PAPER

PRODUCTION OF INSTANT *NIGELLA SATIVA L.* BEVERAGE POWDER BY DRUM DRYING USING ARABIC GUM AS ADJUNCT

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ABSTRACT

This study determined the feasibility of drum drying process in producing drum dried *Nigella sativa L.* beverage powder using the response surface methodology to determine the effect and relationship between percentage of Arabic gum and drying temperature on the quality of the *Nigella sativa* powder produced. About 32.7 % of Arabic gum and 119.1°C of drying temperature were the optimum parameters for the production of *Nigella sativa* powder. Antioxidants and calcium content of *Nigella sativa* powder decreased significantly, while the iron, manganese, and sodium contents increased significantly after the drum drying process.

Keywords: Arabic gum, drum drying, *Nigella sativa* powder

1. INTRODUCTION

Nigella sativa L. (*N. sativa*), popularly known as black cumin or black seed, is a member of *Ranunculaceae* family. It is native to Southern Europe, North Africa, and Southwest Asia, but also widely cultivated in other countries such as Middle Eastern Mediterranean region, South Europe, India, Pakistan, Syria, Turkey and Saudi Arabia (KHARE, 2004). *N. sativa* seeds are extensively used for traditional medicinal purposes in treating various diseases such as skin diseases, jaundice, gastrointestinal problems, hypertension, and diabetes (FOROUZANFAR *et al*., 2014). Black seeds have also been used for thousands of years for culinary purposes as a spice or food (TIRUPPUR *et al.*, 2010). Recently, *N. sativa* has gained the attention of researchers due to its health benefits, attributed to the presence of bioactive components. High phytochemical content in black seed makes it a healthy food product with nutraceutical and functional benefits for human consumption. Various studies have shown that black cumin seed provides anticancer, antidiabetic, antimicrobial, antiradical, anti-inflammatory, antihypertensive, hepatoprotective and renal protective properties (RAMADAN, 2007). Normally, *Nigella sativa* seeds are roasted to give a flavour similar to that of coffee. The roasting also contributes to a high percentage of water soluble extracts, making it a potential substitute to the coffee (KOMES *et al*., 2015). The significant health benefits of black seed and its unique flavour after roasting which is similar to coffee flavour, have increased the interest in developing it into a convenient substitute for coffee brew in the market. With the current consumer shift towards convenient healthy food products, such as easily rehydratable beverage powders, black seeds powder has the potential to open up a new possibility for the usage of black seeds as a healthy beverage in addition to the traditional medicinal and culinary uses. Drum drying method is widely applied in the food industry to convert liquid or paste from heat sensitive food products into a quickly rehydratable flake or powder form product. It is an inexpensive industrial practice for the production of foodstuffs including milk, coffee, fruit purees, baby foods, mashed potatoes, dry soup mixes, etc. The benefits of drum drying include; the ability to handle a wide range of products, economical operation, less labour intensive and high production yields (MUJUMDAR, 1995). However, drying can lead to a significant decrease in the phytochemical contents, thereby, reducing the phenolic contents, antioxidant activity, and flavonoids (RABABAH *et al*., 2015). To the best of our knowledge, a few research has been performed on drum drying *Nigella Sativa* seeds to produce instant beverage powder. The aim of this study was to determine the feasibility of drum drying process in producing *Nigella sativa L.* beverage powder, while using arabica gum as an adjunct. Arabic gum is popularly used as an encapsulating material by entrapping the core compound, helping to retain the volatile substances and provide protection from oxidation (RIGHETTO and NETTO, 2005). Encapsulation is often used in the food industry to isolate the active molecules such as aroma, vitamins, and antioxidants from the environment (TURCHIULI *et al*., 2014). Response surface methodology (RSM) was used to study the effect and relationship between percentage of Arabic gum and drying temperature on the quality of the black seed powder produced

2. MATERIALS AND METHODS

2.1. Materials

Roasted black seeds (*N. sativa*) were obtained from a local manufacturing industry in Penang, Malaysia. The seeds were packed in an airtight container. Arabic gum was supplied by Liangtraco & Sons Sdn. Bhd., Penang, Malaysia. The proximate composition of *Nigella sativa* seeds and drum dried powder was analysed using standard methods prescribed by the Association of Analytical Chemists methods (AOAC, 2000); fat (AOAC method 960.39), moisture (AOAC method 950.46), ash (AOAC method 923.03), crude protein (AOAC method 960.52) and crude fiber (AOAC method 962.09).

2.2. Preparation of feed materials for drying

The roasted black seeds were mixed with distilled water at a ratio of 1:2 and blended using a high-speed blender for 1 min. The blended black seeds solution was then filtered with muslin cloth to remove insoluble particles. The feed material was prepared by mixing the specific amount of arabic gum into the filtered black seeds solution using a mixer (Pensonic Group, Malaysia). Arabic gum was added according to the experimental design in Table 1.

2.3. Experimental design

Response surface methodology (RSM) with statistical commercial package, Design Expert version 6.0 (Statease Inc., Minneapolis, USA) was used in this experiment to study the effects of percentage of arabic gum and temperature of the drum on physical properties (that is, yield, water activity, moisture content, colour, and solubility) of the powder. Face-centred central composite design with two factors and three levels were used as experimental design. The three coded levels used in this experiment were –1, 0 and +1, which corresponded to the low level, mid-level and high level of each independent variable, respectively. The independent variables and representative coded and uncoded levels are given in Table 1.

Table 1. Independent variables and their coded levels in the central composite design.

		Coded values		
Independent Variables	**Symbol**	**–1**	**0**	**+1**
Concentration of Arabic gum (%)	X_1	15	25	35
Temperature (^{o}C)	X	90	120	150

2.4. Drying Operation

The feed materials prepared were dried using a double drum dryer (GF Dryer and Flaker, Model 215, Mathis Machine Corporation, USA) with drum rotation speed of 1 rpm and a 3 mm gap between drums. Drum temperatures were set at three levels: 90, 120, and 150°C. Dried flakes were collected, weighed and stored in a sealed aluminium layered plastic bag. The dried flakes were then ground into powder using a blender for further analysis.

