



**UNIVERSITI PUTRA MALAYSIA**

***RETENTION AND STORAGE STABILITY OF VITAMINS A AND C IN  
EXTRUDED NATIVE AND PREGELATINIZED STARCHES***

***SITI FARHIAH BINTI ABDUL MANAN***

**FSTM 2015 21**



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By

**SITI FARHIAH BINTI ABDUL MANAN**

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

**May 2015**

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

## **RETENTION AND STORAGE STABILITY OF VITAMINS A AND C IN EXTRUDED NATIVE AND PREGELATINIZED STARCHES**

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**May 2015**

**Chair : Assoc. Prof. Sharifah Kharidah Syed Muhammad, PhD**  
**Faculty : Food Science and Technology**

A study was carried out to determine the retention of vitamins A and C in native and pregelatinized starches extruded under fixed conditions in the making of extruded dried noodles. Two percent vitamin A or C was mixed with starch and moisture content of the mixture was adjusted to 20% before extrusion. The extrusion conditions were temperatures of 80 °C for zone 1 and 100 °C for zones 2, 3 and 4 of the barrel, 30 rpm of feeder speed, and 60 rpm of screw speed. Native starches used were waxy corn (99.1% amylopectin), regular corn (26.2% amylose), high amylose corn (70.0% amylose), waxy potato (99.1% amylopectin), regular potato (30.1% amylose), waxy or glutinous rice (92.6% amylopectin), regular rice (19.4% amylose), tapioca (25.9% amylose), sweet potato (29.9% amylose) and sago (30.4% amylose) starches. The pregelatinized starches included pregelatinized waxy corn, regular corn, high amylose corn, waxy potato, regular potato, waxy or glutinous rice, regular rice, tapioca, sweet potato and sago starches. The retention of vitamins A and C, after extrusion, was analysed by reversed-phase high performance liquid chromatography (RP-HPLC). The results obtained showed that the extrusion conditions employed were able to retain high levels of vitamins A (86.7 to 93.4%) and C (88.4 to 98.6%) in all the extruded native starches. Within similar group of native starch source of corn, potato and rice, vitamins A and C retention was significantly higher in extruded waxy corn, potato and rice than in their extruded regular and high amylose starches. In extruded pregelatinized starches, the retention of vitamins A and C was significantly improved compared to that of their extruded native starches counterparts. Vitamin A concentration in extruded pregelatinized starches was the highest in extruded pregelatinized waxy corn ( $11.363 \pm 0.25$  mg/g) and the lowest in extruded pregelatinized regular potato ( $8.447 \pm 0.13$  mg/g). Extruded pregelatinized waxy corn also retained the highest vitamin C concentration ( $19.376 \pm 0.16$  mg/g) while the lowest concentration was found in extruded pregelatinized high amylose corn starch ( $16.467 \pm 0.27$  mg/g). Storage stabilities and determination of degradation kinetics of vitamins A and C in extruded native and pregelatinized starches were conducted at 25 °C in aluminium and nylon packagings. Degradation constant ( $k$ ) of vitamin A was lower in pregelatinized starches as compared to that in their native counterparts, ranging from 5.18 to

$8.67 \times 10^{-2}$ /week in aluminium packaging and 5.43 to  $10.02 \times 10^{-2}$ /week in nylon packaging. Similar pattern was observed for vitamin C with its degradation constant ( $k$ ) ranging from 3.55 to  $5.56 \times 10^{-2}$ /week in aluminium packaging and 3.94 to  $5.77 \times 10^{-2}$ /week in nylon packaging. It was observed that vitamins A and C in extruded starches were more stable in aluminium packaging than in nylon packaging as represented by the smaller degradation constant ( $k$ ). It can be concluded that in all the extruded starches, the stability during extrusion and upon storage was significantly higher for vitamin C than vitamin A. The findings provided information on the application of the fixed extrusion conditions for better retention of vitamin A or vitamin C in starches. Their stabilities in aluminium packaging and pregelatinized starches suggested that both vitamins were sensitive to light and air present in porous pregelatinized starch extrudates respectively.



