UNIVERSITI PUTRA MALAYSIA

FUNCTIONAL AND PHYSICOCHEMICAL PROPERTIES, AND STORAGE STABILITY OF INSTANTIZED PURPLE SWEET POTATO (Ipomoea batatas L.) POWDER

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By

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Thesis submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

November 2015

Abstract of thesis presented to the senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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Chairperson : Assoc. Prof. Roselina Karim, PhD
Faculty : Food Science and Technology

Purple-flesh sweet potatoes (*Ipomoea batatas* L.) are commonly consumed as boiled tuber, traditional cakes and crispy snacks. Developing instantized purple sweet potato powder (IPSPP) from this tuber serves as a vehicle for adding value to this local commodity. Most of studies related to the production of powder from tubers focused only on the antioxidant and physical properties and not much research has been done on the resistant starch. Therefore, this study was carried out to evaluate the effects of processing methods and storage temperatures on the antioxidant, physical and resistant starch contents of IPSPP. IPSPP was produced using a double drum drier (set at 2 rpm and steam pressure of 3 bars). The first part of the research involved studying the effects of two preheating treatments which were boiling and steaming prior to drum drying on the antioxidant, physical and resistant starch properties of IPSPP. It was found that IPSPP pretreated by steaming process had significantly higher ($p \leq 0.05$) total anthocyanin (121.71 mg/100 g), moisture content (3.21% db), powder yield (243.02 g/kg) and resistant starch content (3.06 g/100 g) than boiling pretreatment. There was no significant difference ($p > 0.05$) between boiling and steaming in terms of antioxidant capacity (101.35 to 101.75 µmol TE/g and 134.35 to 134.40 µmol TE/g for DPPH and FRAP, respectively), physical properties such as water activity (0.49 to 0.50), color values, water solubility index (21.76 to 25.54) and non resistant starch content (51.68 to 60.94 g/100 g). The results showed that steaming of raw tuber is recommended as preheating treatment prior to drum drying. In the second part of the research, an attempt was made to increase the resistant starch content of IPSPP by pretreatment of the steamed-mashed tuber with pullulanase enzyme. Effects of the enzyme concentration and hydrolysis time on the resistant starch and total anthocyanin contents were evaluated. The optimum hydrolysis conditions for production of the maximum amount of resistant starch and total anthocyanin contents at 60ºC were 0.5% v/w pullulanase at 8 h of incubation period. Hydrolysis of purple sweet potato (PSP) puree with pullulanase enzymes significantly ($p \leq 0.05$) increased the resistant starch content (5.44 g/100 g), but concurrently reduced the antioxidant capacity of the IPSPP (5.20 µmol TE/g). The latter effect could probably due to long period of incubation and additional heating process required for enzyme inactivation that destroyed the

antioxidants in the IPSPP. The final part of the study focused on the storage stability of IPSPP at different storage temperatures. The total anthocyanin, DPPH radical scavenging activity, moisture and resistant starch contents of the samples were evaluated at every two month intervals for a duration of 12 months. The result showed that storage periods had significant effects ($p \leq 0.05$) on the total anthocyanin (185.32 and 197.61 mg/100 g at ambient and chilled storages, respectively), scavenging activity (74.16 and 77.48 µmol TE/g at ambient and chilled storages, respectively), moisture (6.35 and 8.10 % db at ambient and chilled storages, respectively) and resistant starch content (1.57 and 1.73 g/100 g at ambient and chilled storages, respectively) of IPSPP. The antioxidant, physical and resistant starch properties of IPSPP at 12 months of storage were compared with the control sample. The physicochemical properties of IPSPP changed remarkably after 12 months of storage at ambient (25±2ºC) condition. Sample stored at chilled storage (4±2ºC) had similar properties to the control sample. The most suitable storage temperature for IPSPP is at chilled condition (4±2ºC) because at this condition the antioxidant, physical and resistant starch properties product was stable for 12 months with significant increment in the total anthocyanin content. Based on these findings, it can be concluded that processing methods and storage conditions influence the antioxidant, physical and resistant starch contents of IPSPP.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

SIFAT FUNGSIAN DAN FIZIKOKIMIA, SERTA KESTABILAN PENYIMPANAN SERBUK SEGERA UBI KELEDEK UNGU (*Ipomoea batatas* L.)

