PERFORMANCE AND OPTIMIZATION OF SPRAY DRYING TECHNIQUE ON THE PRODUCTION OF MICROPARTICLES OF HARUAN, *Channa striatus* EXTRACT

LEE YUN HUI

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2015

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ABSTRACT

Channa striatus, the fish a carnivorous, air breathing freshwater species can be found throughout Malaysia including Sabah and Sarawak. It is well known as one of the famous ethno-pharmacology for wound healing and being consumed almost everywhere in the community of Malaysia. Thus, this research is an innovative work to carry out the transformation of *Channa striatus* to microparticles using spray drying technique. To carry out the research, it was firstly, fresh *Channa striatus* supplied by local farmer undergone an overnight hypothermic stress treatment with temperature below -20°C and filleted prior to cook in pressure cooker for an hour with distilled water. The extract was cooled to room temperature, before filtration using vacuum pump for purification. Filtered *Channa striatus* extract was encapsulated with three different types of binders which were carboxymethylcellulose (CMC), maltodextrin and xanthan gum in fixed concentration and spray dried distinctively to produce microparticles. The spray drying process were conducted by altering the process parameters which were drying temperature, air flow rate, pump flow rate and binder concentration for the purpose of optimization in order to produce microparticles that are high in the content of protein and antioxidant with minimum size and moisture content. The *Channa striatus* microparticles then were used for physico-chemical analysis namely particle size distribution, moisture content, protein content using Kjeldahl method and antioxidant activity using DPPH radical scavenging method. The results obtained from two-level factorial designs shows that, the optimum spray drying process to produce *Channa striatus* microparticles in the sequence of drying temperature, pump flow rate and air flow rate are; CMC binder was 130°C, 151.04 ml/hr, 55.13 m^3/hr, 0.7% w/v binder produced microparticles with the size of 4.4µm, moisture content of 5.1%, antioxidant activity of 54.55% and protein content of 49.07% ; maltodextrin binder was 130°C, 150.30 ml/hr, 55.00 m^3/hr, 0.7% w/v binder which produce microparticles with the size of 11.5µm, moisture content of 4.1%, antioxidant activity of 46.22% and protein content of 46.49% and xanthan gum binder was 150°C, 150 ml/hr, 59.51 m^3/hr, 0.5% w/v binder which produce microparticles with the size of 2.98µm, moisture content of 7.9%, antioxidant activity of 53.32% and protein content of 53.52%. The size of the microparticles obtained are in the scale of micron size, there is potential in producing *Channa striatus* particles in nanosize by refining the spray drying process in the aspects of spray drying parameters. Furthermore, the percentage of protein and antioxidant that was observed in the microparticles proven that *Channa striatus* microparticles produced with spray drying technique have a good prospective to be marketed as supplement or drugs for wound healing remedy.