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

**PENGEKALAN DAN KESTABILAN PENYIMPANAN VITAMIN A DAN C  
DALAM HASIL PENYEMPERITAN DARIPADA KANJI NATIF DAN  
PRAGELATIN**

Oleh

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**Mei 2015**

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**Fakulti : Sains dan Teknologi Makanan**

Sebuah kajian telah dijalankan bagi mengenal pasti pengekalan vitamin A dan C dalam hasil penyemperitan kanji natif dan prigelatin menggunakan keadaan penyemperitan mee kering. Dua peratus vitamin A atau C dicampur dengan kanji dan kandungan lembapan diselaraskan kepada 20% sebelum penyemperitan. Keadaan penyemperitan adalah pada suhu 80 °C di zon 1 dan 100 °C bagi zon 2, 3 dan 4 barel, 30 rpm kelajuan penyuar, dan 60 rpm kelajuan skrew. Keupayaan pelbagai kanji natif dan prigelatin dalam pengekalan vitamin A dan C semasa proses penyemperitan telah dianalisis. Kanji natif yang digunakan adalah jagung berliilin (99.1% amilopektin), jagung biasa (26.2% amilosa), jagung tinggi amilosa (70.0% amilosa), ubi kentang berliilin (99.1% amilopektin), ubi kentang biasa (30.1% amilosa), beras pulut (92.6% amilopektin), beras biasa (19.4% amilosa), ubi kayu (25.9% amilosa), ubi keledak (29.9% amilosa) dan sagu (30.4% amilosa). Kanji prigelatin termasuk jagung berliilin, jagung biasa, jagung tinggi amilosa, ubi kentang berliilin, ubi kentang biasa, beras pulut, beras biasa, ubi kayu, ubi keledak dan sagu. Ketahanan vitamin A dan C selepas penyemperitan, telah dianalisis menggunakan kromatografi cecair prestasi tinggi fasa terbalik (RP-HPLC). Keputusan yang diperolehi menunjukkan keadaan penyemperitan yang digunakan mampu mengekalkan vitamin A (86.7 hingga 93.4%) dan C (88.4 hingga 98.6%) yang tinggi dalam semua hasil penyemperitan kanji natif. Dalam kumpulan sumber kanji yang sama bagi jagung, ubi kentang dan beras, vitamin A dan C menunjukkan pengekalan yang signifikan dalam hasil penyemperitan kanji jagung berliilin, ubi kentang berliilin dan beras pulut berbanding hasil penyemperitan kanji biasa dan tinggi amilosa mereka. Bagi hasil penyemperitan kanji prigelatin, ketahanan vitamin A dan C telah meningkat dengan signifikan berbanding hasil penyemperitan kanji natifnya. Kepekatan vitamin A dalam hasil penyemperitan kanji prigelatin adalah tertinggi bagi hasil penyemperitan kanji prigelatin jagung berliilin (11.363±0.25 mg/g) dan terendah dalam hasil penyemperitan kanji prigelatin ubi kentang biasa (8.447±0.13 mg/g). Hasil penyemperitan kanji prigelatin jagung berliilin juga mengekalkan kepekatan vitamin C tertinggi (19.376±0.16 mg/g) manakala kepekatan terendah didapati dalam hasil penyemperitan kanji prigelatin jagung tinggi amilosa (16.467±0.2 mg/g). Kestabilan penyimpanan dan penentuan

