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CHANGES IN PHYSICOCHEMICAL PROPERTIES AND FLAVOR PROFILES OF ROASTED TROPICAL ALMOND NUTS (Terminalia catappa L.)

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By

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

CHANGES IN PHYSICOCHEMICAL PROPERTIES AND FLAVOR PROFILES OF ROASTED TROPICAL ALMOND NUTS (*Terminalia catappa* L.)

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October 2015

Chairman: Associate Professor Lasekan Olusegun Olaniyi, PhD
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T. catappa is a tropical almond nut which belongs to Combretaceae family and is normally called Indian almond, sea almond, tropical almond, wild almond and etc. In some countries such as Malaysia, Jamaica and Nigeria, this tree is being used for shade and ornament while the edible nuts have been used as snacks or tidbits among the children. Roasting is an important process for nuts and oilseeds because proper roasting is crucial to flavor, color and texture development in the roasted product. Results revealed that moisture and protein content decreased while crude fat content of the nuts increased after roasting process. Besides, decreament of reducing sugar and amino acid content indicated the presence of Maillard reaction in the roasted nuts. This non-enzymatic browning reaction is important in determining consumer's acceptability and preferences in nuts. Optimization process revealed that the best roasting conditions for the nuts were of roasting temperature 174.5 ˚C, roasting time 29.9 min and pH 6.08. Color L value decreased from 51.88 to 45.02 whereas color a and b values increased from 3.33 to 3.41 and 8.40 to 9.38, respectively, throughout the roasting process. Meanwhile, browning index increased and fracturability decreased as roasting temperature, roasting time and pH increased. By using gas chromatography-mass spectrometry (GCMS) and gas chromatography olfactometry (GCO) associated with aroma extract dilution analysis (AEDA), flavor compounds that had flavor dilution (FD) factors equal or greater than 16 were selected as the potent odorants in the almond nuts. 12 and 25 potent odorants were identified in the raw and optimized roasted almond nuts, respectively. In raw nuts, the green, sweet and fruity odour impressions were correlated to hexanal, (E)-2-hexenal, hexanol, 2-hexenol, nonanal, ɣ-dodecalactone and ʃ-dodecalactone. On the other hand, dimethylthiazole, octanal, acetylthiazoline, dihydromethylcyclopentapyrazine, (E,E)-2,4-decadienal, decanoic acid, hexadecanone and (E,E)-farnesyl acetate were responsible for the fatty, oily and nutty odour impressions. From the quantification analysis, ɣ-dodecalactone and (E,E)-2,4-decadienal were found to have the highest concentration in raw and roasted nuts respectively. Besides, raw and roasted tropical almond nuts of different roasting levels

were analyzed using fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) in order to determine the changes of functional groups and surface morphology of the nuts. FTIR results showed that an increase in temperature did not produce new carbonyl compounds; it however led to higher percentage of transmittance of the carbonyl compounds. On the other hand, SEM results revealed that the nut surface changed from smooth to rough when the nuts were roasted at high temperature. In recent years, there have been limited literatures on the color/flavor changes in roasted *T. catappa*. Moreover, it is an unexploited nut that had been overlooked and since it has great potential in the food industry, the changes in physicochemical properties as the flavor precursors and flavor profiling of roasted nuts were crucial. Therefore, through this study, we could understand more of the browning changes and also its control in the nuts producing industry.

Abstrak tesis yang dikemukaan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Sarjana Sains

PERUBAHAN DALAM SIFAT FIZIKOKIMIA DAN PROFIL PERISA UNTUK KACANG BADAM TROPIKA PANGGANG (*Terminalia catappa* L.)