2.5 Analysis of Physical Properties

2.5.1 Yield

The yield was calculated based on dry matter. Total weight of solid content used for the preparation of feed material and the weight of the powder obtained after drum drying were recorded. Total weight of solids content was referred to as the summation weight of roasted black seed and arabic gum. Product yield was calculated using Equation 1.

$$\text{Yield (\%)} = \frac{\text{Total weight of resulting powder}}{\text{Total weight of solids content}} \text{ x } 100 \qquad \text{Eq. 1}$$

2.5.2 Water activity

The water activity of the samples was measured directly by a water activity instrument (AquaLab, Model Series 3TE, Decagon Devices Inc., USA). Calibration of the instrument was done prior to measurement.

2.5.3 Moisture content

The moisture content of the samples was determined using oven method (AOAC, 1984). One to two grams of powder was weighed intoa moisture dish and dried in an oven (Memmert UM600, Schwabach, Germany) at 105°C for 24 h. The weight loss after drying in the oven was used to calculate the moisture content of the powder. The moisture content was expressed as the percentage of the original sample (Equation 2).

$$\text{Moisture content (\%)} = \frac{\text{Total weight loss}}{\text{Total weight of sample}} \text{ x } 100 \qquad \text{Eq. 2}$$

2.5.4 Colour measurement

The colour of the samples was measured using a spectrophotometer (Minolta Model Series CM-3500d, Minolta Corporation, Japan). Prior to the measurements, the spectrophotometer was calibrated using zero calibration box (CM-A100) and white calibration plate (CM-A120) provided by the manufacturer. The colour was expressed in terms of L* (lightness), a* (redness) and b* (yellowness) in CIE system.

2.5.5 Solubility

The solubility was determined using the method by CANO-CHAUCA *et al.*, (2005) with slight modifications, where 1 g of powder was mixed with 100 mL of distilled water and stirred for 10 min. Then, the solution was transferred to a centrifuge tube and centrifuged at 3,500 rpm for 10 min (Centrifuge Model 4000, Kubota Corporation, Japan). An aliquot of 15 mL of the supernatant was then transferred to pre-weighed crucibles and dried overnight in an oven (UM600, Memmert Corporation, Schwabach, Germany) at 105°C. The solubility was calculated by the weight difference after drying and expressed as a percentage of the original weight (Equation 3).

$$\text{Inhibition (\%)} = \frac{\text{Absblank weight loss}}{\text{Total weight of supernatant}} \text{ x } 100 \qquad \text{Eq. 3}$$

2.6. Analysis of antioxidant properties

2.6.1 Preparation of Sample Extract

A sample of 0.5 g was extracted with 20 mL of distilled water. Extraction was carried out at 80°C in a water bath (SW-23, Julabo, Seelbach, Germany) and shaken at 200 rpm for 120 min. After the extraction was complete, the suspension was centrifuged at 3500 rpm for 15 min by a centrifuge (Centrifuge Model 4000, Kubota Corporation, Tokyo, Japan). The supernatant was collected. The extract was used for the determination of radical scavenging activity, total phenolic contents, and total flavonoid content.

2.6.2 DPPH free radical scavenging activity

Determination of DPPH free radical scavenging activity was based on the method described by VARASTEGANI *et al.,* 2015). An aliquot of 10 μL of sample extract was mixed with 1090 μL of distilled water. Then, 3.9 mL of 25 mM DPPH methanolic solution was added. The mixture was thoroughly done using vortex and stored under dark condition for 30 mins. Absorbance was measured at wavelength of 515.0 nm using a UV-V spectrophotometer (UVmini-1240, Shimadzu Corporation, Kyoto Japan) against the blank. The blank was prepared by replacing the sample extract with distilled water. The free radical scavenging activity was expressed as a percentage of inhibition and was calculated from Equation 4.

$$\text{Inhibition (\%)} = \frac{Abs_{blank} - Abs_{sample}}{Abs_{blank}} \text{ x } 100 \qquad \text{Eq. 4}$$

where,
Abs_{blank} = Absorbance of the blank
Abs_{sample} = Absorbance of the sample

2.6.3 Total Phenolic Content (TPC)

Determination of total phenolic content was based on Folin-Ciocalteu (FC) method as described by SINGLETON and ROSSI, (1965) but with slight modification. An aliquot of 40 μL of sample extract was added with 3.12 mL of distilled water, followed by 0.2 mL of FC reagent. Then, 0.6 mL of 20% (w/v) sodium carbonate solution was added. The solution was mixed well using vortex, and incubated in 40°C water bath (SW-23, Julabo, Seelbach, Germany) for 30 mins. Finally, UV-Vspectrophotometer (UVmini-1240, Shimadzu Corporation, Kyoto Japan) was used to measure the absorbance of the samples at 765 nm. Standard solution of gallic acid with different concentrations was used to prepare a standard curve for TPC determination. TPC was calculated from the prepared gallic acid standard curve. The results were expressed as gallic acid equivalent (mg GAE/g).

2.6.4 Ferric Reducing Antioxidant Power (FRAP)

FRAP was assessed using a modified method proposed by BENZIE and STRAIN, (1996). This method is based on the reduction of Fe^{3+} ferric-TPTZ (tripyridyltrianzin) to a blue colour solution (Fe^{2+} ferric-TPTZ). FRAP reagent was prepared in the ratio of 10:1:1 by mixing 300 mM acetate buffer (pH 3.6), 20 mM ferric (III) chloride hexantydrate (FeCl3·6H2O) and 2,4,6-tris (1-pyridyl)- 5-triazine (TPTZ) solution in 40 mM HCl. About 3.8 mL of the prepared FRAP reagent at 37°C was mixed with 200 μL of extract and vortexed. This mixture was then incubated for 30 min at 37°C in the dark, after which the absorbance was read at 593 nm (UVmini-1240, Shimadzu Corp, Japan) against a blank. A standard curve was made using the aqueous solution of ferrous sulphate heptahydrate (FeSO4·7H2O) solution (200-800 μmol). The FRAP value was recorded in terms of ferrous equivalent (μmol/mg of sample).

