extracts using response surface methodology

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ABSTRACT

Pouzolzia zeylanica is a medicinal source that people of Asian countries have used to treat various kinds of diseases by traditional method. A lot of research showed that Pouzolzia zeylanica extract contains many bioactive compounds with antioxidant, antimicrobial and antifungal properties. In spray drying process, the carrier was significant effect on physicochemical characteristics. The response surface methodology (RSM) with central composite design (CCD) was applied to optimize maltodextrin (5÷15%) and arabic gum percent (0.06÷0.10%) during spray drying of Pouzolzia zeylanica extract. The physico-chemical characteristics of spray dried powder (bioactive compounds, moisture content as well as particle size distribution) were analyzed.

The results showed that the optimum concentrations of maltodextrin and arabic gum were 8.743% and 0.083%, respectively. At these optimal conditions, the anthocyanin, flavonoid, polyphenol, tannin, moisture content and particle size of obtained spray dried powder were 7.411 mg CE/100g; 30.931 mg QE/g; 27.296 mg GAE/g; 24.654 mg TAE/g, 6.540% and 6.029 µm, respectively.

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1 INTRODUCTION

Nowadays, the economic development has changed the trend of food consumption from calories assurance to nutrition-enriched diet. Consumers are interested in natural products which were made from medicinal plant sources. Herbal medicine is the most ancient form of health care known to humankind and it always played an important role as remedies in the treatment of human ailments. The World Health Organization has estimated that 80% of people from all over the world rely upon the traditional medicine or herbal medicine for their primary health care needs (Tee *et al*., 2012).

Pouzolzia zeylanica can be found in the Mekong delta. It is cultivated alternate in fruit garden. In Vietnam, this herb can be used as fresh or dried plant, decoction drunk to treat cough, pulmonary tuberculosis, sore throat, enteritis, dysentery (Võ Văn Chi, 2012). In Indian, leaf and stem paste is applied locally once or twice daily for itching (Bhattacharjya and Borah, 2008); paste of crushed shoots is applied as poultice to bone fractures (Ratnam and Raju, 2008).

Spray drying has been widely used in pharmaceutical and food industries in dehydration of liquid foods such as coffee and fruit juices. Spray drying will form powders with low water activity and ease in transportation and storage. The physicochemical properties of spray-dried powders depend on the process variables such as the characteristic of liquid feed including feed viscosity, flow rate and the drying air in term of pressure and temperature as well as the type of atomizer (Tee *et al*., 2012). Besides, spray drying was also used in the microencapsulation of food ingredients susceptible to deterioration by external agents and consists of entrapping an active agent (solid particles, liquid droplets or gaseous compounds) in polymeric matrix, in order to protect bioactive compounds from adverse conditions. The immediate drying of the mixture led to the formation of a matrix system in which the polymer formed a tridimensional network which contained the encapsulated material (Tonon *et al*., 2010). Bayberry powder was successfully obtained when the juice was spray dried with maltodextrin as the carrier and suggested that spray drying was a satisfactory technique for drying heat sensitive polyphenols (Fang and Bhandari, 2012). The aim of this study was to evaluate the effects of carrier types *i. e.* maltodextrin, arabic gum and their added percent on the anthocyanin, flavonoid, polyphenol, tannin, moisture content and particle size distribution of spray dried powder from *Pouzolzia zeylanica* extracts.

2 MATERIALS AND METHODS

2.1 Chemicals and reagents

Folin-Ciocalteu, Folin-Denis reagents and quercetin, gallic acid, tannic acid were obtained from Sigma Chemical Co. (USA) and Merck Chemical Supplies (Germany). All chemicals and solvents were of analytical grade.

