UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF ANTIOXIDANT-RICH PURPLE SWEET POTATO (Ipomoea batatas L.)-BASED EXTRUDED BREAKFAST CEREAL

SRI SAMPATH JANAKA SENEVIRATHNA

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By

SRI SAMPATH JANAKA SENEVIRATHNA

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

March 2022

DEDICATION

This thesis is dedicated to my beloved wife, son, father, mother and brother for their unconditional love, engorgement and support.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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

Ready-to-eat extruded breakfast cereal is extremely popular among consumers because it is easy and very convenient to prepare, but is often loaded with sugars and unhealthy food additives. The increasing demand of consumers toward functional foods triggers the development of ready to eat breakfast cereals rich in antioxidant. Purple sweet potato powder (PSPP) which is rich in anthocyanins and other polyphenols, provides excellent antioxidant and other biological activities with potential health benefits to the consumers. Therefore, the main objective of this study was to develop an antioxidant-rich extruded puffed breakfast cereal from PSPP as the main ingredient. The anthocyanin content and colour of purple sweet potato are influenced by drying methods and processing conditions. In the first part of this study, the effects of citric acid (CA) concentration, steam pressure (SP) and drum rotation speed (DS) on the physicochemical and functional properties of drum-dried PSPP were investigated by response surface methodology (RSM). The results indicated that the moisture content (MC) and water activity (a_w) significantly ($p<0.05$) decreased with increasing SP and reduced DS. Addition of CA significantly ($p<0.05$) affected the colour and total anthocyanin content (TAC) of PSPP. High SP and low DS negatively influenced the antioxidant properties of PSPP, with the optimal physicochemical and functional properties of drum dried PSPP achieved at 0.59% CA, 499.8 kPa SP and 3.0 rpm DS. Twelve anthocyanins were identified by HPLC-MS/MS; and it was observed that cyanidine based anthocyanins were dominant in PSPP. The second part of the research evaluated the effect of formulation on the physiochemical, textural and functional properties of the PSP-based extruded puffed breakfast cereals prepared from a mixture of PSPP (30.0%-70.0%), wholegrain red rice flour (WRRF) (15.0%-50.0%) and (cornflour) CF (15.0%-50.0%) using a single screw extruder via a D-optimal mixture design approach. The findings demonstrated that PSPP, WRRF and CF had a pronounced effect on the extrudates' textural and functional properties. The PSPP indicated a most significant positive impact on antioxidant capacity, whereas the CF contributed towards a better expansion of the final product. The optimum formulation of breakfast cereal with an acceptable textural properties and highest antioxidant properties was produced using 55.0% PSPP, 15.0% WRRF and 30.0% CF. In the third part of this research, the RSM was used to optimise the extrusion processing condition for the development of breakfast cereals. The independent variables studied include barrel temperature, screw speed and feed moisture in the range of 140.0°C-180.0°C, 100.0 rpm - 140.0 rpm and 13.0%-18.0%, respectively. All the responses were significantly affected by the process variables. The linear terms of barrel temperature and feed moisture content were found to be the main significant ($p<0.05$) factors affecting the product's functional and physicochemical properties. The expansion property of extrudate was significantly ($p<0.001$) increased at low temperature, high screw speed and low feed moisture. The recommended optimum extrusion conditions of barrel temperature, screw speed and feed moisture content were at 157.0°C, 126.0 rpm and 13.0%, respectively. Under these optimum conditions, significantly high retention (75.0%) of anthocyanin content was detected in the final extruded product. Furthermore, the scanning electron micrographs depicted that the optimised breakfast cereals had a better cell structure with smoother and thin cell walls than the non-optimised samples. The final part of this study investigated the effect of packaging on the physicochemical and functional properties of antioxidant-rich PSP-based breakfast cereal under accelerated conditions (38.0±1.0°C, 90.0% relative humidity) packaged in metalised (MET) and polypropylene (PP) pouches for 12 weeks. The magnitude of quality changes of packaged PSP breakfast cereals was monitored throughout the storage period. The colour change, TAC reduction, MC and a_w gain of breakfast cereals followed zero order kinetics during storage, and the half-life of anthocyanin was recorded as 24.63 and 10.13 weeks for MET and PP, respectively. The kinetic constant for all properties studied was higher for the product packed in PP than MET pouches. It was observed that moisture migration is the limiting factor of the shelf-life of the PSP breakfast cereals. Sensory analysis results showed that the PSP extruded product had an average overall acceptability score ranging from 7.15 to 8.29 indicating the panellists like the product moderately to very much. In conclusion, an extruded puffed breakfast cereals with attractive natural purplish-red colour and acceptable functional properties was successfully developed in this study.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

PEMBANGUNAN BIJIRIN SARAPAN YANG KAYA DENGAN ANTIOKSIDAN BERASASKAN UBI KELEDEK UNGU (*Ipomoea batatas* L.)