Oleh

GITA ADDELIA NEVARA

November 2015

Pengerusi : Prof. Madya Roselina Karim, PhD
Fakulti : Sains dan Teknologi Makanan

Pada kebiasaannya, ubi keledek ungu (*Ipomoea batatas* L.) sering dinikmati sebagai ubi rebus, kuih tradisional dan snek rangup. Pembangunan serbuk segera ubi keledek ungu (IPSPP) dari ubi keledek ini berfungsi sebagai cara untuk menambah nilai kepada komoditi tempatan. Kebanyakan kajian yang berkaitan dengan penghasilan serbuk dari ubi memberi fokus hanya kepada sifat antioksidan dan sifat-sifat fizikal dan kajian terhadap kanji resistan adalah terhad. Oleh itu, kajian ini dijalankan untuk menilai kesan kaedah pemprosesan dan suhu penyimpanan keatas nilai antioksidan, sifat fizikal dan kandungan kanji resistan IPSPP. IPSPP dihasilkan menggunakan pengering drum ganda (diset pada 2 rpm dan tekanan stim 3 bar). Bahagian pertama kajian ini adalah untuk melihat kesan dua kaedah prapemanasan iaitu perebusan dan pengukusan sebelum proses pengeringan drum keatas sifat antioksidan, sifat fizikal dan kanji resistan IPSPP. IPSPP yang telah melalui proses praolahan pengukusan mempunyai antosianin total (121.71 mg/100 g), kandungan kelembapan (3.21% db), hasil serbuk (243.02 g/kg) dan kandungan kanji resistan (3.06 g/100 g) yang lebih tinggi dan signifikan ($p \leq 0.05$) berbanding yang melalui proses praolahan perebusan. Tiada perbezaan yang signifikan ($p > 0.05$) antara perebusan dan pengukusan dari segi kapasiti antioksidan iaitu DPPH (101.35 - 101.75 µmol TE/g) dan FRAP (134.35 - 134.40 µmol TE/g) dan sifat-sifat fizikal iaitu aktiviti air (0.49 - 0.50), nilai warna, indeks keterlarutan air (21.76 - 25.54) dan kandungan kanji bukan resistan (51.68 - 60.94 g/100 g. Keputusan kajian ini menunjukkan bahawa pengukusan ubi mentah adalah disarankan sebagai proses prapemanasan sebelum pengeringan drum. Dalam bahagian kedua kajian ini, usaha bagi meningkatkan kandungan kanji resistan IPSPP melalui praolahan ubi yang dikukus dan dilecek menggunakan enzim pullulanase telah dijalankan. Kesan kepekatan enzim dan tempoh hidrolisis keatas kanji resistan dan jumlah kandungan antosianin total telah dinilai. Keadaan hidrolisis optimum bagi penghasilan kandungan kanji resistan dan antosianin total yang maksimum pada 60ºC ialah dengan 0.5% v/w pullulanase selama 8 jam inkubasi. Hidrolisis puri ubi keledek ungu menggunakan enzim pullulanase telah meningkatkan kandungan kanji resistan secara signifikan (5.44 g/100 g), tetapi dalam masa yang sama telah mengurangkan kapasiti antioksidan IPSPP (5.20 µmol TE/g). Kesan kedua dapat mungkin disebabkan

oleh tempoh panjang inkubasi dan proses pemanasan tambahan yang diperlukan untuk pengnyahaktifan enzim yang memusnahkan antioksidan IPSPP. Bahagian akhir kajian ini pula memfokuskan kestabilan penyimpanan IPSPP pada suhu penyimpanan yang berbeza-beza. Jumlah antosianin total, aktiviti memerangkap radikal, kandungan lembapan dan kanji resistan sampel telah dinilai pada setiap dua bulan selama 12 bulan. Keputusan menunjukkan bahawa tempoh penyimpanan memberi kesan yang signifikan ($p \leq 0.05$) keatas jumlah antosianin total (185.32 dan 197.61 mg/100 g pada keadaan ambien dan dingin), aktiviti memerangkap radikal DPPH (74.16 dan 77.48 µmol TE/g pada keadaan ambien dan dingin), kandungan lembapan (6.35 dan 8.10 % db pada keadaan ambien dan dingin) dan kanji resistan (1.57 dan 1.73 g/100 g pada keadaan ambien dan dingin) IPSPP. Sifat antioksidan, fizikal dan kanji resistan IPSPP sepanjang 12 bulan penyimpanan telah dibandingkan dengan sampel kawalan. Sifat-sifat fizikokimia IPSPP berubah dengan ketara selepas 12 bulan pada keadaan ambien (25±2ºC). Sampel yang disimpan pada keadaan dingin (4±2ºC) mempunyai sifat yang sama dengan sampel kawalan. Suhu penyimpanan yang paling sesuai untuk IPSPP adalah pada keadaan dingin (4±2ºC) kerana pada keadaan ini sifat antioksidan, fizikal dan kanji resistan produk adalah stabil untuk 12 bulan dengan peningkatan ketara bagi kandungan antosianin total. Berdasarkan dapatan ini, dapat disimpulkan bahawa kaedah pemprosesan dan keadaan penyimpanan mempengaruhi kandungan antioksidan, fizikal dan kanji resistan IPSPP.