2.2 Sample preparation

The dried samples were extracted with water using airtight extractor (model GPA CC1-181907, Didatec Technologie France, 2007). The stirring rate, temperature, time and solution to solid ratio of extraction sample were maintained at 90 rpm, 81°C, 30 min and 27:1 v/w, respectively. The extract was filtered by cloth and determined their volumes. After that, the extract was blended with maltodextrin and arabic gum concentration following experimental design before undergoing spray drying process. The inlet hot air temperature and feed flow speed of spray drying process were kept (180°C and 18 rpm) in this study based on a preliminary study (notes: 10 rpm is 350 ml/hr and 20 rpm is 600 ml/hr)

2.3 Equipment

Spray drying process was carried out in a laboratory scale spray dryer (SD-05, LabPlant™, United Kingdom), with co-current flow regime (the spray dried product and the drying air flow are in the same direction). The drying chamber has diameter of 215 mm and height of 500 mm. The mixture was fed into the main chamber through a peristaltic pump and the feed flow rate was controlled, internal diameter of which was 0.5 mm. Powder collection system by cyclone was used to recovery the dried product. The flow rate of drying air was fixed at 60 m^3h^{-1} and the atomizing air at a pressure of 1.1 bar.

2.4 Physicochemical properties of sample analysis

Powder product characteristics

Residual moisture content and total content solids of the product were measured using the infrared humidity analyzer (model AND MS-50, Japan). The particle size of the different samples were obtained in the particle analyzer (model ZEOL-5500, Japan).

Bioactive compounds analysis

The anthocyanin content was determined using the pH differential method (Ahmed *et al*., 2005; Santos *et al*., 2013). The results were expressed as mg cyanidin-3-glycoside equivalents (CE) per gram product. The aluminum chloride colorimetric method was used for flavonoids determination and the amount of flavonoid was calculated as quercetin equivalent (QE) per gram of product (Eswari *et al*., 2013; Mandal *et al*., 2013). The polyphenol content was determined by Folin-Ciocalteu reagent method and the results were expressed as milligrams of gallic acid equivalents (GAE) per gram of product (Hossain *et al*., 2013). Tannin content was determined by Folin-Denis method and the results were showed as milligrams of tannic acid equivalents (TAE) per gram of product (Laitonjam *et al.,* 2013).

2.5 Experimental design and data analysis

In order to assess the effect of maltodextrin (5 to 15%) and arabic gum rate (0.06 to 0.10%) on moisture content, particle size distribution and bioactive compounds (anthocyanin, flavonoid, polyphenol and tannin content), a full factorial design (3^2) was applied with five replicates in the center point to fit the surface plot for the responses and to estimate the pure error of the multiple regression models (Myers *et al*., 2009), totaling 13 sample preparations. The inlet hot air temperature and feed flow speed of spray drying process were kept in 180°C and 18 rpm, respectively.

The experimental design and statistical analysis were performed using Statgraphics plus 15.0 for Windows. A quadratic equation (second degree polynomial equation) (Equation 1) was used to fit the results:

$$Y = \beta_o + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{\substack{i=1 \\ i \langle j}}^{k-1} \sum_{j=2}^{k} \beta_{ij} X_i X_j \qquad \text{(Eq. 1)}$$

Where Y is the predicted response parameter, β_o is a constant, β_i, β_{ii} and β_{ij} are the regression coefficients and X_i and X_j are the levels of the independent variables (maltodextrin and arabic gum percent). Experimental data were then fitted to the selected regression model to achieve a proper understanding of the correlation between each factor and different responses. This correlation was obtained by estimating the numerical values of the model term (regression coefficients), whose significance was statistically judged in accordance with t-statistic at confidence interval of 95%. Non-significant (P-value > 0.05) term were deleted from the initial equation and data were refitted to the selected model. The quality of the mathematical models was fitted by RSM and evaluated by ANOVA, based on the F-test, the probability value (P-value) of lack-of-fit and on the percentage of total explained variance (R^2) and also on the adjusted determination coefficient (R^2_{adj}). These variance provide a measurement of the variability in the observed response values that could be explained by the experimental factors and their linear and quadratic interactions. A simultaneous optimization of the desirability function was performed in order to maximize the anthocyanin, flavonoid, polyphenol, tannin content and to minimize moisture content and particle size distribution.

3 RESULTS AND DISCUSSION

The use of maltodextrin and arabic gum was one of the main factors of the spray drying process. It was not only effective to moisture content, particle size distribution but also the maintenance of bioactive compounds content in spray-dried powder product.

