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Encapsulization of *Channa striatus* Extract by Spray Drying Process

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Abstract: *Channa Striatus* is known as snakehead fish or local name as haruan, has been always associated with its medical value especially in wound healing. This fish is rich in protein as well as others biochemical compounds such as polyunsaturated fatty acids and antioxidants. Usually, the haruan is extracted and marketed in the form of liquid concentrated as health food supplement. In this study, encapsulated haruan extract were produced using spray drying process. The main purpose is for easy handling and the preservation of the biochemical compounds. The biochemical compound in the powder produced is expected to have properties such as more stable and longer shelf life. K-carrageenan was used as coating material for the encapsulzation during the spray drying process. The properties of encapsulated powder produced were observed in term of particle size distribution, Fish Protein Hydrolysates (FPH) and moisture content. The process parameters of spray drying process studied were hot air inlet flow rate, temperature and the liquid feed flow rate. The experimental run and optimization were designed using Box-Behnken method as suggested by Response Surface Methodology (RSM). The optimum operation conditions for highest protein extracted with lowest moisture content and smallest particle size distribution were obtained at hot air inlet temperature and flow rate of 144.51°C and 400 mL h^{-1}, respectively; whereas, the liquid feed flow rate is at 47 m^3 h^{-1}. The optimal properties of encapsulated powder obtained were 5.2850 μm, 91% of protein and 8.7% in moisture content.

Key words: K-carrageenan, wound healing, fish protein hydrolysates, health food supplement

INTRODUCTION

Haruan or also known as murrel in west countries is belonged to the family Channidae or Ophiocephalidae and its scientific name is *Channa striatus* (Pillay and Kutty, 2005). *Channa striatus* is different from other fishes as it possesses ability to breathe atmospheric oxygen and this keeping them alive long even out of water. It is well known as food fish in South and Southeast Asian countries. In Malaysia, haruan is also considered by most Malaysian as a good source of health food. Besides the high quality of the flavor and texture of the flesh, *C. striatus* is widely believed by Malaysians to have remedial effects in ameliorationg wound lesion, port-parturition and various skin diseases. This is because the *C. striatus* contains lots of polyunsaturated fatty acids (PUFA). A fatty acid compositional study of the flesh of Haruan revealed unusually high arachidonic acids (ARA) but almost no eicosapentaenoic acids (EPA) which were hypothesized to be active component in the initiation of wound repair (Mat Jais *et al*., 1994). Essential amino acids such as glycine and essential fatty acid such as arochidonic acid have been shown to actively participate in the normal blood clotting mechanisms by facilitating wound healing as well as in enhancing the antinociceptive activity (Mat Jais *et al*., 1997; Baie and Sheikh, 2000). There were extensively studies on the beneficial effect of *C. striatus* in wound healing, antinociceptive activities and composition of the amino acids in the *C. striatus*. However, this study is limited to the extract of the fish fillet in the liquid form and no research has been done to study on the extract in spray dried powder form. Currently, there are products of the haruan extract in the market are in the liquid form for consumption purpose and also in the cream form which utilized for external treatment on the skin. The essence of haruan in liquid form often exhibits some problems like short lifespan, handling problems. The haruan extract in liquid form also very often present a physicochemical instability during their storage as there is present of water. Moreover, the extracts in liquid form have restriction in the mobility of the product as it may be too heavy to be carried along by the consumers.

Therefore, in this study, spray drying method is utilized to spray dried the haruan extract. Spray drying is the common method for producing a dry powder from a

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liquid (Masters, 1985). It results in powders with controllable particle sizes, low moisture and easier transport and storage (Tonon *et al*., 2008). In this study, the relationship between the some process variables and resulting the characteristics of encapsulated haruan extract is also studied. The encapsulisation process parameters explored were spray dryer inlet temperature, liquid feed rate and the drying air flow. The powder characteristics such as size distribution, protein and moisture content were analyzed.