2.6.5 Total Flavonoid Content (TFC)

Total flavonoid content was determined using a slightly modified colorimetric assay proposed by RAMAMOORTHY and BONO, (2007). A properly diluted 1.5 Ml extract was mixed with 1.5 mL of 2% aluminium chloride ($AlCl_2$) in methanol. After 10 min incubation at 37°C, absorbance of the mixture was read at 415 nm (UVmini-1240, Shimadzu Corp, Japan) against the blank. The blank was prepared by replacing the extract with distilled water in the mixture. A standard curve was prepared using (-)-Epicatechin (10–90 mg/L) to calculate the TFC. TFC was expressed on weight basis as mg (-)-Epicatechin equivalent (ECQ/g of sample).

2.7. Mineral content

Mineral content was determined based on the method described by SPEIGHT, (2015) although, with slight modification. Mineral contents (calcium, iron, manganese, and sodium) were analysed using atomic absorption spectroscopy (AAS). Atomic absorption spectrophotometer (AA-700, Shimadzu Corporation, Singapore) was used to analyse all the samples. Samples were digested for 1 h in microwave digester (MARS 6 240/50, CEM Corporation, USA) for extraction prior to mineral content determination. An aliquot of 10 mL of HNO_3 and 1 mL of 30% (v/v) H_2O_2 were added into 1 g of sample for digestion.

2.7.1 Calcium

A series of calcium standard solutions with concentrations of 0, 1, 2, 3, and 4 ppm was prepared with various amount of stock solution, 20 mL of lanthanum oxide (La_2O_3) and diluted up to 100 mL with deionised water. Sample was prepared by adding 20 mL of La_2O_3 into the digested sample and made up to 100 mL with deionised water. La_2O_3 solution was prepared by first adding 50 mL of deionised water into 58.64 g of La_2O_3 powder. Then, 250 mL was added into the mixture and the solution was made up to 1 L with deionised water in a volumetric flask.

2.7.2 Iron

A series of iron standard solutions with concentrations of 0, 1, 2, 3, and 4 ppm were prepared with respective amount of stock solution and diluted up to 100 mL with deionised water. The digested sample was diluted up to 100 mL with deionised water to obtain an analytical sample.

2.7.3 Manganese

A series of manganese standard solutions with concentrations of 0, 0.5, 1.0, 1.5, and 2.0 ppm were prepared from respective amount of stock solution and diluted up to 100 mL with deionised water. Analytical sample was prepared by diluting the digested sample up to 100 mL with deionised water.

2.7.4 Sodium

A series of sodium standard solutions with concentrations of 0, 0.5, 1.0, 1.5, and 2.0 ppm were prepared from respective amount of stock solution, 5 mL of 10% (w/v) potassium chloride and diluted up to 100 mL with deionised water. Analytical sample was prepared by adding 5 mL of potassium chloride into the digested sample and made up to 100 mL with deionised water.

2.8. Sensory evaluation

Sensory evaluation of the beverage derived from the drum dried instant *Nigella sativa* powder was carried out with thirty panellists comprising students and staff at the School of Industrial Technology, University Sains Malaysia. Testing was conducted in the sensory laboratory. Panellists were required to evaluate the sensory attributes such as appearance (color), aroma, taste, texture (mouth feel) and overall acceptability of the reconstituted instant beverage derived from the spray dried product, using a 9-point hedonic scale with 1= dislike extremely, 2= dislike very much, 3= dislike moderately, 4= dislike slightly, 5= neither like nor dislike, 6= like slightly, 7= like moderately, 8= like very much, and 9=like extremely. The reconstituted instant black seed drink was served warm with the brewed roasted black seed drink as control. Every sample was uniquely coded, which made the evaluation single blinded.

2.9. Statistical analysis

The experimental data were reported as mean±standard deviation of triplicate measurements. Experimental data were analysed using IBM SPSS Statistics version 20 software (IBM Corporation, New York, USA). Statistical analysis was performed using Duncan's paired samples at the significance level of $\alpha = 0.05$.

3. RESULTS AND DISCUSSION

3.1. Proximate composition

The proximate compositions of the raw and roasted *Nigella sativa* seeds and drum dried powder are presented in Table 2. The moisture content was significantly lower in roasted black seed (1.31%) compared with other samples. The moisture content of spray-dried powder was lower than 5%, which is ideal for product shelf life. Protein content in raw black seed was significantly higher than in roasted and drum drying powder due to heat treatment in those samples.

Table 2. Proximate composition of *Nigella sativa* seeds and drum dried *Nigella sativa* powder.

	Ash (%)	Moisture %)	Fat (%)	Protein (%)	Fibre (%)	Carbohydrate (%)
Raw black seeds	2.86±0.07^{a}	1.80±0.03^{b}	38.74±0.32^{a}	2.97±0.002^{c}	9.64±0.18^{a}	43.99±0.56^{c}
Roasted black seed	2.01a±0.08^{b}	1.31±0.09^{c}	21.5±0.15^{b}	1.56±0.003^{a}	8.17±0.12^{b}	65.45±015^{b}
Drum Dried Powder	1.18± 0.05^{c}	2.89±0.18^{a}	2.58±0.02^{c}	0.61±0.005^{b}	2.12±0.04^{c}	90.53±0.18^{a}

Values are means of three replications±standard deviation, dry matter basis. Means in row not sharing the same letter are significantly different ($P < 0.05$).

Drum drying resulted in a significant decrease in fat content compared with the raw samples. This decrease in fat content could be advantageous to both the drying process and also the output powder in terms of extending shelf life (COSTA *et al.*, 2015). Drum drying with Arabic gum as adjunct increased the amount of non-NS solids in the powder samples, which significantly lowered the fibre content of the resulting dried powder. The significantly elevated carbohydrate percentages found can be easily explained due to the addition of carrier agents, such as polysaccharides (Arabic gum). The resulting powder was also higher in carbohydrate percentage (COSTA*et al*., 2015).