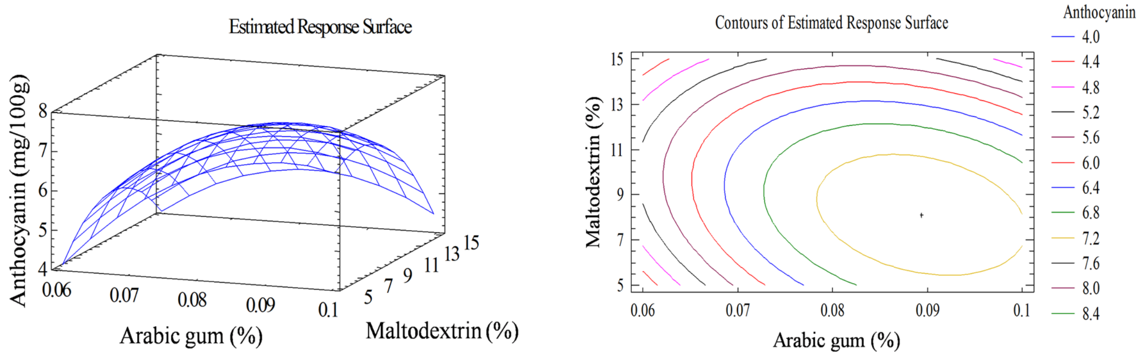
Maintaining the highest level of bioactive compounds in the product is of primary interest in the spray drying process. Besides, moisture content and particle size distribution of the products might also be interested. The results in Table 1 indicated that maltodextrin and arabic gum had effects on bioactive compounds content, moisture content and particle size. The anthocyanin content varied from 4.12 to 7.65 mg CE/100g, flavonoid content ranged from 23.61 to 30.49 mg QE/g, polyphenol content altered from 25.25 to 27.38 and tannin content changed from 22.06 to 24.65 mg TAE/g. Moisture content and particle size of product were 6.45 to 7.69%, 6.01 to 6.55 µm, respectively.

Table 1: Coded and real values of maltodextrin and arabic gum concentration in spray drying process and results from the chemical and product characteristic assays

Number Run	Factors		Responses variables					
	Arabic gum (%)	Maltodextrin (%)	Anthocyanin (mg/100g)	Flavonoid (mg/g)	Polyphenol (mg/g)	Tannin (mg/g)	Moisture (%)	Particle size (µm)
1	0.08 (0)	10 (0)	7.65	30.49	27.24	24.43	6.45	6.07
2	0.08 (0)	5 (-1)	6.55	30.11	26.67	24.50	7.09	6.01
3	0.1 (+1)	10 (0)	6.85	29.93	26.97	24.39	6.95	6.16
4	0.08 (0)	15 (+1)	5.35	23.72	25.96	23.74	6.89	6.41
5	0.08 (0)	10 (0)	7.20	30.38	27.27	24.59	6.49	6.09
6	0.1 (+1)	15 (+1)	4.55	23.61	25.25	24.41	7.29	6.49
7	0.06 (-1)	15 (+1)	4.12	23.65	26.36	22.06	7.68	6.55
8	0.08 (0)	10 (0)	7.19	30.45	27.19	24.62	6.52	6.08
9	0.1 (+1)	5 (-1)	6.98	29.90	26.82	24.18	7.55	6.11
10	0.06 (-1)	5 (-1)	4.18	30.35	26.38	23.40	7.69	6.03
11	0.08 (0)	10 (0)	7.10	30.40	27.38	24.42	6.48	6.05
12	0.08 (0)	10 (0)	7.05	30.30	27.31	24.65	6.51	6.06
13	0.06 (-1)	10 (0)	5.20	30.08	27.29	23.03	7.19	6.16