MATERIALS AND METHODS

The research conducted here was involving of selection of materials, extraction of bio-compounds from haruan fish, encapsulisation of the fish extract and characterization of encapsulated product. The extraction of bio-compounds from fish was using conventional boiling technique, whereas the production of extract powder by spray drying technique. The characterization of extract powder was particle size distribution, protein and moisture content. Experimental design was also used to assist the selection of the range of process parameter.

Materials: The haruan fish was supplied from the local farm and the weight of the fishes were in the range of 400 to 700 g. The wall materials (K-carrageenan) for encapsulisation was obtained from Fluka. Other analytical chemicals were purchased from Sigma Aldrich.

Preparation of haruan extract: Haruan fish weighing 400 to 700 g were utilized. The live fish was weighed and pre-cleaned with distilled water and is placed into a plastic bag which filled with distilled water. The distilled is at ratio 1:1 to the weight of the fish. The plastic bag containing the fish was then being placed into the freezer at 0°C for overnight. This is because the haruan will secret the mucus as protection when placed in low temperature environment. This can ease the process of cleaning the fish as all the mucus has been secreted out into the distilled water. The frozen fish was then thawed and the fish fillets were obtained by carefully cutting the fish lengthwise along the backbone to get the maximum amount of flesh without any bones. The skin of the haruan fish is cleaned as only fish fillet needed in this study. The fish fillet was then rinsed using distilled water and was weighed for the extraction process.

The fish extraction process is carried out using a pressure cooker set at 100°C for 2 h (Mat Jais *et al*., 1994). The boneless fillet was cleaned using distilled water and weighed. It then was placed into the stainless steel pressure cooker and distilled water was added into it. The distilled water was added with the ratio (fish: water volume) of 1:4. The fish fillet is cooked for 30 min, distilled water was added to original volume before it was further cooking for another 30 min. The steps were repeated every 30 min up to total 2 h cooking time. Finally, the used fish fillets were discarded and the liquid extract was collected. The fish extract was then filtered and stored at 4°C.

Spray drying: Prior the spray drying process, a solution of 0.5% K-carrageenan was added to the fish extract at the volume ratio (K-carrageenan:haruan extract) of 1:9. Spray drying was performed in a laboratory scale spray dryer LabPlant SD-05 (Huddersfield, England), with a 1.5 mm diameter nozzle and a main spray chamber of 500× 215 mm. The mixture was fed into the main chamber through a peristaltic pump and the feed flow rate was controlled by the pump rotation speed. Inlet air temperature varied from 130 to 150°C, the feed flow rate varied from 200 to 400 mL h^{-1} and the drying air flow rate varied from 47 to 62 m^3 h^{-1}. The air pressure was maintained at 1.2 MPa.

Analytical methods

Particle size distribution: The particles size distribution was analyzed using the Laser Scattering Particles size Distribution Analyzer LA-300 (Horiba) (Mat Jais *et al*., 2008). The measurement technique was conducted according to the operation manual of the instrument given by instrument manufacturer.

Protein content: Kjedahl method was applied to measure the protein content in the haruan extract powder (Zakaria *et al*., 2007). Three steps were involved in the determination of protein content which are digestion, titration and distillation. Digestion was done with digester at 80°C for 2 h. The process of titration and distillation was conducted using Kjeltech instrument which is a part of the operation of this instrument.

Moisture content: The moisture content was analyzed using the HG53 Halogen Moisture Analyzer (Mettler Toledo). A quantity of 0.3 g of the extract powder sample was placed in an aluminum plate, the powder was heated to dry off the moisture for 5 min and the moisture content was identified.