3.2. Response surface analysis

The effect of concentration of arabic gum and drum temperature on the yield, water activity, moisture content, colour values, and solubility of the drum dried *N. sativa* beverage powder is shown in Table 3. The independent and dependent variables (responses) were fitted into respective model equation and goodness of fit was determined. Table 4 shows the significance of the independent variables on responses. The data indicated that percentage of arabic gum used and temperature were significant factors at 95% confidence level p- ≤ 0.05 for all the responses studied (that is, yield, water activity, moisture content, colour L, a*, and b* values, and solubility). There was no significant lack-of-fit ($p > 0.05$) in all dependent variables. This indicated that the independent and dependent variables are accurately fitted into the models. Coefficient of determination (R^2) determines the overall predictive capability of the model (BAŞ and BOYACI, 2007). All responses showed satisfactory level of R^2except colour values L and a*. This implied that the models used for colour values of L and a* did notproperly represent the model of the data. Therefore, they are used mainly for trend observation. The non-satisfactory level of R^2could be due to the large range of variations and noises.

Table 3. Central composite design and experimental results for the response variables.

Run	Independent variables		Response variables						
	X_1	X_2	Yield (%)	Water Activity, a_w	Moisture content (%)	Colour values			Solubility (%)
						L	*a**	*b**	
1	−1	−1	47.64	0.336	4.34	26.51	9.32	14.29	84.81
2	+1	+1	55.19	0.138	0.76	32.23	8.96	15.01	66.31
3	0	−1	56.25	0.326	4.10	27.56	8.68	14.80	80.82
4	0	0	49.89	0.179	1.26	28.01	8.8	14.15	77.78
5	−1	0	45.58	0.203	1.49	27.57	9.17	13.84	83.43
6	+1	0	55.49	0.178	1.14	30.08	8.79	14.40	73.60
7	0	+1	48.88	0.169	0.98	29.34	9.32	15.35	69.13
8	0	0	49.67	0.184	1.34	27.87	9.04	14.49	76.97
9	+1	−1	62.95	0.317	3.82	29.55	8.69	15.33	74.90
10	−1	+1	42.61	0.191	1.37	28.30	9.64	15.00	75.90
11	0	0	50.25	0.173	1.28	28.11	9.56	14.30	78.59
12	0	0	48.08	0.191	1.42	26.99	9.19	14.36	77.24
13	0	0	48.92	0.185	1.36	29.31	8.82	14.29	78.22

Table 4. Analysis of variance on the independent variables as model and interaction terms on the response variables.

Source	Response Variables						
	Yield (%)	Water Activity, a_w	Moisture Content (%)	Colour Values			Solubility (%)
				L	*a**	*b**	
Model (*p*-value)	< 0.0001	< 0.0001	< 0.0001	0.0005	0.0118	< 0.0001	< 0.0001
Lack-of-fit (*p*-value)	0.1850	0.5801	0.4718	0.6056	0.9578	0.5046	0.2925
R-squared (%)	96.21	99.38	99.80	77.79	58.85	96.09	98.14

3.3. Physical properties

3.3.1 Yield

As shown in Table 4, the percentage of arabic gum used and drum temperature were significant factors ($p \leq 0.05$) affecting the yield of *N. sativa* powder. The higher the percentage of arabic gum incorporated, the higher yield resulted (Fig. 1). Yield was calculated on dry matter basis. Higher percentage of arabic gum incorporated into the black seed had contributed to a higher total solid content. Thus, the percentage yield increased with an increase in the percentage of arabic gum. Meanwhile, the yield was lowered as the temperature increased (Fig. 1). Drum dryer is operated by heating the steam at the inner surface of the drum (TANG *et al*., 2003). Therefore, the steam pressure used correlates with the temperature. Previous research also reported that the percentage yield of drum dried pitaya peel decreased with an increase in steam pressure (CHIA and CHONG, 2015b), which implied that the yield increased with the temperature.

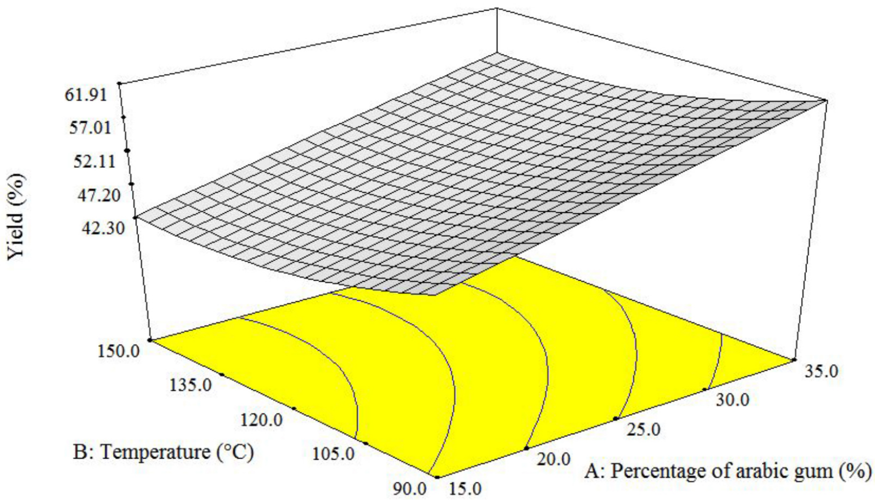

Figure 1. Response surface plot and contour plot for the interactive effects of percentage of arabic gum on the yield of drum dried N. sativa powder.

3.3.2 Water activity and moisture content

3.3.2 Water activity and moisture content

Water activity (aw) is defined as the ratio of vapour pressure of water in a food system to the vapour pressure of pure water at the same temperature (TROLLER, 1978). Water activity measures the availability of free water for any biochemical reactions. On the contrary, moisture content measures the water composition. The lower the water activity, the lesser free water is available for the biochemical reactions and hence, the longer the shelf life of black seed powder (QUE *et al.*, 2007). Figs. 2 and 3 demonstrated that water activity and moisture content decreased with the increase in drying temperature.