3.1 Effect of maltodextrin and arabic gum on bioactive compounds

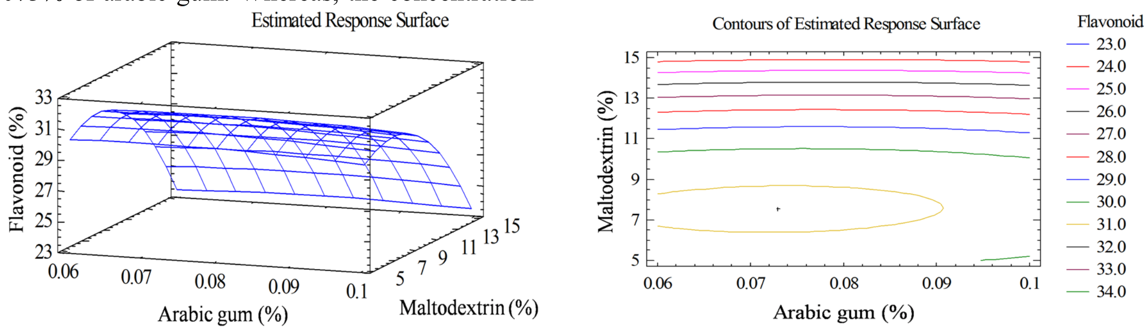
Figure 1a and 1b showed that the concentration of arabic gum and maltodextrin had a positive quadratic effect (P-value < 0.01) on anthocyanin content in obtained spray dried powder. Anthocyanin contents increased with the increases arabic gum concentration between 0.08 to 0.10% and achieved optimal values at 0.089%. In addition, anthocyanin was also achieved high values in maltodextrin concentration of approximately 5.0 to 9.0% and reached an optimum of 8.112%.



(a) (b)

Fig. 1: Response surface (a) and contour (b) plots for anthocyanin content in different maltodextrin and arabic gum concentrations

It could be noticed from Figure 2a and 2b that the concentration of arabic gum had negative quadratic influence (P-value < 0.05) to flavonoid content in products. The flavonoid content achieved high values with arabic gum concentration of range from 0.06 to 0.09% and reached optimum values at 0.073% of arabic gum. Whereas, the concentration of maltodextrin had a clear quadratic impact (P-value < 0.01) on flavonoid content in products. The flavonoid content achieved high values at maltodextrin concentration from 6 to 9% and the optimum values searched at supplemental maltodextrin concentration of 7.538%.

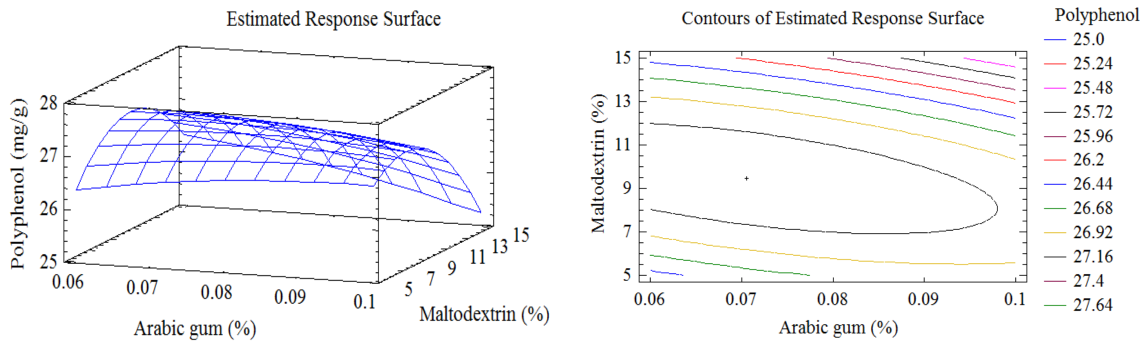


(a) (b)

Fig. 2: Response surface (a) and contour (b) plots for flavonoid content in different maltodextrin and arabic gum concentrations

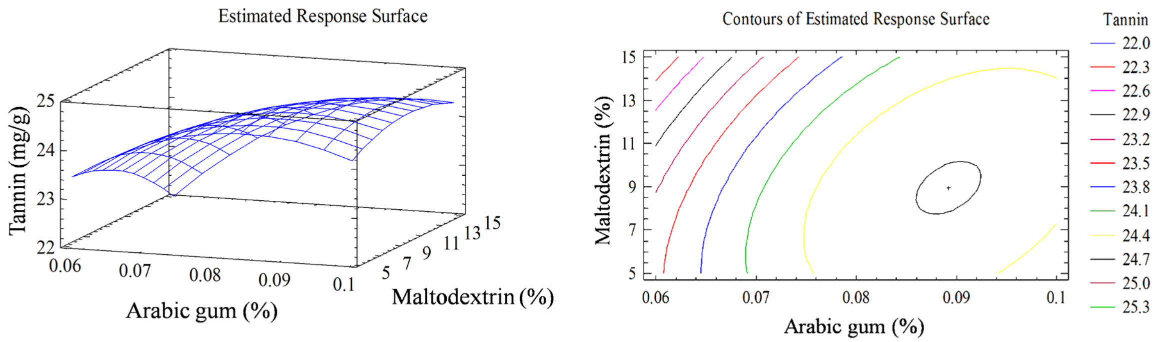
The response surface and contour plots in Figure 3a and 3b showed that the arabic gum concentration had less obvious quadratic impact (P-value < 0.01) than the polyphenol content in the product. The high polyphenol content obtained in using the arabic gum range from 0.06 to 0.095% and achieved optimal values at 0.071% concentration. However, the concentration of maltodextrin had a positive quadratic influence (P-value < 0.01) to the polyphenol content in the product. The polyphenol content increased with maltodextrin concentration increases in the range from 7.0 to 12.0% and the optimum value found in additional maltodextrin is 9.469%.