Experimental design: The experimental design was done with the aid of the Design Expert software which uses the Response Surface Methodology, Box-Behnken design to determine the number of runs needed in this study. Response surface methodology was used as this study was aimed at process optimization which means finding out the best combination of parameters which can produce the quality fine particles (Awang Bono *et al*., 2008). In Box-Behnken design, each numeric factor which is the process parameter was varied over 3 levels (-1,0,+1). The range for each and every parameter was; inlet air temperature: 130, 140 and 150 °C, feed flow rate: 200, 300 and 400 mL h^{-1} and air flow rate: 47, 45.5 and 62 m^3 h^{-1}.

RESULTS AND DISCUSSION

Response surface analysis: The responses measured from the experimental work are shown in Table 1. Total up to 17 run out of three factors. The ANOVA analysis is shown in Table 2.

The quadratic models for particle size, protein content and moisture content in terms of coded factors are shown, respectively in the Table 2.

Particle size distribution: The influence of the different inlet drying temperatures (130, 140 and 150°C) on the particle size distribution is shown in Fig. 1a. The particle size decreased gradually with increasing temperature until it reached a minimum particle size at about 140°C. Above 140°C, the particle size started to increase gradually. The increase on inlet air temperature resulted in larger particles, which is related to the higher swelling caused by higher temperature. The particles remains shrunk when inlet drying air temperature is low and thus with smaller diameter (Tonon *et al*., 2008). According to Reiniccius (2001), larger particles will be formed when it is dried at conditions that result in faster drying rates than drying conditions that result in slower drying rate. This is due to the fact that very fast drying sets up as early structure and does not allow the particles to shrink during drying. When inlet temperature is low, it will results in slower drying and the particle will shrink and thus results in smaller particle diameter. Nijdam and Langrish (2006) who carried out study with the production of milk powder at 120 and 200°C obtained similar results. Hsu *et al*. (1996), presented a theory based on the fact that a skin is formed on the outer surface of the spray droplets at high inlet drying air temperature. The outer skin formed is destroyed when the inner water phase is evaporated and the outer surface collapse. From Fig. 1b, the particle size increases with increasing air flow rate until a maximum particle size is produced at air flow rate of about scale 35. Above that, the particle size decreases with the increasing air flow rate. Theoretically, the particle size should reduce with increased inlet air flow rate. According to Stahl *et al*. (2002), increased atomization nozzle flow which is equivalent to the inlet air flow rate reduced the particle size. This is due to the higher the atomization flow or air flow rate, the more energy is supplied for breaking up the liquids into droplets during the atomization step, resulting in smaller droplets formed (Masters, 1985). From the Fig. 1c, it is cleared that the mean particle size increases with the pump flow rate which also known as feed flow rate of the *C. striatus* extract with k-carrageenan. According to Rattes and Oliveira (2007), the increase in

Table 1: Measurement results of particle sizes, protein content and the moisture content suggested by experimental design

Run	Inlet air temperature	Feed flow rate	Air flow rate	Particle size (μm)	Protein content (%)	Moisture content (%)
1	0	0	0	18.4313	86.596	8.70
2	-1	0	-1	20.1868	83.0351	10.26
3	0	0	0	20.8186	85.4244	9.49
4	0	-1	-1	2.1042	88.2681	8.73
5	+1	+1	0	4.7985	85.4463	8.70
6	+1	0	+1	6.4442	83.1857	9.89
7	0	+1	+1	5.0857	83.6808	8.66
8	0	+1	-1	5.4960	90.9866	8.66
9	0	-1	+1	15.8730	82.7044	8.66
10	-1	0	+1	17.3525	82.0154	12.31
11	0	0	0	15.2504	86.1161	8.93
12	0	0	0	15.5015	86.0858	9.22
13	0	0	0	19.8664	84.9359	9.59
14	-1	+1	0	22.0553	84.4846	14.29
15	-1	-1	0	25.3214	81.5035	11.07
16	+1	0	-1	16.8815	92.1062	13.79
17	+1	-1	0	22.7043	88.1954	14.98

Table 2: ANOVA analysis for response surface quadratic model of the haruan extract powder properties