Oleh

SRI SAMPATH JANAKA SENEVIRATHNA

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Pengerusi : Prof. Madya Roselina Karim,PhD
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Bijirin sarapan tersemperit sedia untuk dimakan amat popular di kalangan pengguna kerana ia sangat konvenien dan senang disediakan, tetapi selalunya sarat dengan gula dan bahan tambahan makanan yang tidak sihat. Peningkatan permintaan pengguna terhadap makanan fungsian mencetuskan pembangunan bijirin sarapan sedia untuk dimakan yang kaya dengan antioksidan. Serbuk ubi keledek ungu (PSPP) yang mempunyai kandungan antosianin dan polifenol lain yang tinggi membekalkan aktiviti antioksidan dan aktiviti biologi lain yang sangat baik yang berpotensi memberi manfaat kesihatan kepada pengguna. Oleh itu, objektif utama kajian ini adalah untuk membangunkan bijirin sarapan tersemperit mengembung yang kaya dengan antioksidan menggunakan PSPP sebagai bahan utama. Kandungan antosianin dan warna ungu ubi keledek dipengaruhi oleh kaedah pengeringan dan pemprosesan. Pada bahagian pertama kajian ini, kesan kepekatan asid sitrik (CA), tekanan wap (SP) dan kelajuan putaran (DS) terhadap sifat fizikokimia dan kefungsian PSPP yang dikeringkan secara pengeringan dram dikaji dengan menggunakan kaedah respons permukaan (RSM). Hasil kajian menunjukkan bahawa kandungan kelembapan (MC) dan keaktifan air (aw) ($p < 0,05$) menurun secara signifikan dengan peningkatan SP dan pengurangan DS. CA mempunyai kesan yang signifikan ($p < 0.05$) terhadap warna dan kandungan antosianin total (TAC) PSPP. Nilai SP yang tinggi dan DS yang rendah memberi kesan negatif terhadap sifat antioksidan PSPP, dengan pencapaian sifat fizikokimia dan kefungsian optimum pada 0.59% CA, 499.8 kPa SP dan 3.0 rpm. Dua belas jenis antosianin dikenal pasti melalui kaedah HPLC-MS/MS dan antosianin berasaskan sianidin adalah dominan dalam PSPP. Bahagian kedua penyelidikan menilai kesan rumusan terhadap sifat fizikokimia, tekstur dan kefungsian bijirin sarapan tersemperit berasaskan PSP yang disediakan dari campuran yang terdiri dari PSPP (30-70%), WRRF (15-50%) dan CF (15-50%) menggunakan penyemperit skru tunggal melalui pendekatan reka bentuk campuran D-optimum. Hasil kajian menunjukkan bahawa PSPP, WRRF dan CF menunjukkan suatu kesan jelas terhadap tekstur dan sifat kefungsian produk tersemperit. PSPP menunjukkan kesan yang sangat signifikan pada