The graph of response surface and contour in Figure 4a and 4b showed that arabic gum and maltodextrin concentration had obvious quadratic influence (P-value < 0.01) on tannin content in the product. The high tannin content obtained in arabic gum about 0.085 to 0.095% and achieved the highest value at 0.089% of arabic gum. Additionally, the highest tannin content was obtained in the maltodextrin percent ranging from 8.0 to 10.0% and optimum value was found in supplemental maltodextrin was 8.96%.



(a) (b)

Fig. 3: Response surface (a) and contour (b) plots for polyphenol content in different maltodextrin and arabic gum percent



(a) (b)

Fig. 4: Response surface (a) and contour (b) plots for tannin content in different maltodextrin and arabic gum percent

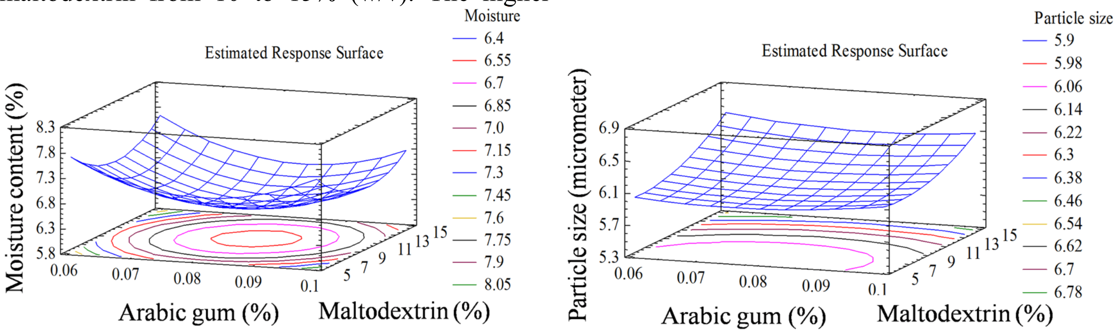
The bioactive compounds (anthocyanin, flavonoid, polyphenol and tannin) presented in final products which depended on the supplemental carrier percent of maltodextrin and arabic gum. The bioactive compounds content increased with the additional maltodextrin concentration increases from 5 to 9%. But, it decreased with the additional maltodextrin concentration increases from 9.0 to 15%. Moreover, study results showed that the maltodextrin levels were more obvious quadratic impact on bioactive compounds in the product than the arabic gum levels. The most of bioactive compounds achieved the highest values when the concentration of maltodextrin and arabic gum were added to the extract in range from 7.5 to 9.0% and from 0.073 to 0.089%, respectively. Bhusari and Kumar (2014) also showed the polyphenol content increased in accordance with increase of the concentration of carrier agent addition. Masniza *et al.* (2013) reported that the best quality of Garcinia powder with additional maltodextrin concentration was 5%. The beetroot-orange juice powder also obtained with the best functional properties and the conservation of betalain was high in the addition of 5% of maltodextrin (Ochoa-Martinez *et al.,* 2015). The best quality of Ber powder was obtained with encapsulating material, 8% maltodextrin (Singh *et al.,* 2014). Whereas, the use of a 10:1 maltodextrin/pectin weight ratio (11% w/v) led to encapsulate 3% w/v polyphenol-rich extract forming stable powder made up of well-formed and micronized particles suitable for storage and handling (Sansone *et al*., 2011). The pink guava powder produced with 15% was found to be more convenient than others where low moisture content indicates more stability with the highest bulk density (Shishir *et al.,* 2015). In order to obtain a pequi pulp powder with high nutritional quality (vitamin C, carotenoid) was the additional carrier rated 18% of maltodextrin (Santana *et al*., 2016).