	Intercept	A	B	C	A^2	B^2	C^2	AB	AC	BC
Particle size	17.97	-4.26	-3.57	0.01	4.41	-3.67	-7.17	-3.66	-1.90	-3.54
Protein content	85.83	2.24	0.49	-2.85	-1.12	0.20	0.38	-1.43	-1.98	-0.44
Moisture content	9.19	-0.07	-0.39	-0.24	2.98	0.094	-0.60	-2.38	-1.49	0.02

Where, A: Inlet drying temperature, B:Pump flow rate or feed rate and C: Air flow rate

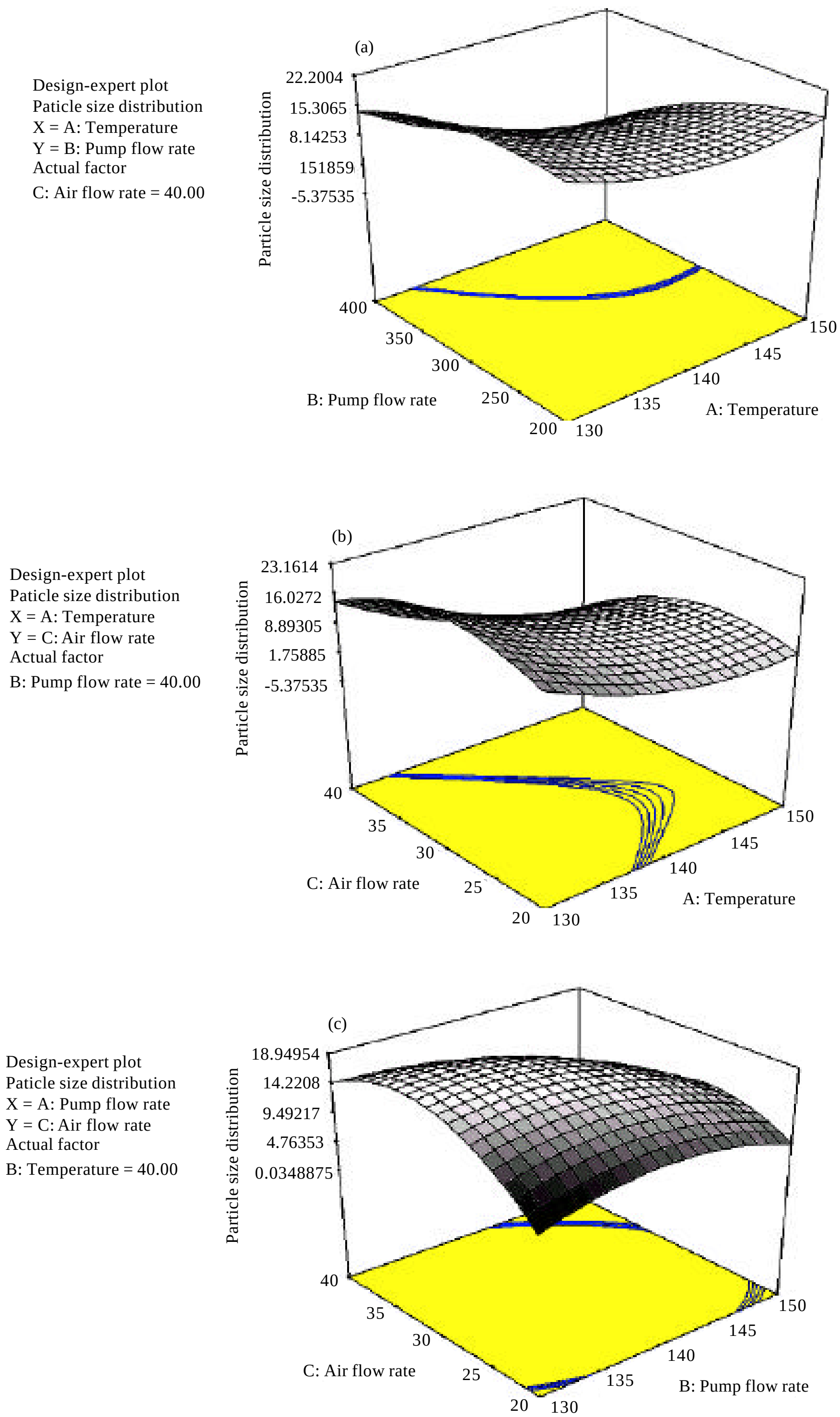

Fig. 1: (a) Particle size distribution for air flow rate of scale 40 which is 62 $m^3 h^{-1}$, (b) Particle size distribution for pump flow rate of scale 400 mL h^{-1} and (c) Particle size distribution for inlet air temperature of 140°C

feed flow rate augment the mean diameter of the atomizing drops during spray drying and thus contribute for the increase in the mean powder diameter.

Protein content: From Fig. 2a, it is cleared that the protein content increased exponentially with increasing inlet drying temperature. The effect of the inlet drying temperature on the protein content depends on its effect on moisture content of the powder produced. The moisture content is inversely proportional to the protein content of the powder. The higher the moisture content of the powder, the lower the protein content contained in the powder formed. This is due to as the water evaporated from the spray droplets, the protein content inside the powder become more concentrated and thus resulting in higher protein content within the powder itself. Thus, the protein content increased with increasing inlet air drying temperature. Besides, when the inlet drying air temperature increased, the size of the spray droplets also increased. When the size of spray droplets become larger, the inside component that can be shielded by the external shell also increases and contribute to higher protein content (Zhou *et al.*, 2004).

The effect of the inlet drying air flow rate and pump flow rate on the protein content also depends on its effect on the moisture content of the powder. When the inlet drying air flow rate increases, the residence time of the spray droplets in the drying chamber is decreased and thus resulting in higher moisture content and thus lower protein content of the powder formed (Fig. 2b, c).