degradasi kinetik bagi vitamin A dan C dalam hasil penyemperitan kanji natif dan kanji prigelatin dijalankan pada suhu 25 °C di dalam pembungkusan aluminium dan nilon. Pemalar degradasi ( $k$ ) vitamin A lebih rendah dalam kanji prigelatin berbanding kanji natif, dari julat 5.18 hingga  $8.67 \times 10^{-2}$ /minggu dalam pembungkusan aluminium dan 5.43 hingga  $10.02 \times 10^{-2}$ /minggu dalam pembungkusan nilon. Pemerhatian yang sama didapati bagi pemalar degradasi ( $k$ ) vitamin C dari julat 3.55 sehingga  $5.56 \times 10^{-2}$ /minggu dalam pembungkusan aluminium dan 3.94 to  $5.77 \times 10^{-2}$ /minggu dalam pembungkusan nilon. Keadaan ini menunjukkan bahawa vitamin A dan C dalam hasil penyemperitan kanji adalah lebih stabil dalam pembungkusan aluminium berbanding nilon yang diwakili oleh nilai pemalar degradasi ( $k$ ) yang lebih kecil. Kesimpulannya, dalam semua hasil penyemperitan kanji, kestabilan semasa penyemperitan dan sepanjang penyimpanan adalah lebih tinggi secara signifikan bagi vitamin C berbanding vitamin A. Hasil kajian ini memberikan maklumat tentang penggunaan keadaan penyemperitan yang tetap untuk pengekaln vitamin A dan C yang lebih baik dalam kanji. Kestabilan vitamin-vitamin dalam pembungkusan aluminium dan hasil penyemperitan kanji prigelatin mencadangkan bahawa vitamin-vitamin adalah sensitif kepada cahaya dan udara dalam hasil penyemperitan kanji prigelatin yang berongga, masing-masing.

## ACKNOWLEDGEMENTS

Bismillahirrahmanirrahim. Alhamdulillah, all praise to Allah, the One who has given me the opportunity to be in this three years journey. I have learnt a lot and I cannot ask for more from Him for the blessings that He has given me with all these experiences and many great and kind hearted people around me. Without Him and all the people, I will never be able to go through this alone, Alhamdulillah.

The first person that I really want to convey my deepest appreciation and gratitude is my supervisor, Assoc. Prof. Dr. Sharifah Kharidah Syed Muhammad. Her guidance and supervision during this process are limitless and her patience in mentoring me and all of us in the research laboratory really means a lot, not only now but for our future. She is always there to give me support, encouragement and advice, from many points of views. Not to forget my co-supervisors, Assoc. Prof. Dr. Roselina Karim and Mr. Dzulkifly Mat Hashim for their advice and inputs.

I am also thankful to Universiti Putra Malaysia and Ministry of Education for the sponsorship during my Master's study, without the sponsorship I will not be able to pursue my postgraduate study.

I would also like to thank the staff and students of UPM-BERNAS Research Laboratory, Faculty of Food Science and Technology, Chemistry Department of Faculty of Science and Microscopy Unit at the Institute of Bioscience for their assistance and cooperation when I was conducting my laboratory work at their premises. I would like to thank Sri, Bala, Putri, Hamidah, Zhafarina, Safura, Kak Azmah, Mrs. Yoge, Makeri, Kak Teh, Kak Izan and Kak Suzana for being part of my academic quest.

My heartfelt appreciation goes to my mother Mrs. Huzaimah Che Zakaria, my father, Mr. Abdul Manan Ali, my brother Mohd Abdul Fatah Abdul Manan and my big family members for their moral support and prayers along this journey of mine.



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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## LIST OF ABBREVIATIONS

AACC	American Association of Cereal Chemists
AF	Aluminium foil
AL	Aluminium
ANOVA	Analysis of variance
AOAC	Association of Official Analytical Chemists
DP	Degree of polymerization
EDTA	Diaminoethanetetra-acetic acid disodium salt
EDX	Energy dispersive X-ray detector
g	Gram
IBM	International Business Machines Corporation
kV	Kilovoltage
L	Litre
LDPE	Low density polyethylene
mA	Milliampere
mg	Milligram
mL	Millilitre
mm	Millimeter
NAD	Nicotinamide adenine dinucleotide
NADPH	Nicotinamide adenine dinucleotide phosphate
nm	Nanometer
NY	Nylon
PE	Polyethylene
RDI	Recommended daily intake
RH	Relative humidity
RP-HPLC	Reversed-phase high performance liquid chromatography
RPM	Revolutionary per minute
SC-CO <sub>2</sub>	Supercritical carbon dioxide
SEM	Scanning electron microscopy
SPSS	Statistical package for the social science
VAD	Vitamin A deficiency
w/v	Weight per volume
XRD	X-ray diffraction
µg	Microgram
µL	Microlitre
µm	Micrometer

## CHAPTER 1

### INTRODUCTION

Micronutrient deficiency with a focus on vitamin A, iron, iodine and zinc has been reported worldwide. The most affected groups of people are children, young women, and pregnant women with vitamin A deficiency (VAD) being the most common deficiency in many developing countries (Hussain et al., 2014). In Asia, Africa and Latin America, especially in the lower-income to middle-income countries, a large number of their population are facing undernutrition and suffer from poverty-related diseases. Deficiency in micronutrients can cause many health problems; most of them related to physical and cognitive developments (Khambalia et al., 2012).