3.2 Effect of maltodextrin and arabic gum on physical characteristics of powder product

The moisture content has an inflence on the keeping quality of the powder (Goula *et al*., 2004). Figure 5a and 5b showed that the additional carrier concentration also had positive quadratic impact on the moisture content and particle size distribution of spray dried powder product (P-value < 0.01). The moisture content was decreased with increasing maltodextrin and arabic gum concentration. The study result was also similar to the results reported of Fernandes *et al.* (2012), Wang and Zhou (2013), Sabhadinde (2014), Sarabandi *et al.* (2014). The low moisture content of product obtained the maltodextrin and arabic gum levels in range of 9 to 12% and 0.075 to 0.09% respectively. And the lowest moisture content achieved at maltodextrin of 10.434% and arabic gum of 0.082% (Figure 5a).

The levels of maltodextrin used for development of the *Pouzolzia zeylanica* powder varied between 5 to 15% (w/v) which were less than 10 to 30% that were used by Abadio *et al.* (2004), Tonon *et al.* (2008) and Kha *et al.* (2010). Moisture content of sample decreased as maltodextrin concentration increased from 5 to 9%. Abadio *et al.* (2004) also found a decrease in moisture content in final pineapple juice powder with the increase in the level of maltodextrin from 10 to 15% (w/v). The higher concentration of maltodextrin used could be increased the level of feed solids and reduced the level of total moisture for evaporation (Grabowski *et al.*, 2006; Kha *et al.*, 2010).

Whereas, arabic gum concentration had effects on particle size with P-value > 0.05. The mean particle size had increased with increasing additional maltodextrin concentration (Figure 5b). The result from Sharifi *et al.* (2015) reported that as level of maltodextrin increased from 7.5 to 15% SEM micrographs of the powder indicated the increasing trend in particle size as result of increase of concentration of maltodextrin as drying aid. However, Fermandes *et al.* (2012) reported that no correlation was found between particle size distribution and different carbohydrate proportions.



(a) (b)

Fig. 5: Response surface and contour plots for moisture content (a) and particle size (b) in different maltodextrin and arabic gum concentrations

A statistical analysis was performed on the experimental results to obtain the regression models. ANOVA was used to evaluate the significance of each variable on the received model. The quadratic model for all responses of coded factors were shown in Table 2.

Table 2: Mathematical equations that describe the response variables (anthocyanin, flavonoid, polyphenol, tannin, moisture content and particle size) in response to maltodextrin and arabic gum concentrations

Response variables	Regression Equation	R^2	R^2 (adjusted for d.f.)	P-value (lack-of-fit)
Anthocyanin (mg CE/100g)	$Y = -21.987 + 542.537X_1 + 1.296X_2 - 2766.380X_1^2 - 5.925X_1X_2 - 0.047X_2^2$ (Eq. 2)	0.986	0.977	0.889
Flavonoid (mg QE/g)	$Y = 21.211 + 74.209X_1 + 1.924X_2 - 561.207X_1^2 + 1.025X_1X_2 - 0.133X_2^2$ (Eq. 3)	0.998	0.997	0.056
Polyphenol (mg GAE/g)	$Y = 19.723 + 82.845X_1 + 0.990X_2 - 327.155X_1^2 – 3.875X_1X_2 – 0.038X_2^2$ (Eq. 4)	0.995	0.992	0.881
Tannin (mg TAE/g)	$Y = 12.449 + 287.615X_1 – 0.125X_2 – 1809.05X_1^2 + 3.925X_1X_2 – 0.013X_2^2$ (Eq. 5)	0.984	0.973	0.292
Moisture content (%)	$Y = 17.773 – 228.787X_1 – 0.359X_2 +1428.88X_1^2 – 0.625X_1X_2 + 0.02X_2^2$ (Eq. 6)	0.998	0.996	0.549
Particle size (µm)	$Y = 7.296 – 31.368X_1 – 0.039X_2 + 218.996X_1^2 – 0.35X_1X_2 + 0.006X_2^2$ (Eq. 7)	0.995	0.992	0.422

Note: X_1 = Arabic gum concentration (%, w/v); X_2 = Maltodextrin concentration (%, w/v)

The goodness-of-fit of the regression model showed that the experiment and predict data were fitted and the coefficient of determination $R^2 > 0.8$ (Guan and Yao, 2008). In addition, the probability value of lack-of-fit was non-significant P-value > 0.05 (Zabeti *et al.*, 2009).