Moisture content: From the Fig. 3a, the moisture content decreased with increasing inlet drying temperature until minimum moisture content at about 140°C. Above the 140°C, the moisture content started to increase gradually until 150°C. Ranging over the pump flow rate, the same trend happened. The powder moisture content decreased with increasing inlet drying temperature. The inlet drying temperature was the variables that showed greatest influence on powders moisture content. This is because at higher inlet air temperatures, there is a greater temperature gradient between the atomized feed and the drying air, there is a greater heat transfer into the particles, resulting in a greater drying force for water evaporation and thus producing powders with lower moisture content (Quek *et al.*, 2007). Goula and Adamopolous (2005) who was working with the spray drying of the tomato pulp in dehumified air also concluded that an increase in the inlet air drying temperature leads to a decrease in moisture content. When the drying medium is air, temperature plays a vital role. As water is driven from the particles in the form of water vapor, it must be carried away. Or else, the moisture will create a saturated atmosphere at the particle surface and this will slow down the rate of subsequent water removal. The hotter the air, the more moisture it will be hold before becoming saturated. Thus, high temperature in the vicinity of the drying particles will take up the moisture that being driven out from the food to a greater extent compared to cooler air (Goula and Adamopolous, 2005) and thus resulting in smaller particles formed. Rattes and Oliveira (2007) and Grabowski *et al.* (2006) also observed the same finding that is a reduction of powder moisture content with the increasing inlet drying air temperature, studying the spray drying of watermelon juice, sodium diclofenac and sweet potato puree, respectively.

However, the increases of inlet air drying temperature leads to a lower moisture contents do have exception for the increase from 140 to 150°C with a drying air flow rate of 62 $m^3 h^{-1}$ (Fig. 3b). Goula and Adamopolous (2003) also observed this exception when the inlet air temperature increases from 130 to 140°C, the moisture content of the particle increases. In this case, due to the high air temperature, the surface of the thermoplastic particles remains plastic resulting in sticking of the drying droplets among themselves and the drying rate decreases as the surface area of contact decreases (Goula and Adamopolous, 2003).