In Malaysia, iron deficiency has been reported to be the most frequent cause of anaemia especially in pregnant women and women in reproductive age (Milman, 2015). Besides iron deficiency, anaemia was also mentioned to be caused by lack of hematinic vitamins such as folate and vitamin B<sub>12</sub> among these groups of women. A study by Poh et al. (2013) on dietary intake among Malaysian children aged between 6 months to 12 years old compared the nutrient intakes by these children to Malaysian Recommended Daily Intakes (RDI) and the finding revealed that one-third of the children were insufficient in energy, calcium and vitamin D. The study also found that most of the children were more prevalent towards overnutrition than undernutrition.

Food fortification is widely used as an effort to overcome malnutrition problems. Fortification of selected and targeted nutrients in suitable food vehicles is a cost-effective and sustainable effort because through this, the target group of people can easily obtain access to their fortified staple food. Food vehicles can be varied according to population background and staple food. In Asia, the population consumes a lot of rice and noodles, providing opportunity to fortify these vehicles so as to overcome micronutrient deficiency. Ultra Rice® is an example of a reconstituted rice product fortified with vitamin A, iron, and vitamin B<sub>1</sub> (Li et al., 2011) and serves as a staple food for more than half of the world's population.

Since fortified rice has already been developed, noodles, which are popular in Asia, can also be considered as vehicles for fortificants. There are several types of commercial noodles available, which are cereal- or legume-based with starch added as a binder. They are sold fresh or in dried form. The production of dried noodles via the conventional method involves forming the noodles, cooking of the noodles in water or by steaming followed by drying. Dried noodles produced by machines require a mixer working at low or normal pressure, a press with a forming die, a boiling, steaming or frying unit, a drying unit, and a packaging device (Wójtowicz, 2011).

For noodles made via extrusion cooking, the press with a forming die unit is replaced by a single or twin screw extruder. The conventional method of dried noodle production applies a high degree of starch gelatinisation at higher moisture and temperature in the steaming, boiling or frying process. The drying of these noodles takes longer time to reach the final desired moisture content. Extrusion helps improve the production of dried noodles especially in terms of cooking and drying time compared to the conventional method because of the low moisture required in the heating process. The dried noodles prepared by extrusion require a shorter time for production and drying. Plus, it is easy to prepare and serve afterwards. The combinations of the barrel zone temperatures ranging from 40 to 110 °C, screw speeds of 20 to 150 rpm, feeder speeds from 20 to 60 rpm with a moisture content of 30 to 35% have been employed in the extrusion of various cereal- and legume-based noodles (Charutigon et al., 2008; Sereewat et al., 2015; Wang et al., 2014).

Due to the extrusion conditions, fortification of dried noodles as part of the effort to overcome malnutrition could be conducted. A study on the fortification of rice noodles prepared using the conventional method and fortified with vitamin A, folic acid, and iron had been carried out (Malahayati et al., 2014). It was found that iron was more stable than vitamin A and folic acid after several heating and processing conditions of the rice noodle. The retentions of the fortificants were not only dependent on their stability (the minerals were more stable during processing) but also dependent on properties of the macro-components present in the noodles (such as starch, protein, and fibre) with noodle's major component is starch.

Therefore, the objectives of this study were:

1. To determine the vitamin A and C retention in native starches extruded using conditions for the production of dried noodle.
2. To study the effects of pregelatinized starches on vitamin A and C retention during extrusion.
3. To conduct storage stability tests of vitamin A and C in extruded native and pregelatinized starches.
4. To evaluate degradation kinetics of vitamins in extruded native and pregelatinized starches.

The significance of this study was to provide information on the application of fixed extrusion conditions for better retention of vitamin A or vitamin C in starches. The selection of vitamins A and C was to represent the model system of fat-soluble and water-soluble vitamins.

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