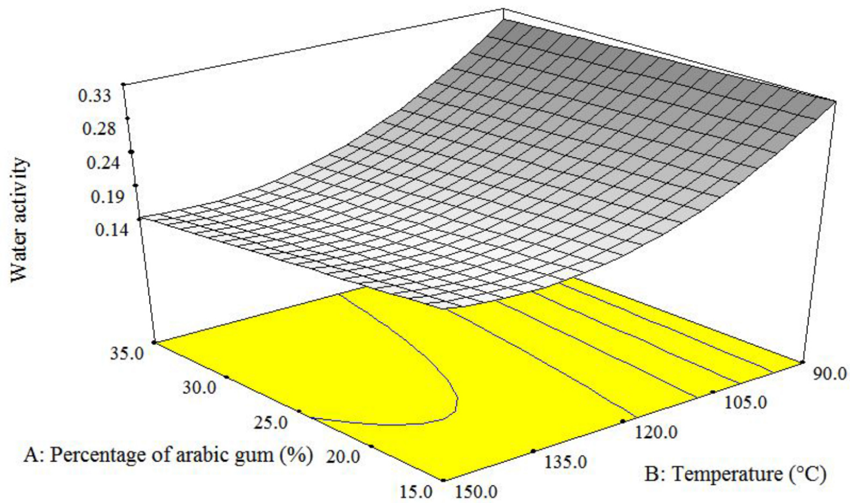

Figure 2. Response surface plot and contour plot for the interactive effects of percentage of arabic gum on the water activity of drum dried *N. sativa* powder.

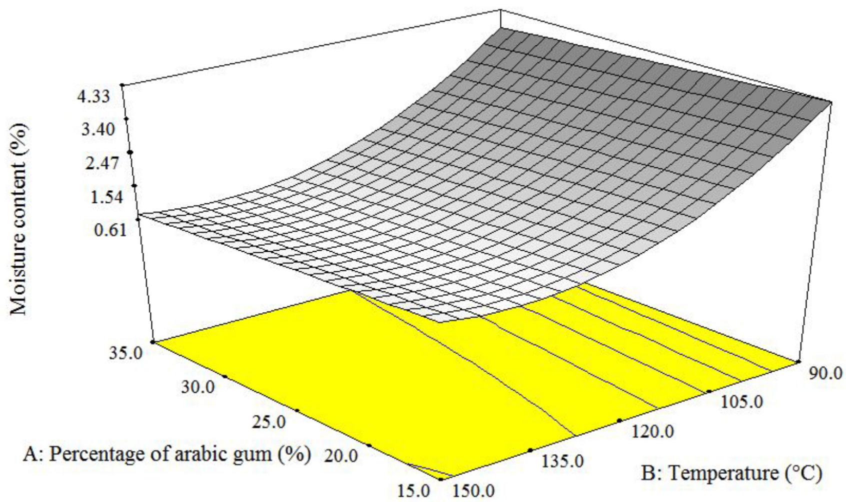

Figure 3. Response surface plot and contour plot for the interactive effects of concentration of arabic gum on the water activity of drum dried *N. sativa* powder.

The results were consistent with other findings in which the water activity and moisture content decreased with an increase in the temperature during the production of pregelatinized maize starch using the drum drying method (VALOUS *et al.*, 2002). The increase in the temperature increases the moisture transfer rate (JITTANIT *et al.*, 2011) and moisture removal rate (KAKADE *et al*., 2011), thus reducing the moisture content at a faster rate. Sorption isotherm explains the relationship between water activity and moisture content. High moisture content usually leads to high water activity, although the sorption isotherm is not in a linear regression (CHIA and CHONG, 2015a). Meanwhile, water activity decreased with the increase in the percentage of arabic gum. However, the decrease in the water activity was very small or negligible due to the effect of arabic gum percentage. These results were in agreement with the study by PUA *et al.* (2007), where they demonstrated that there was no significant contribution of percentage of arabic gum on the water activity of jackfruit powder.

3.3.3 Colour

In this study, results indicated that all the colour L, a*, and b* values were significantly affected by both variables at $p \leq 0.05$ (Table 3). These results were consistent with other research done. According to PUA *et al*., (2007), colour values of drum dried jackfruit powder were significantly affected by percentage of arabic gum used. The change in colour is probably due to colour nature of the arabic gum. Meanwhile, CAPARINO *et al*., (2012) reported that the change in colour of the drum dried mango flakes or powder was attributed to the high drying temperature. The change in the colour was due to Maillard reaction, which resulted in browning (CHIA and CHONG, 2015b). Colour L values indicate the darkness or whiteness while lower L value indicates darker black (TOPUZ *et al.*, 2009). The L value increased when the temperature increased. This indicated that the powder became browner or less black as the processing temperature increased. There is also a possibility that the Maillard reaction or the browning reaction incorporated brown colour in the drum dried product, leading to loss of the original dark black colour. Fig. 4 (a) and Fig. 4 (b) show that both the L and a* values increased with increase in drying temperatures.

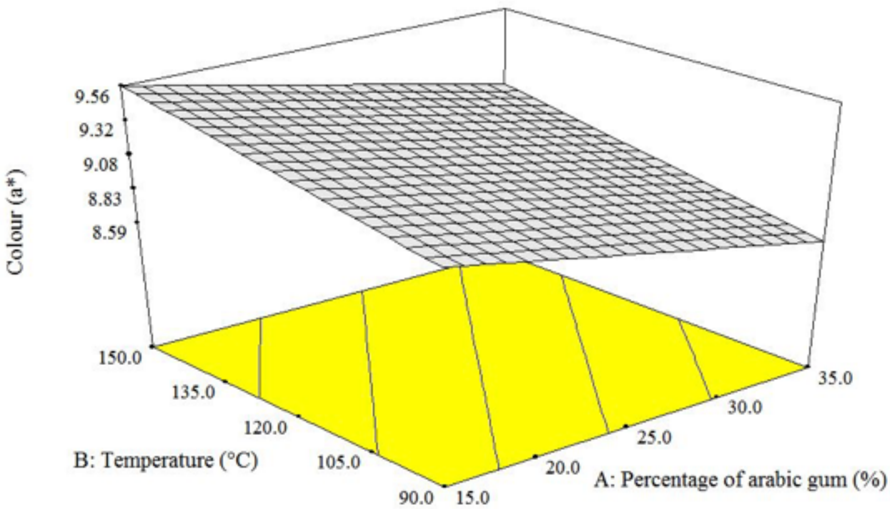

Figure 4A. Surface plot and contour plot for the interactive effects of percentage of arabic gum on the (*a**) colour values of drum dried *N. sativa* beverage powder.