From the Fig. 3b, the powder moisture content increases with an increase in the drying air flow rate. Generally, the amount of drying air determines the energy available for evaporation. The movement of air predetermines the rate and degree of droplets evaporations by influencing the passage of the spray through the drying zone in the drying chamber, the concentration of the product in the region of dryer's walls and the extent to which the semi-dried particles re-enter the hot areas around the air disperser (Goula and Adamopolous, 2005). When the drying air flow rate is low, it causes an increase in the product sojourn time in the drying chamber (Masters, 1979) and enforces circulation effects (Goula and Adamopolous, 2004; Oakley and Bahu, 1991). Increased residences times in the drying chamber lead to a greater degree of moisture removal. In conclusion, an increase in the drying air flow rate decreases the residence time of the spray droplets in the drying chamber and thus led to lower moisture removal and higher moisture content in the spray powder. The pump flow rate or feed flow rate negatively influences the powder moisture content as shown in the Fig. 3c. The increase in the pump flow rate will result in the powder with greater moisture content. This is because of higher flow rates imply in a shorter contact time between the feed and the drying air, making the heat transfer between the

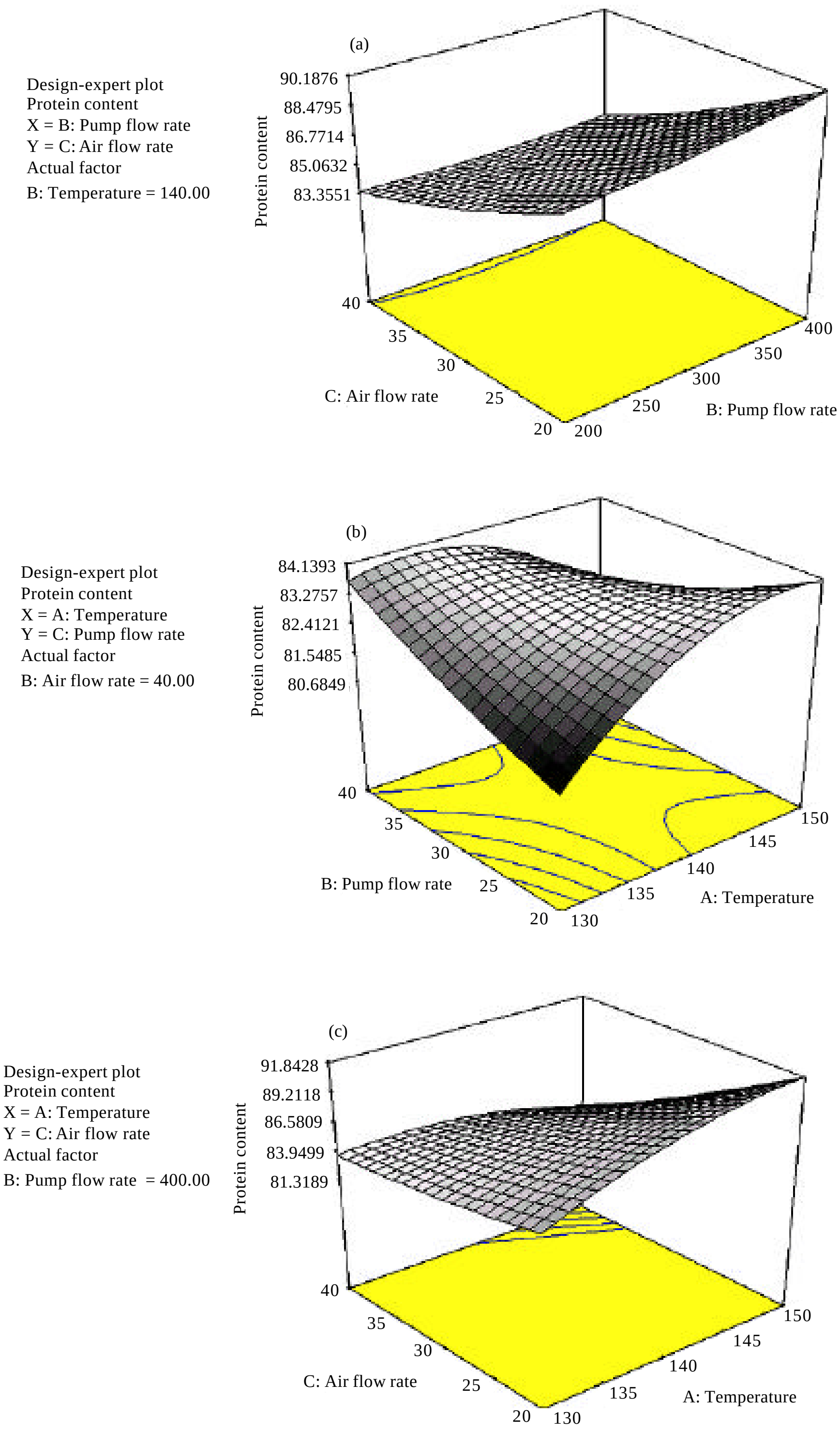

Fig. 2: (a) Powder protein content for inlet air temperature of 140°C, (b) Powder protein content for air flow rate of scale 40 which is 62 $m^3 h^{-1}$ and (c) Powder protein content for pump flow rate of scale 400 $mL h^{-1}$