ABSTRAK

PENGOPTIMUMAN PARAMETER DALAM MENGHASILKAN ZARAH MIKRO HARUAN (CHANNA STRIATUS) DENGAN PROSES SEMBURAN KERING

Channa striatus, ikan karnivor, spesis air tawar pernafasan udara yang boleh didapati di seluruh malaysia termasuk Sabah dan Sarawak. Haruan dikenali sebagai salah satu etno-farmakologi untuk pemyembuhan luka dan merupakan makanan yang dinikmati oleh masyarakat Malaysia. Channa striatus adalah asli untuk Malaysia, dan projek ini merupakan sebahagian daripada penemuan R & D untuk menghasilkan ekstrak Haruan sebagai agen terapeutik untuk penyembuhan luka dan anti-kesakitan. Kajian ini merupakan penyelidikan inovatif yang dilaksanakan untuk mengubah cecair ekstrak akueus Haruan menjadi serbuk dengan menggunakan teknik semburan kering, untuk menghasilkan zarah mikro yang kecil. Hipotesis untuk projek ini adalah teknik semburan kering berupaya menghasilkan zarah nano, yang kemudiannya berpotensi untuk dirangkumkan ke dalam kapsul atau tablet sebagai ubat. Pertama kalinya, penyelidikan ini dijalankan dengan membuat rawatan hipotermik ke atas Haruan segar untuk satu malam dibawah suhu -20°C. Kemudiannya,isi ikan Haruan akan dimasak dalam periuk tekanan selama satu jam dengan air suling untuk meghasilkan ekstrak. Ekstrak dibiarkan sejuk pada suhu bilik, untuk tujuan penapisan dan penulenan. Ekstrak Haruan yang ditapis akan dikapsulkan dengan tiga jenis pengikat iaitu carboxymethylcellulose (CMC), maltodekstrin dan gam xanthan dalam kepekatan yang ditetapkan dan disembur kering untuk menghasilkan serbuk Haruan. Proses semburan kering dijalankan dengan beberapa parameter iaitu suhu pengeringan, kadar aliran udara dan kadar aliran pam untuk mengoptimumkan proses penghasilan zarah Haruan bersaiz minimum, kandungan protein dan antioksidan yang tinggi dan rendah lembapan. Keputusan yang diperolehi dari reka bentuk faktorial dua peringkat menunjukkan bahawa, keadaan optimum proses semburan kering dalam menghasilkan zarah Haruan mengikut urutan suhu pengeringan, kadar aliran pam dan kadar aliran udara adalah; pengkapsulan CMC haruan adalah 130°C, 151,04 ml/jam, 55.13 m^3/jam, 0.7% w/v pengikat dan zarah Haruan bersaiz 4.4µm, lembapan 5.1%, aktiviti antioksidan 54.55% dan kandungan protein 49.07%; pengkapsulan maltodekstrin Haruan adalah 130°C, 150.30 ml/jam, 55.00 m^3/jam, 0.7% w/v pengikat menghasilkan dan zarah Haruan bersaiz 11.5µm, lembapan 4.1%, aktiviti antioksidan 46.22% dan kandungan protein 46.49% dan pengkapsulan gam xanthan Haruan adalah 150°C, 150 ml/jam , 59.51 m^3/jam, 0.5% w/v pengikat menghasilkan dan zarah Haruan bersaiz 2.98µm, lembapan 7.9%, aktiviti antioksidan 53.32% dan kandungan protein 53.52%. Saiz zarah Haruan yang diperolehi adalah dalam skala mikron, nano saiz dapat dihasilkan jika proses ditambah baikkan. Keputusan protein dan antioksidan dalam zarah Haruan membuktikan bahawa zarah Haruan yang dihasilkan dengan teknik semburan kering mempunyai prospek yang baik untuk dipasarkan sebagai suplemen atau ubat penawar untuk penyembuhan luka.

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Lee Yun Hui

3 December 2014

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LIST OF ABBREVIATIONS

nm	-	Nanometer
µm	-	Micronmeter
^oC	-	Degree celcius
β	-	Beta
%	-	Percentage
w/v	-	Weight over volume
g	-	Gram
Kg	-	Kilogram
L	-	Liter
CMC	-	Carboxymethlcellulose
mL/ hr	-	Milliliter over hour
m^3/ hr	-	Meter cube over hour
DPPH	-	1, 1-diphenyl-2-picryhydrazyl
mM	-	Millimole
K_2SO_4	-	Potassium sulphate
$CuSO_4.5H_2O$	-	Copper sulphate pentahydrate
H_2SO_4	-	Sulfuric acid
HCl	-	Hydrochloric acid
NaOH	-	Sodium hydroxide
H_3BO_3	-	Boric acid
RSM	-	Response Surface Methodology
g/mL	-	Gram over milliliter
3D	-	Three dimension
HPLC	-	High performance liquid chromatography
GCMS	-	Gas chromatography mass spectrometry

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CHAPTER 1

INTRODUCTION

1.1 Overview

Channa striatus is known as Haruan or snakehead fish is an indigenous to Malaysia. This fish usually called as 'Haruan' (malay) or 'Sang Yi' (chinese). This species come from the family of *Channidae* which is a fresh water or pond-cultured fish. It is also known as juveniles that can perform high levels of cannibalism (Qin and Fast, 1996:314). This type of fish can be widely found in Southeast Asian countries such as Malaysia, Indonesia, Philippines and Thailand. This species of fish usually can be found anywhere as long as water is present; such as lakes, rivers, ponds, paddy fields and river mouths. Furthermore, it is considered as the source of protein for many of the countries in the region of Asia Pacific (Mat Jais, Dambisya, and Lee, 1997: 125). The common range of physical weight for Haruan is 1.0 to 2.0 kg and their length of 25 to 30 cm (Rahim, Rozila and Mat Jais, 2009: 994).