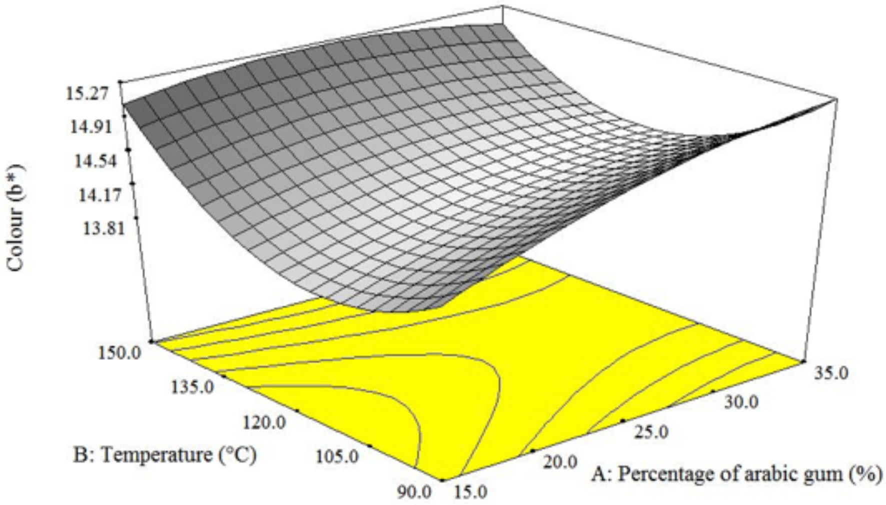

Figure 4B. Response surface plot and contour plot for the interactive effects of percentage of arabic gum on the (b^*) colour values of drum dried *N. sativa* beverage powder.

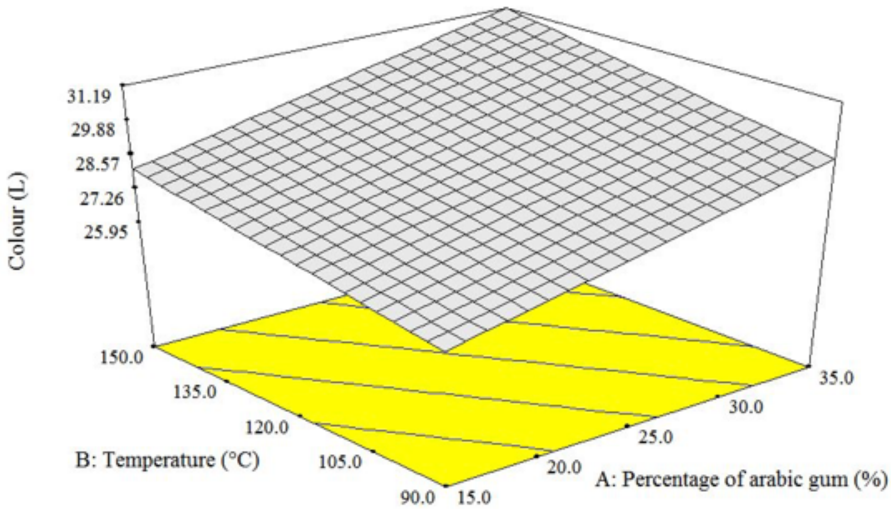

Figure 4C. Response surface plot and contour plot for the interactive effects of percentage of arabic gum on the (L) colour values of drum dried *N. sativa* beverage powder.

The trends showed that the L and a* values of drum dried powder significantly vary from its raw material when the temperature was higher. It can be speculated that such effect was due to the colour degradation caused by the high processing temperatures (ABONYI *et al.*, 2002).

3.3.4 Solubility

Solubility is achieved after the powder undergoes dissolution steps of sinkability, dispersibility and wettability (CHEN and PATEL, 2008). The solubility decreased as a function of both independent variables, such as percentage of arabic gum and drying temperature (Fig. 5). CANO-CHAUCA *et al.* (2005) pointed out that solubility problems arise with high processing temperature and high concentration of solids. FERNANDES et *al*. (2013) reported that longer time is needed to dissolve solids if the production occurred at higher inlet temperature, while depending on the concentration of wall material (arabic gum). This correlates well with the solubility results achieved in this study. Higher temperature could result in further degradation of the structure. Collapsed structures have led to a more compact and rigid product with lower porosity (CAPARINO *et al.*, 2012). The water uptake ability is facilitated by highly porous structure (GOHEL *et al.*, 2004). Thus, solubility is higher in highly porous structures.