ACKNOWLEDGEMENTS

In the name of Allah, the Most Gracious and the Most Merciful. First of all, Alhamdulillah, I thank Allah for giving me His uncountable blessings in my life. Peace be upon the prophet Muhammad S.A.W. All praises to Allah for giving me the opportunity to finish my research and complete this thesis.

My sincere gratitude goes to my supervisor, Associate Professor Dr Roselina Karim, who has guided and supervised me in doing my postgraduate study productively. I also thankful to my co-supervisors, Associate Professor Dr Sharifah Kharidah Syed Muhammad and Professor Hasanah Mohammad Ghazali for their constructive suggestions throughout the research.

I am deeply grateful to all the labmates, staffs and students of Faculty of Food Science and Technology, Universiti Putra Malaysia, especially Sriizzati, Nasirah, Kak Farhiah, Safura, Zhafarina and Hamidah. I would not have been able to do my Master research effectively without their kind help and support. My acknowledgement also goes to Geran Putra IPS (Universiti Putra Malaysia) and LPDP (Ministry of Finance, Republic of Indonesia) for the financial support of this research and thesis.

My deepest gratefulness goes to my beloved husband, Rio Candra. Countless thanks for your prayers, guidance, love and trust. I am greatly indebted to my beloved parents, Mr. Pentaroza Azwar and Mrs. Kasmawati, and also my dearest brothers Lukman Nul Hakim and Habib Nabil Syathir for their endless prayers and love. My great appreciation to my parents-in law, Mr. Raziman and Mrs. Syamsiwasni, my brother and sisters-in law Mr. Cipto, Mrs. Mina and Ms. Nino for their supporting and understanding. Also not forgetting, sincere thanks to Mrs. Nofhayati’s family for their valuable assistance.

Many thanks go to my lovely friends in Indonesian Student Association (PPIUPM) for their support throughout these years, especially Riri, Putri, Murti and Indah. Last but not least, my sincere appreciation goes to all those who indirectly contributed to my postgraduate study. Sincerely, a million thanks for all of you.

I certify that a Thesis Examination Committee has met on 6 November 2015 to conduct the final examination of Gita Addelia Nevara on her thesis entitled "Functional and Physicochemical Properties, and Storage Stability of Instantized Purple Sweet Potato (*Ipomoea batatas* L.) Powder" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

Members of the Thesis Examination Committee were as follows:

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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a*	Redness (+) to greenness (-)
b*	Yellowness (+) and blue (-)
AF	Anthocyanin fraction
$AlCl_3$	Aluminium chloride
AMG	Amyloglucosidase
ANOVA	Analysis of variance
Aw	Water activity
$C_2H_4O_2$	Acetic acid glacial
CA	Caffeic acid
CE	Catechin equivalent
CQA	Caffeoylquinic acid
Cy	Cyanidin
Db	Dry basis
DPPH	2,2-diphenyl-1-picrylhydrazyl
ESI-MS	Electron spray ionization mass spectrometry
FAMA	Federal agricultural marketing authority
Fe	Ferrum
$Fe(III)(TPTZ)_2$	Ferric 2, 4, 6-tripyridyl-s-triazine
$FeCl_3.6H_2O$	Iron (III) chloride hexahydrate
FRAP	Ferric reducing ability power
fw	Fresh weight
GABA	γ -aminobutyric acid
GAE	Gallic acid equivalent
GOPOD	Glucose oxidase plus peroxidase
HCl	Hydrochloric acid
HPLC	High performance liquid chromatography
IC_{50}	Half maximum inhibitory concentration
IMS	Industrial methylated spirits
IPSPP	Instantized purple sweet potato powder
KCl	Potassium chloride
KOH	Potassium hydroxide
L*	Lightness
M	Molar
MC	Moisture content
MD	Maltodextrin
mg	Miligram
mL	Mililitre
mM	Milimolar
MS	Mass spectrometry
Na_2CO_3	Sodium carbonate
$NaNO_2$	Sodium nitrate
NaOH	Sodium hydroxide
nm	Nanometer
NRS	Non resistant starch
ORAC	Oxygen radical absorbance capacity
OSP	Orange sweet potatoes
Pn	Peonidin

PSP	Purple sweet potato
PSPLABD	Purple sweet potato lactic acid bacteria drink
PUN	Pullulanase unit novo
RDS	Rapidly digesting starch
rpm	Round per minute
RS	Resistant starch
RT	Room temperature
SD	Standard deviations
SDS	Slowly digesting starch
SEM	Scanning electron microscope
TAC	Total anthocyanin content
t-BHP	Tert-butyl hydroperoxide
TE	Trolox equivalent
TFC	Total flavonoid content
TPC	Total phenolic content
TPTZ	2, 4, 6-tripyridyl-s-triazine
TS	Total starch
Uv-Vis	Ultraviolet-visible
v/w	Volume per weight
WAC	Water absorption capacity
WSI	Water solubility index
µm	Micrometer
µM	Micromolar
ΔE	Total color difference