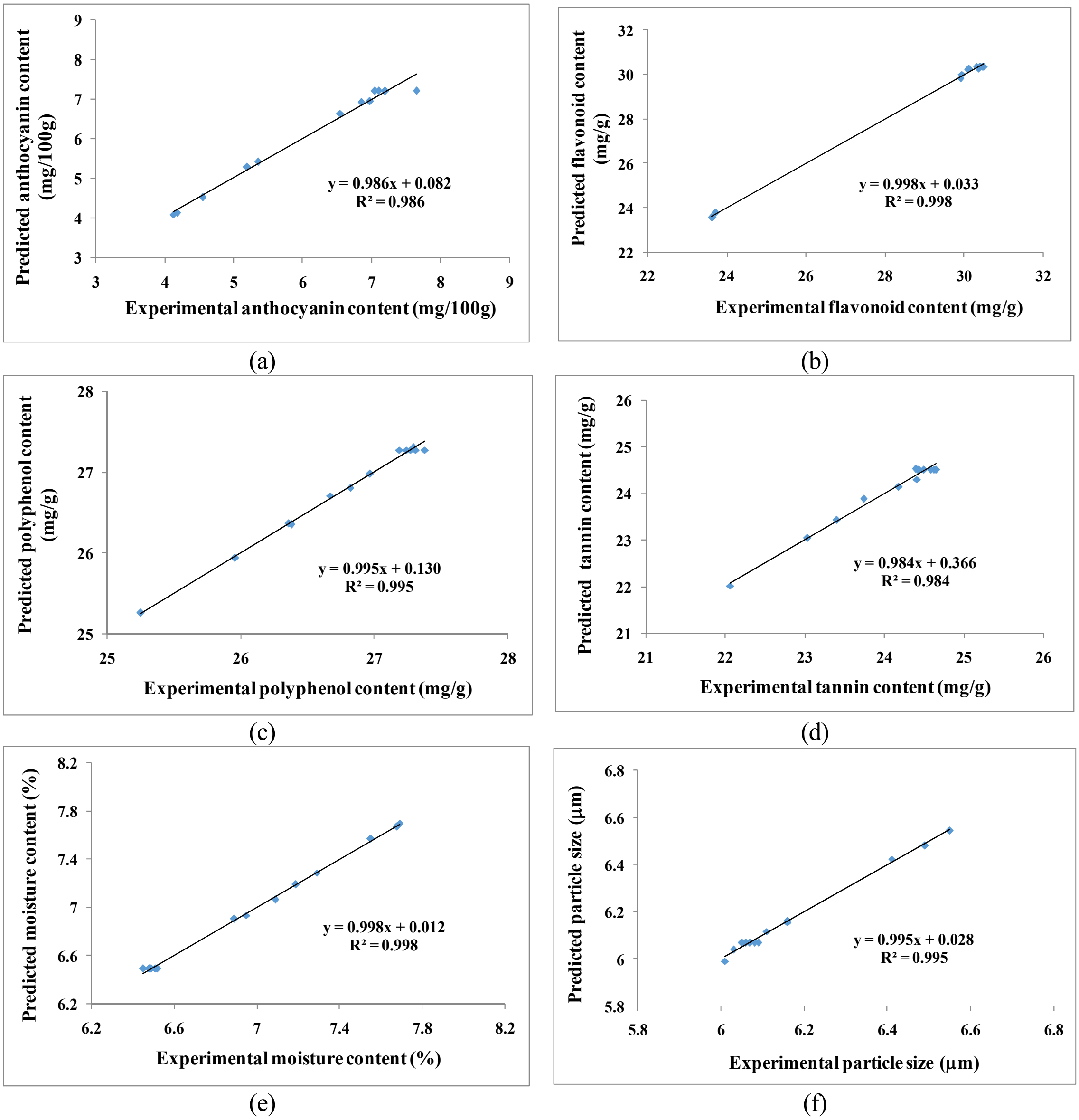


Fig. 6: Correlation between the experimentally and the estimated values for anthocyanin (a), flavonoid (b), polyphenol (c), tannin (d), moisture content (e) and particle size (f) using the models described in equation 2, 3, 4, 5, 6, 7, respectively as shown in Table 2