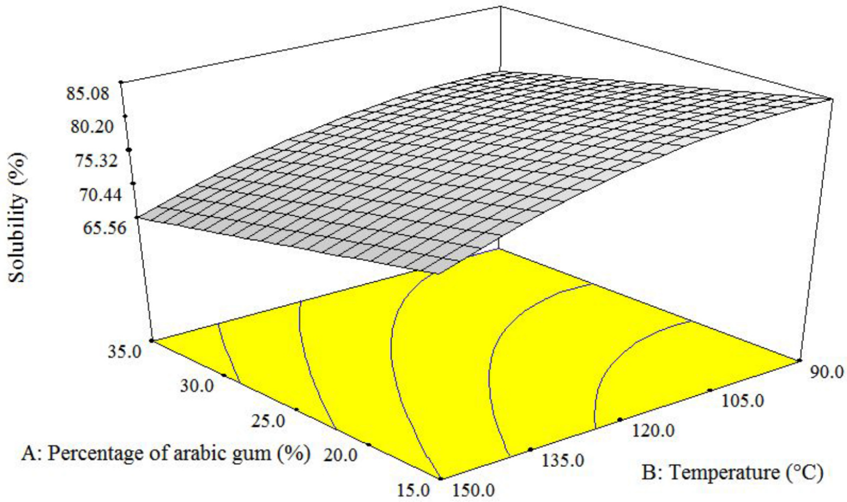

Figure 5. Response surface plot and contour plot for the interactive effects of percentage of arabic gum on the solubility of drum dried *N. sativa* powder.

3.3.5 Optimization

Optimization was carried out using response surface methodology to determine the optimum conditions for producing *N. sativa* powder with optimum physicochemical properties. This methodology utilizes graphical approach of superimposing the different response surfaces (Figs. 1-5) to generate an experimental region with desired values of the responses (ARTEAGA *et al.*, 1994). The suggested concentration of arabic gum and the temperature were 32.7% and 119.1°C respectively. Validation was done based on this optimal condition suggested, with modified drying temperature of 120°C due to the limitation of drum dryer. The results of the responses are recorded in Table 5.
Water activity of this drum dried N. powder fell under the same category as the whole milk powder. Water activity of less than 0.60 implies that the product is microbiologically stable (STEELE, 2004). The moisture content was within the range with reference to food products such as milk powder and instant coffee. Food standards by Food and Drug Administration (FDA) stated that the maximum desirable moisture content for milk powder is 5%, while the maximum desirable moisture content for instant coffee is 6% (F. D. A., 2013). However, Table 4 shows that the solubility of the powder produced at this optimum condition was not satisfactory, as no significant improvement was observed (p >

0.05) for the product to be reconstituted rapidly. Modifications on the processing methods or changing of the drying method should be made in order to achieve better solubility.

Table 5. Quality changes of roasted black seeds after drum drying.

Quality	Mean Value[A]±Standard Deviation		
	Raw Black seeds	Roasted Black Seeds	Drum Dried Powder[B]
Water activity, a_w	0.37±0.04[c]	0.31[a]±0.02	0.20[b]±0.03
Moisture Content (%)	1.53±0.05[c]	1.34[a]±0.06	1.26[a]±0.09
Colour:			
L	17.42±0.15[c]	20.23[a]±0.21	29.73[b]±0.16
*a**	1.73±0.12[c]	2.74[a]±0.67	8.77[b]±0.14
*b**	1.87±0.19[c]	2.15[a]±0.15	14.43[b]±0.22
Solubility (%)	68.53±0.75[c]	71.67[a]±0.76	75.38[a]±0.99

Different superscript letters in lower case are significantly different at $p< 0.05$ compared to roasted black seeds. [A]mean value±standard deviation (n = 3). [B]drum dried powder produced at optimal condition (percentage of arabic gum = 32.7%; drying temperature = 120°C).

3.4. Antioxidant properties

3.4.1 DPPH free radical scavenging effect

DPPH is a stable radical in which the maximum absorbance was observed at 515 nm. It steadily undergoes reduction by an antioxidant and is used to measure the antioxidant activity (ERKAN *et al.*, 2008). The DPPH radical scavenging activity determines the inhibitory activity of antioxidant constituents. Inhibition activity is attributed to a series of antioxidant components such as polyphenols, carotenoids, ascorbic acids, and others (SHIN *et al.*, 2015). Drying methods can affect the antioxidant activity significantly and the loss of 26.7% was observed with the roasted black seeds. HSU *et al.*, (2003) reported that the highest radical scavenging activity was observed in freeze dried yam flour compared with drum dried and airdried yam flour. Table 6 showed that the reduction of inhibitory activity in drum dried sample was significant ($p < 0.05$) compared to the roasted black seed. CHIA and CHONG, (2015a) also reported significant loss of antioxidant activity in drum dried dragon fruit peel powder. Heat treatments can breakdown phytochemicals. Thus, the integrity of cell structure was affected, leading to various chemical reactions and migration of components (DAVEY *et al.*, 2000). The reduction in antioxidant activity can be attributed to the degradation of phytochemicals during thermal processing (ZHANG and HAMAUZU, 2004).

3.4.2 Total Phenolic Content (TPC)

Phenolic compound constitute a large portion of plants antioxidants. The structure of phenolic compounds consists of one hydroxyl group substituted at different positions on the aromatic ring. Number and position of the hydroxyl groups affect the antioxidant as well as the identity of the main substituents. An example of the main substituent is carboxylic acid or linkage of the aromatic ring to another ring (RABABAH *et al.*, 2015). Compared with the roasted black seeds, which are the raw material for drum dried black seeds powder, the level of TPC decreased significantly ($p \leq 0.05$) in the drum dried seeds powder, the level of TPC decreased significantly ($p \leq 0.05$) in the drum dried powder (Table 6). Heating is capable of degrading phytochemicals. In addition, the inactivation of degradative enzymes such as polyphenol oxidases (which degrades phenolic compounds) occur during drying treatments leading to TPC loss. Intense heat treatment may be responsible for extensive loss of antioxidants, as most of these compounds are relatively unstable (LIM and MURTIJAYA, 2007). Extensive investigations were done previously on TPC reduction in relation to drying. However, no literature about drum dried *N. sativa* was recorded. Different heat treatments can lead to different degrees of TPC reduction. RABABAH *et al*., (2015) reported that the reduction of phenolic compounds in herbs was greater in oven drying method (40°C) than air drying method (24°C). In the present study, drum dried black seeds powder showed 31.4% of reduction in TPC. For drum dried products, CHIA and CHONG, (2015a) reported that 98.6% of TPC was retained in drum dried dragon fruit peel. In contrast, an almost complete loss of phenolic compounds such as tocopherols and tocotrienols was observed in drum dried oats. Other phenolic compounds such as avenanthramides and total cinnamic acids also showed a significant decrease in amount (BRYNGELSSON *et al*., 2002).