CHAPTER 1

INTRODUCTION

Purple sweet potato (*Ipomoea batatas* L.) has received considerable attention from researchers recently. It is due to the health benefits from their antioxidant content, particularly anthocyanins which give them strong purple color. Besides their great potential as natural food colorants, the anthocyanins of purple sweet potato (PSP) also have various nutraceutical properties such as anti-mutagenicity, scavenging activity, antihypertensive effect and anti-carcinogen activity (Oki *et al.*, 2002) which is found to be better than that of elderberry, red cabbage, purple corn and grape skin (Rumbaoa *et al.*, 2009).

Unfortunately, storage of fresh sweet potatoes generally requires controlled relative humidity (85-95%) (Padda and Picha, 2008; Mortleyl *et al.*, 1994) and temperature (13-15ºC) (Reesa *et al.*, 2003), which is not only energy consuming but also requires advanced and expensive equipments. In some countries, for example Malaysia and Indonesia, about 12.3 and 15.2 t/ha of sweet potatoes were produced (Ministry of Agriculture and Agro-based Industry Malaysia, 2012; Ministry of Agriculture Indonesia, 2014). However, as perishable tubers, sweet potatoes losses are usually caused by bruising, sprouting, rotting (bacteria and fungi) and senescence when they are stored in unsuitable conditions (FAO, 1983). In term of consumption, PSP could be directly consumed as boiled tubers, fried cakes or fried crisps. It can also be sliced and dried before converting it into flour or powder to avoid the abovementioned loss. Convertion of PSP tubers into PSP flour or powdered form will contribute to several advantages such as a longer shelf life, less space for storage and ready to be used.

There are several drying methods which can be used to produce the instantized PSP powder (IPSPP). Among these, the drum dryer is generally used for drying of commercial starchy food products with different feed flow rates. It is because this drying technique can be applied for samples with a wide range of viscosities, such as sample in the form of pastes, suspensions and solutions. Many heat sensitive products are also suitable to be drum dried because high temperature drying process is limited to a few minutes (Pua *et al.*, 2007). During the production of IPSPP, preheating treatments prior to drum drying such as boiling and steaming on the PSP tubers are necessary because PSP undergo browning which will affect the final quality of the products.

Instant or instantized powder in this study refers to a finely-ground powder that has gone through pregelatinization stage, a process that involves heating the flour with hot water or steam, then drying it out. It is formulated to dissolve rapidly in either hot or cold liquids, making it an ideal thickening agent in creating lump-free sauces and gravies (Bowman, 2014).

Resistant starches (RS) are starches which are not digested in the human small intestine and were reported to have desired combination of functional and physiological properties compared to natural fiber (Hódsági, 2011). RS have been reported to be beneficial for health in preventing colonic cancer and reduction of gall stone formation, improving lipid and glucose metabolism, reducing the risk of colorectal cancer and other gastrointestinal disorders as well as diabetes mellitus type-2, coronary and heart diseases (Hódsági, 2011; Nugent, 2005). IPSPP is low in RS, but it is high in anthocyanin. If the RS content of IPSPP which is high in anthocyanin can be increased, a new product with improved functional properties can be developed from PSP. Several methods can be employed to increase the RS content of IPSPP. An enzymatic treatment using debranching enzyme (in this case, pullulanase enzyme) could be one of the appropriate methods to prevent or minimize the loss of anthocyanin during heating - cooling processes. The usage of pullulanase had been reported to increase the RS content in food starch (Leong *et al.*, 2007; Lehmann *et al.*, 2003). However, to date, no study has been reported on the production of IPSPP that is high in both the anthocyanin and RS contents. The production of IPSPP with antioxidant and RS properties will offer more health benefit to the consumers.

Moreover, the intense purple color, flavor, natural sweetness and nutrients as well as granulation property of PSP powder (i.e water absorption capacity and water solubility) made it suitable to be used as an ingredient with an instantized property or an instant powder, a thickener in soup, as gravy, in bakery products and fabricated snacks (Ahmed *et al.*, 2010a). Thus, IPSPP is highly potential to be used as one of the functional ingredients in the food industries.

This study was carried with the following objectives:

(i) To determine the effects of preheating treatments (boiling and steaming) on the antioxidant, physical and resistant starch properties of IPSPP,

(ii) To determine the effects of pullulanase pretreatment on the resistant starch and total anthocyanin contents, and scavenging activity of IPSPP, and

(iii) To evaluate the stability of IPSPP in terms of antioxidant, physical and resistant starch properties during storage at different temperatures.

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