kapasiti antioksidan, manakala CF memberikan kesan pengembangan produk akhir yang lebih baik. Rumusan optimum bijirin sarapan dengan tekstur yang elok dengan kandungan antioksidan yang tinggi boleh diperbuat menggunakan 55.0% PSPP, 15.0% WRRF dan 30.0% CF. Pada bahagian ketiga penyelidikan ini, RSM digunakan untuk mengoptimumkan proses penyemperitan untuk pembangunan bijirin sarapan. Di antara pembolehubah tak bersandar yang dikaji adalah laras suhu penyemperitan (140.0 -180.0 °C), kelajuan skru (100.0 -140.0 rpm) dan kelembapan serbuk yang dimasukkan (13.0 -18.0%). Ketiga-tiga respons yang dikaji dipengaruhi oleh pembolehubah proses secara signifikan. Terma linear bagi laras suhu penyemperitan dan kandungan kelembapan serbuk adalah faktor paling signifikan ($p<0.05$) yang mempengaruhi sifat kefungsian dan fizikokimia produk. Pengembangan produk penyemperitan meningkat dengan ketara ($p<0.001$) pada suhu yang rendah, kelajuan skru yang tinggi dan kelembapan serbuk yang rendah. Keadaan penyemperitan optimum yang disyorkan adalah pada laras suhu penyempritan, kelajuan skru dan kandungan kelembapan serbuk masing-masing 157.0 °C, 126.0 rpm dan 13.0%. Di bawah keadaan optimum ini, kandungan antosianin yang tinggi (75.0%) dikesan pada produk akhir penyemperitan. Selain itu, pengimbasan elektron mikrograf menunjukkan bahawa bijirin sarapan yang dioptimumkan mempunyai struktur sel yang lebih baik dengan dinding sel yang lebih licin dan lebih nipis berbanding dengan sampel yang tidak dioptimumkan. Bahagian terakhir penyelidikan mengkaji kesan pembungkusan terhadap sifat fizikokimia dan kefungsian bijirin sarapan berasaskan PSP yang kaya dengan antioksidan pada keadaan penyimpanan dipercepat (38 ± 1 °C, kelembapan relatif 90%) yang dibungkus dalam pembungkus logam (MET) dan polipropilena (PP) selama 12 minggu. Perubahan kualiti bijirin sarapan PSP yang dibungkus dipantau sepanjang tempoh penyimpanan. Perubahan warna, TAC, MC dan aw bijirin sarapan menunjukkan kinetik tertib sifar semasa penyimpanan dan separuh hayat antosianin dicatatkan masing-masing adalah 24.63 dan 10.13 minggu untuk MET dan PP. Pemalar kinetik untuk semua sifat yang dikaji adalah lebih tinggi untuk produk yang dibungkus dalam PP berbanding MET. Pemindahan kelembapan merupakan faktor terhad bagi jangka hayat produk bijirin sarapan PSP. Keputusan analisis sensori menunjukkan bahawa produk PSP mendapat skor purata kebolehterimaan dalam julat antara 7.15 ke 8.29, yang mengambarkan produk tersebut adalah disukai secara sederhana hingga sangat disukai oleh ahli panel. Kesimpulannya, bijirin sarapan tersemperit dengan warna ungu kemerahan semula jadi yang menarik serta sifat kefungsian yang lebih baik berjaya dihasilkan dalam kajian ini.

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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 Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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a*	Redness (+) to greenness (-)
$AlCl_3$	Aluminium chloride
ANOVA	Analysis of variance
a_w	Water activity
b*	Yellowness (+) and blue (-)
BT	Barrel temperature
CA	Citric acid
CE	Catechin equivalent
CF	Cornflour
CQA	Caffeoylquinic acid
Cy	Cyanidin
db	Dry basis
DPPH	2,2-diphenyl-1-picrylhydrazyl
DS	Drum speed
ER	Expansion ratio
ESI-MS	Electron spray ionisation mass spectrometry
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
GAE	Gallic acid equivalent

g	Gramme
h	Hour
HCl	Hydrochloric acid
HPLC	High performance liquid chromatography
KCl	Potassium chloride
kg	Kilogram
L	Length
L*	Lightness
M	Molar
MC	Moisture content
mg	Miligram
mL	Mililitre
mM	Milimolar
MS	Mass spectrometry
Na_2CO_3	Sodium carbonate
$NaNO_2$	Sodium nitrate
NaOH	Sodium hydroxide
Pn	Peonidin
PSP	Purple sweet potato
rpm	Round per minute
RH	Relative humidity
RT	Room temperature
SD	Standard deviations
SEM	Scanning electron microscope

SP	Steam pressure
SS	Screw speed
TAC	Total anthocyanin content
TE	Trolox equivalent
TFC	Total flavonoid content
TPC	Total phenolic content
TPTZ	2, 4, 6-tripyridyl-s-triazine
Uv-Vis	Ultraviolet-visible
v/w	Volume per weight
WAC	Water absorption capacity
WRRF	Whole red rice flour
WSI	Water solubility index
µg	Microgram
µL	Micro Litter
µm	Micrometer
µM	Micromolar
ΔE	Total colour difference

CHAPTER 1

INTRODUCTION

Breakfast cereal is a ready-to-eat food commonly consumed for breakfast. Typically, it is made from cereal grains by extrusion, thus is high in starch with few functional properties. Currently, there is a demand for a healthy breakfast cereal with added physiologic and therapeutic benefits. Purple sweet potato powder (PSPP) is rich in anthocyanins and other polyphenols, with good antioxidant and other biological activities, hence contributes to potential health benefits to the consumers. Gras, Nemetz, Carle, and Schweiggert (2017) stated that the anthocyanin content of purple sweet potato (PSP) might vary depending on the variety, ranging from 558 to 2477 mg/100 g dry weight (DW) for freeze-dried PSP from different provenances. Furthermore, Nevara, Yea, Karim, Muhammad, and Mohd Ghazali (2019) discovered that the anthocyanin content of drum-dried PSP powder varied from 83.72 to 121.71 mg/100 g depending on pre-treatment before drum drying. Therefore, breakfast cereal made from PSPP will improve the product's functional qualities.