Haruan is well recognized as remedy to promote wound healing after injuries or surgery due to the content of acids that can initiate tissues for wound healing (Manan, Jais, Matori, Kittakoop, and Sowanborirux, 1998: 561). Haruan contain high biocompounds such as polyunsaturated fatty acids (PUFA) and amino acids that responsible in the synthesis of lipids compound, prostaglandin which found in animal tissue and thus enhance the wound healing process. Biochemical components such as amino acids (protein) and polyunsaturated fatty acids (lipid) play an important in preserving the health of mankind. This can be proven by the study of Mat Jais that *Channa striatus* contains ample of fatty acids and amino acids with the amount of seven and seventeen of fatly acids and amino adds respectively (Mat Jais, McCulloch and Croft, 1994; Zuraini Somchit, Solihah, Goh, Arifah, Zakaria, Rajion and Mat Jais, 2006). Other than lipid and protein, this study also includes the antioxidant activity in Haruan. The research of Galla et al., 2012, has shown the

existence of bioactive compound of antioxidant in Haruan's roe (Galla, Pamidighantam, Akula, and Karakala, 2012). Antioxidants are known as the natural biocompound that contain high health value which receive much attention and interest among the researchers. Plants and fruits are the all-time natural antioxidants sources; however fishes also contain antioxidants that are beneficial to human health (Arbeloa, Uez, Bertolotti, and Churio, 2010).

There are many research that are related to the analysis of *Channa striatus* globally such as the content of Angiotensin converting enzyme (ACE) inhibitory peptides in Haruan to fight high blood pressure (Ghassem, Arihara, Salam, Said, and Ibrahim, 2011). The study of the influence of temperature, PH and naloxone on the activity of Haruan (Mat Jais *et al.*, 1997; Dambisya, Lee, Sathivulu, and Mat Jais, 1999). The composition of fatty acid and amino acid (Mat Jais et al., 1994) and physical and chemical morphology of Haruan (Rahim *et al.*, 2009) in Malaysia. Not only that, the rate of cannibalism of Haruan in United States of America (Qin and Fast , 1996) and as well as the isolation of Mycobacterium conferee from snakehead fish for tuberculosis lesion analysis in Italy (Tortoli, Bartoloni, Bozzetta, Burrini, Laccini, Mantella, Penati, Simonetti and Ghittino, 1996). The effect of synthetic phyrethroid on the enzyme system of *Channa striatus* in India (Singh and Srivastava, 1999) and many more. The beneficial properties of haruan to human being are the main reason of these studies to be carried out. Furthermore, the high potential of haruan in food and pharmaceutical fields has gain the interest for more researches to be done. The Haruan products that available in market are the wound healing cream, Haruan essence for food Haruan capsule and Haruan syrup. These prove the potential on Haruan to be commercialized and further research works are needed for the expansion of Haruan applications.

However, least works are done in the aspects of the solid form of Haruan products. Liquid or cream type products have shorter storage duration, lower efficiency and difficult to handle if compare to the solid particles. Hence, there is a need in producing Haruan in the form of powder or microparticles. In this research, the focus is in optimizing the microencapsulation of Haruan using the technique of spray drying.

The idea of transforming Haruan to solid particles form are due to the reasons that most of the biocomponents are more stable, longer shelf life and easy to handle if compare to liquid forms (Carneiro, Tonon, Grosso, and Hubinger, 2013). The details of the benefits of solid particles will be discussed in the next chapter of literature review. According to study of Carneiro et al., 2013, polyunsaturated fatty acids (PUFAs) which can be found in Haruan are prone to oxidation and protection of the PUFAs can be done with microencapsulation. Besides that, the biocompoents or nutrients in micron-size are easily absorbed by human body due to the increase in dimensional area and this indirectly can lead to the development of Haruan in pharmaceutical and food industries. Thus, high encapsulation efficiency is very crucial in order to produce good quality microparticles of Haruan. In order to achieve this, binders from the group of modified cellulose and gum with different concentrations are applied in the microencapsulation of Haruan's extract. Binders are able to protect the biocompoents from direct contact with the heat by forming a shield layer that enclosed the biocomponents. Despite of acting as protector, binders also believe can improve the physical characteristics of the particles produced in terms of morphology and size. In this study, microencapsulations of Haruan's extract with the few binders were conducted under different spray drying conditions. This is important to obtain the optimum spray drying conditions for the production of Haruan microparticles.

1.2 Research objectives

The following are the four main objectives for this research:

1. To extract proteins and antioxidants from Haruan (*Chana striatus*).
2. To produce microparticles of Haruan with selected binders using spray drying process.
3. To characterize the Haruan microparticles produced.
4. To optimize the process parameter of spray drying to produce Haruan microparticles.