Oleh

NG SIEW SEE

Oktober 2015

Pengerusi: Profesor Madya Lasekan Olusegun Olaniyi, PhD
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Terminalia catappa ialah kacang badam tropika yang tergolong dalam keluarga Combretaceae dan biasanya dipanggil badam India, badam laut, badam tropika, badam liar dan lain-lain lagi. Dalam sesetengah negara seperti Malaysia, Jamaica dan Nigeria, pokok ini digunakan untuk lindungan dan hiasan manakala kacang yang boleh dimakan telah digunakan sebagai makanan ringan di kalangan kanak-kanak. Pemanggangan merupakan satu proses yang penting untuk kacang dan bijirin kerana pemanggangan yang bersesuaian adalah penting dalam pembangunan rasa, warna dan tekstur bagi produk panggang. Keputusan menunjukkan bahawa kandungan air dan protein berkurangan manakala kandungan lemak kacang meningkat selepas proses pemanggangan. Selain itu, pengurangan gula menurun dan asid amino telah menunjukkan kehadiran tindak balas Maillard dalam kacang panggang. Tindak balas pemerangan tanpa kehadiran enzim tersebut adalah penting dalam menentukan penerimaan dan keutamaan pengguna dalam pemilihan kacang. Proses optimum mendedahkan bahawa keadaan pemanggangan terbaik untuk kacang adalah pada suhu pemanggangan 174.5 ˚C, masa pemanggangan sebanyak 29.9 min dan pH 6.08. Nilai warna L menurun daripada 51.88 kepada 45.02 manakala nilai warna a dan b meningkat daripada 3.33 kepada 3.41 dan daripada 8.40 kepada 9.38 masing-masing sepanjang proses pemanggangan. Sementara itu, indeks pemerangan meningkat dan frakturabiliti menurun apabila suhu pemanggangan, masa pemanggangan dan pH meningkat. Dengan menggunakan gas kromatografi-spektrometri jisim (GCMS) dan kromatografi gas olfaktometri (GCO) bersama-sama dengan analisis pencairan ekstrak aroma (AEDA), kompaun yang mempunyai faktor pencairan (FD) yang sama atau melebihi 16 telah dipilih sebagai penyumbang perisa utama dalam kacang badam. 12 dan 25 kompaun utama telah didapati dalam kacang badam sebelum dan selepas proses pemanggangan masing-masing. Untuk kacang badam sebelum proses pemanggangan, 12 kompaun utama yang dikenalpasti adalah termasuk lima alkohol, tiga aldehida, dua keton, satu asid karboksilik dan satu kompaun sulfur; manakala untuk kacang badam tropika yang telah dioptimumkan, terdapat 25 kompaun utama yang terdiri daripada enam alkohol, lima keton, empat aldehida, tiga ester, dua asid karboksilik, dua kompaun sulfur, satu alkena dan satu pirazin telah didapati. Dalam kacang badam tropika sebelum pemanggangan, bau kehijauan, manis dan buah telah dikaitkan dengan

kompaun heksanal, (E)-2-heksenal, heksanol, 2-heksenol, nonanal, ɣ-dodekalakton dan ʃ-dodekalakton. Sementara itu, kompaun dimetiltiazol, oktanal, asetiltiazolin, dihidrometilsiklopentapirazin, (E,E)-2,4-dekadienal, asid dekanoik, heksadekanon dan (E,E)-asetat farnesil telah memainkan peranan dalm bau lemak, berminyak dan kacang. Daripada analisis kuantifikasi, ɣ-dodekalakton dan (E,E)-2,4-dekadienal didapati mempunyai kepekatan tertinggi dalam kacang sebelum dan selepas proses pemanggangan masing-masing. Selain itu, fourier transform infra-merah spektroskopi (FTIR) dan mikroskop imbasan elektron (SEM) telah digunakan untuk menentukan perubahan dari segi kumpulan berfungsi dan morfologi permukaan kacang sebelum dan selepas proses pemanggangan. Keputusan FTIR menunjukkan bahawa peningkatan suhu tidak menghasilkan sebarang kompaun karbonil baru, namun begitu perbandingan dapat dikenalpastikan dari segi kepekatan kompaun kompaun karbonil. Sementara itu, SEM mendedahkan bahawa permukaan kacang telah berubah daripada permukaan licin kepada lebih kasar apabila kacang badam tropika dipanggang pada suhu yang tinggi. Kebelakangan ini, kajian pada perubahan warna dan perisa untuk kacang badam ini adalah amat terhad. Tambahan pula, pokok ketapang ialah satu kacang yang telah terabai dan belum dieksploitasi. Memandangkan kacang ini adalah amat berpotensi dalam industri makanan, perubahan dalam sifat-sifat fizikokimia sebagai prekursor perisa dan profil perisa kacang panggang adalah amat penting. Oleh itu, melalui kajian ini, kita dapat memahami perubahan pemerangan dan juga kawalannya dalam industri penghasilan kacang secara lebih lanjut.

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I certify that a Thesis Examination Committee has met on 27 October 2015 to conduct the final examination of Ng Siew See on her thesis entitled "Changes in Physicochemical Properties and Flavor Profiles of Roasted Tropical Almond Nuts (*Terminalia catappa* L.)" 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.