The annual global production of sweet potato is more than 90 million metric tonnes (Alam, 2021); however, Waramboi, Gidley, and Sopade (2014) reported that postharvest losses of sweet potatoes can reach 50% due to insufficient packaging, handling, storage, and transportation, leading to broken roots spoilage. The bulky and highly perishable nature of the sweet potato often results in a lower market, hence, processing is used to increase the value, availability, and storage stability of perishable products like sweet potato (Grabowski, Truong, & Daubert, 2008; Siddiq, Kelkar, Harte, Dolan, & Nyombaire, 2013). Additionally, farmers often face difficulties in selling small and deformed tubers because of the low demand and rejection by most supermarkets, with relatively low sweet potato consumption in developed countries.

Drying is a traditional food preservation method used to extend the shelf life of foods like fruits and vegetables. Among all the different drying methods, drum drying is one of the most energy-efficient and commercially feasible techniques commonly used to dehydrate purees and high viscous slurries during the manufacture of powders (Caparino et al., 2012; Pua et al., 2010). Extrusion is a high thermal rapid processing technology that is extensively used to produce different types of foods such as snacks, pasta, baby foods, and breakfast cereals (Bisharat, Oikonomopoulou, Panagiotou, Krokida, & Maroulis, 2013; Sue Shan, Sulaiman, Sanny, & Nur Hanani, 2015). Therefore, the processing is an excellent alternative to value add and create demand for these less valuable and bulky sweet potato tubers.

The development of an antioxidant-rich breakfast cereal from PSPP may open a new market opportunity to cater for the high demand generated by western and developed countries for healthy functional food choices. In recent years, natural food colouring or pigments are preferred by consumers because the usage of synthetic pigments in food products are often linked to harmful health effects. Anthocyanins are used as food colourants instead of artificial pigments and natural insect carmine because of their safety. However, the susceptibility of anthocyanins to different processing and preservation conditions affects the colour stability, hence it becomes a constraint to the industrialisation of anthocyanin containing foods (Aliaño-González et al., 2020; Cortez, Luna-Vital, Margulis, & Gonzalez de Mejia, 2017; Gras et al., 2017; He, Li, Lv, & He, 2015; Jiang et al., 2019; Lafarga et al., 2019; Li et al., 2013; Jing Li, Song, Dong, & Zhao, 2014; Mu, Sun, Zhang, & Wang, 2017). Therefore, purple sweet potatoes have the potential to be used as the raw material for production of healthy purple coloured antioxidant-rich breakfast cereals, but the anthocyanin pigments need to be preserved and stabilised.

Thus, the usage of food-grade phenolic plant extracts, various phenolic acids and different types of acids to improve the stability of anthocyanin extract have been previously reported (Chen et al., 2019; Gras et al., 2017; Li et al., 2013; Jing Li et al., 2014). Li, Walker, and Faubion (2011) proposed that citric acid can be used to retain the colour and maximum level of anthocyanin content of blue corn oven-baked cookies. In contrast, the use of citric acid to improve the thermal stability of drum dried PSPP anthocyanin has only been scarcely studied. On the other hand, incorporating high levels of PSPP and whole red rice flour could gain potential health benefits; however, incorporation of high fibre raw materials in an expanded extruded product may lead to loss of textural properties. This may lead to deteriorating the primary quality attributes of the extruded breakfast cereals (Chassagne-Berces et al., 2011). Furthermore, extrusion process parameters significantly affect on physicochemical and phytochemical properties of extrudates (Samyor, Deka, & Das, 2018). Therefore, optimisation of formulation and extrusion process parameters is of utmost importance to obtain the final product's better functional and textural properties.

Therefore, the specific objectives of this study were as follows:

(i) To assess the effects of drum drying parameters and citric acid level on anthocyanin stability of drum-dried purple sweet potato powder;

(ii) To study the effects of ingredients in the formulation on the physicochemical, functional and textual properties and to develop an acceptable formulation for antioxidant-rich breakfast cereal containing purple sweet potato powder;

(iii) To investigate the effects of extrusion process parameters on physicochemical, functional and textual properties and to optimised the barrel temperature, screw speed and feed moisture of the developed antioxidant-rich purple sweet potato-based breakfast cereal;

(iv) To evaluate the effect of packaging on the physicochemical, functional, and textural properties of breakfast cereal under accelerated conditions (90±1% relative humidity and 38±1°C).

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