The results of ANOVA analysis showed that the linear, quadratic and interaction factors of maltodextrin and arabic gum concentration had effect on anthocyanin, flavonoid, polyphenol, tannin and moisture content; and particle size distribution of obtained powder product reached the reliability of 95%. The coefficient of determination of the predicted models in the response was $R^2 > 0.98$, $R^2_{adj} > 0.97$ and Lack of fit had P-value > 0.05. These values would give a relatively good fit to the mathematic model. Moreover, the correlation between experimental and predictable data of goal functions such as anthocyanin, flavonoid, polyphenol, tannin, moisture content and particle size also showed in Figure 6.

3.3 Multiple response optimization

The simultaneous optimization of multiple responses might be the main concern for industrial applications (Tsai *et al.,* 2010), especially the energy cost of the process significantly diminished when extraction parameters are optimized (Spigno *et al.*, 2007). The response variables including anthocyanin, flavonoid, polyphenol, tannin, moisture content and particle size were optimized separately with different maltodextrin and arabic gum concentrations. Therefore, in order to find out generally optimal parameter for maltodextrin and arabic gum concentration to blend which extract undergoing spray drying process, it is necessary that simultaneous optimization of multiple response variables mainly aim at basing on bioactive compounds of product. Yet, the desirability function in the RSM was utilized to reveal the combination of the parameters (maltodextrin and arabic gum concentration) which are capable of simultaneously maximizing or minimizing the responses. The overplay plot shows the outlines superposition of all the studied responses and the simultaneous optimum for all responses is showed by the black spot (Figure 7).

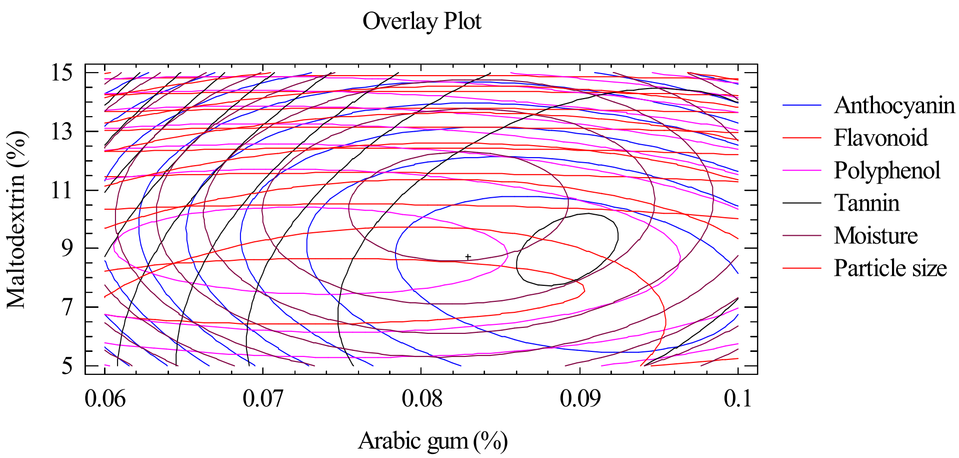


Fig. 7: Superposition contour plots, showing the best experimental parameters that maximize bioactive compounds content and minimize powder product characteristics (the black spot shows the optimum for all the responses)

4 CONCLUSIONS

The effects of the carrier (maltodextrin and arabic gum) on the powder quality of the spray dried *Pouzolzia zeylanica* extract were investigated successfully by factorial experimental design. The result of simultaneous optimum for all responses showed that the optimum supplemental carrier concentration to produce spray dried powder with the highest content of bioactive compounds, the lowest moisture content and the smallest particle size were obtained at the blending of maltodextrin and arabic gum concentration was 8.743% and 0.083%, respectively.

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