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

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I hereby confirm that:

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This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
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LIST OF ABBREVIATIONS

AABA	Alpha-aminobutyric acid
AAS	Amino acid standard
AEDA	Aroma extract dilution analysis
ANOVA	Analysis of variance
AOAC	Association of Official Analytical Chemists
AOCS	American Oil Chemists' Society
BI	Attenuated total reflectance
ATR	Browning index
cm	Centimeter
℃	Celsius
CCD	Central composite design
CHD	Cardiovascular heart disease
DTGS	Deuterated Triglycine Sulphate
EDX	Energy Dispersive X-ray
FAME	Fatty acid methyl esters
FAO	Food and Agriculture Organization
FD	Dilution factor
FID	Flame Ionization Detector
FTIR	Fourier Transform Infrared Spectroscopy
g	Gram
g/s	Gram per second
GCMS	Gas Chromatography Mass Spectrometry
GCO	Gas Chromatography Olfactometry
h	Hour
HCl	Hydrochloric acid

HPLC	High Performance Liquid Chromatography
kg	Kilogram
kcal	Kilocalorie
kV	Kilovolt
m	Meter
min	Minute
mL	Mililitre
mL/min	Mililitre per minute
mm	Milimeter
µg	Microgram
µL	Microlitre
µm	Micrometer
MS	Mass Spectrometry
N	Nitrogen
NaCl	Sodium chloride
NaOH	Sodium hydroxide
NO	Nitric oxide
NIF	Nasal impact frequency
%	Percent
%T	Percentage of transmittance
p	Probability
PITC	Phenylisothiocyanate
R^2	Regression
RSM	Response surface methodology
s	Second
SEM	Scanning Electron Microscopy
T. catappa	*Terminalia catappa*

3D	Three-dimensional
TDF	Total Dietary Fibre
v/v	Concentration volume per volume
w/v	Mass per volume
WAO	World Allergy Organizaiton
WHO	World Health Organization

CHAPTER 1

INTRODUCTION

1.1 Background

T. catappa is a tropical almond nut with fibrous husk and edible almond-like kernel inside the husk (Ahmad, 2010). It belongs to Combretacea family and is commonly called tropical almond, wild almond, sea almond, India almond and etc. It is normally planted on well-drained and sandy soil in coastal environment. The whole *T. catappa* tree has many uses such as being planted for ornamental or shade purposes along the road, the leaves are used as folk medicines to prevent hepatitis; and the almond kernel is eaten raw by Nigerian children and as a value added snack incorporated in the main dishes (Biego et al., 2012).

Physicochemical properties of the raw tropical almond nuts were determined as the key flavor precursors. Proximate analysis showed that the nuts contained high quantity of protein and lipid (Oliveira et al., 2000; Ezeokonkwo, 2007). In this study, the tropical almond nuts were roasted to develop color and flavor to enhance the sensorial properties and the overall acceptability of the nuts. The independent variables applied were roasting temperature (100-180 ℃), roasting time (0-30 min) and pH (5-7) of the nuts.

In order to obtain the most desirable roasting conditions, response surface technology (RSM) was applied to create an experimental design of different combinations of independent variables to determine the effects of the variables on the responses which were color L, a, b, browning index (BI) and fracturability. Many studies had applied RSM technique on their studies such as acrylamide development in *Terminalia catappa* nuts (Lasekan and Abbas, 2011); physicochemical characteristics of torch ginger (Juhari et al., 2012); and quality characteristics of potato (Eren and Kaymak-Ertekin, 2007).

Besides optimization using RSM, fourier transform infrared (FTIR)-attenuated total reflection (ATR) spectroscopy and scanning electron spectroscopy (SEM) were now being used to obtain the flavor prints and monitor structural properties. FTIR has been used to discriminate between different types of coffee beans and monitor the degree of roasting. During roasting, flavor generated resulted from Maillard reaction, a reaction between amino acid and reducing sugar of nuts. Flavor, the combination of aroma and taste, is the major factors in consumer preference.

Gas chromatography olfactometry (GCO) assisted with aroma extract dilution analysis (AEDA) were used to identify and quantify the aroma compounds in both raw and optimized roasted tropical almond nuts. To screen those potent aroma compounds that major contribute to the flavor of roasted nuts, flavor dilution (FD) factor with higher value were the key odorants. Investigation on aroma of different nuts were carried out by several studies using GCO-AEDA technique for examples in roasted pistachio nut (Aceña et al., 2011), peanuts (Chetschik et al., 2008) and hazelnuts (Burdack-Freitag and Schieberle, 2010).

Due to the increasing nuts consumption (FAO, 2012), the underutilization of *T. catappa* makes it a potential and cheaper alternative plant protein for livestock and

human consumption. Thus, the observations of the physicochemical properties changes and flavor development during roasting were crucial to obtain the most desirable nuts. The results on color and flavor development from this study could help us understand more of the browning changes and also its control in the nuts industry producing.

Therefore, the objectives of this study were:

1. To determine the physicochemical properties of the raw tropical almond nuts (*T. catappa* L.), fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) analysis of the roasted tropical almond nuts;
2. To optimize the roasting conditions on color ant texture development of the tropical almond nuts (*T. catappa* L.) using response surface methodology (RSM); and
3. To profile the potent odorants in the raw and roasted tropical almond nuts (*T. catappa*) using gas chromatography olfactometry (GCO).

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