Table 6. Antioxidant properties of roasted black seeds and drum dried powder.

Antioxidant Properties	Mean Value[A]±Standard Deviation		
	Raw Black Seed	Roasted Black Seeds	Drum Dried Powder
TPC (mg/g)	17.83±0.30[c]	34.69±3.03[a]	24.74±0.54[b]
TFC(mg/mL)	16.40±0.63[c]	26.37±0.09[a]	18.65±0.18[b]
DPPH (%)	32.74±0.32[c]	45.66±0.75[a]	34.02±0.72[b]
FRAP(mmol/mg)	0.70±0.002[c]	0.87±0.003[a]	0.75±0.005[b]

Results display mean values±standard deviations (n = 6). [abc]Mean values within the same row that have different superscript letters indicate a significant difference ($P < 0.05$).

3.4.3 Total flavanoid content

The most abundant flavonoids found in Nigella sativa are epicatechin, (+) − catechin, quercetin, apigenin, amentoflavone and flavone (BOURGOU *et al.*, 2008). TPC and TFC are highly degradable due to heating and oxidation (FANG and BHANDARI, 2011; PATRAS *et al*., 2010). Results from this study showed that roasted black seeds were significantly higher in TPC, TFC, DPPH and FRAP compared to raw black seeds and the drum dried powder (Table 6). The low TFC, TPC and DPPH values in dried powder compared to roasted black seeds could be attributed to the presence of the carrier agent; Arabic gum. This has been proven in the past whereby increasing Arabic gum in samples of amla juice reduced the TPC, which in turn reduced the radical-scavenging capacity of the resulting powder (MISHRA *et al*., 2014). In spite of the decrease in antioxidant capacity, Arabic gum is known to be capable of preserving the antioxidative activity (COSTA*et al.,* 2015). This is evident from the fact that there was little difference in FRAP value, although significant. This could be due to the nature of Arabic gum, which has higher oxidative stability (COSTA *et al.,* 2015). Likewise, the heat in the drum drier could have contributed to the loss in the TPC. Heat treatments typically cause loss of naturally occurring polyphenol compounds, hence leading to reduced antioxidant capacity (DAGLIA *et al.,* 2000).

3.5. Minerals content

Table 7 shows the changes in various mineral contents after drum drying process. The results showed that calcium content decreased significantly ($p \leq 0.05$) after drum drying, whereas the iron, manganese and sodium content increased significantly ($p \leq 0.05$) after drying. However, calcium content was still the highest among the mineral content analysed, regardless of sample (either before or after drying). ÖZCAN *et al.*, (2005) reported that mineral content in basil were lost after drying. The decrease in mineral content and quality of dried product could be attributed to the high processing temperature used. The increase in concentration of mineral contents such as iron, manganese and sodium were most probably enhanced by the mineral contents found in the arabic gum itself.

Table 7. Minerals content of roasted black seeds and drum dried powder.

Minerals Content	Mean Value^A±Standard Deviation		
	Raw Black Seed	Roasted Black Seeds	Drum Dried Powder
Calcium (ppm)	7.23±0.13^a	6.63±0.27^b	4.34±0.05^c
Iron (ppm)	2.07±0.06^a	0.72±0.01^c	1.90±0.03^b
Manganese (ppm)	0.82±0.02^a	0.06±0.01 ^{b}	0.37±0.01^b
Sodium (ppm)	6.32±0.14^a	3.04±0.08^b	5.54±0.10^b

Results display mean values±standard deviations (n = 3). ^{abc}Mean values within the same row that have different superscript letters indicate a significant difference ($P < 0.05$).

3.6. Sensory characteristics

Results of sensory evaluation in terms of appearance, aroma, taste, texture and overall acceptability are presented in Table 8. The panellists judged each specific sensory parameter as acceptable, with a mean score equal to 6 (like slightly) or higher. The evaluation showed significant differences in taste and aroma among samples, with the beverage from roasted black seed powder scoring higher, compared to the beverage prepared from drum dried black seed powder.

Table 8. Mean of hedonic scales for panellist's acceptance of beverage prepared from drum dried black seed powder and from roasted black seed powder.

	Appearance	Aroma	Taste	Texture	Overall acceptability
Drum dried black seed	5.89±1.54^a	5.89±1.08^b	5.02±1.12^b	6.12±0.87^a	6.13±1.21^a
Roasted black seed	6.14±1.03^a	6.59±1.19^a	6.32±1.75^a	5.56±1.43^b	6.10±1.37^a

Results display mean values±standard deviations (n = 30). ^{abc}Mean for each characteristic followed by the same superscript letter within the same column are not significantly different at $P < 0.05$.

However, drum dried powder scored higher in texture than the roasted black seed powder. There was no significant difference in appearance and overall acceptability between samples. Hedonic scales for the overall acceptability of both beverages from roasted black seed powder and drum dried black seed powder were higher than 6 (like slightly), indicating that both samples were at acceptable level.

4. CONCLUSIONS

RSM is useful in the optimisation of product formulation and processing conditions. The two independent variables, namely percentage of arabic gum and drying temperature significantly affected all the responses measured which include yield, water activity, moisture content, colour values L, a*, and b*, and solubility of the final product. Based on the optimization, the optimum percentage of arabic gum and the drying temperature are 32.7% and 119.1°C respectively. However, at these optimised conditions, the solubility of the powder was low, which could significantly affect the feasibility of using the method to produce instant N. sativa powder. The drum dried product showed significant reduction ($p \leq 0.05$) in total phenolic content as well as antioxidant activity as determined by the DPPH free radical scavenging activity. Significant loss ($p \leq 0.05$) was detected for calcium content in the drum dried product, but the iron, manganese and sodium contents increased significantly ($p < 0.05$) after drying. The overall sensory acceptability score was higher than 6 (like slightly), indicating the beverage was at an acceptable level.

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