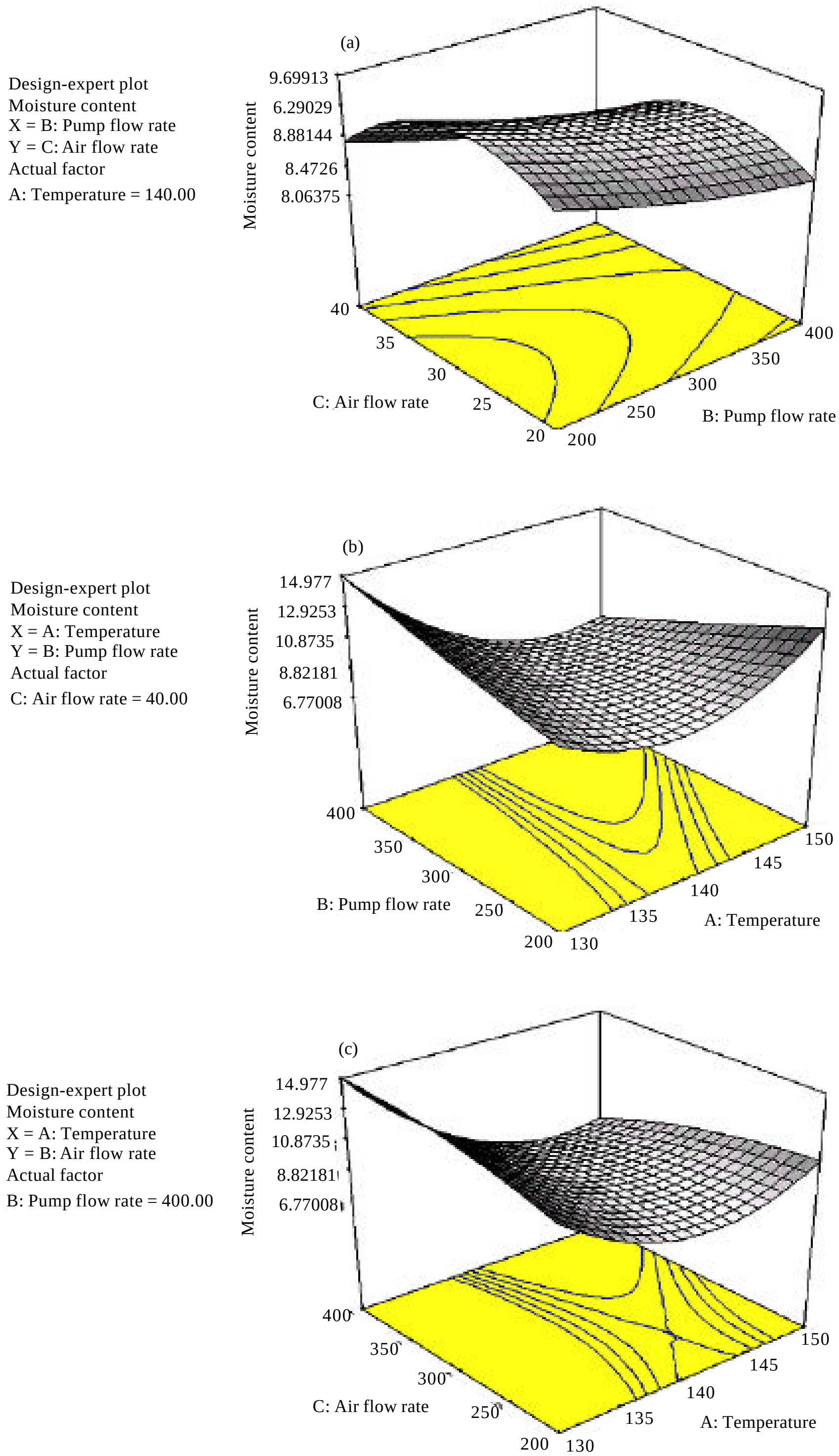

Fig. 3: (a) Powder moisture content for inlet air temperature of 140°C, (b) Powder moisture content for air flow rate of scale 40 which is 62 $m^3 h^{-1}$ and (c) Powder moisture content for pump flow rate of scale 400 $mL\ h^{-1}$

feed and the drying air less efficient and resulting in lower water evaporation (Tonon *el al.*, 2008). Hong and Choi (2007) who carried out study on the physicochemical properties of protein bound polysaccharide from Agaricus blaze Murill by ultrafiltration and spray drying process, had verified that the powder moisture content increased with increasing pump flow rate and with decreasing inlet air temperature and the effect of the temperature was greater than the effect of the pump rate.

Optimization of process parameters on spray drying process: The result obtained from RSM showed that the optimum process parameters, the inlet drying temperature is 144.56°C, the pump flow rate is 400 mL h^{-1} and air flow rate is 47 m^3 h^{-1} whereas the optimum responses of the process, particle size distribution is 5.2744 μm, protein content is 91.2124% and mean size and moisture content is 8.6915% with desirability of 0.923.

CONCLUSION

The effects three process parameters or variables which are inlet drying temperature, the pump flow rate or feed rate and nozzle gas flow rate on the physical and chemical properties of the spray dried *C. striatus* extract have been evaluated by 3 levels Box-Behnken experimental design. In conclusion, the inlet drying temperature showed significant effect on all the responses; the particle size distribution, the protein content, the moisture content studied. It was observed that:

- The powder particle size decreases with a decrease of feed flow rate and inlet temperature. However the decreases of air flow rate will rised the powder particles sizes
- The decreases of air flow rate and feed flow rate will raise the protein content. The increases of air temperature will also raise the protein content of the powder
- The powder moisture content was reduced with the increases of inlet air temperature and decreases in both of feed and air inlet. flow rate

The result obtained from RSM showed that the optimum process parameters, the inlet drying temperature is 144.56°C, the pump flow rate is 400 mL h^{-1} and air flow rate is 47 m^3 h^{-1} whereas, the optimum responses of the process, particle size distribution is 5.2744 μm, protein content is 91.2217% and mean size and moisture content is 8.6915%. There were seven combinations of the optimized process parameters with optimum value with predicted desirability.

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