UNIVERSITI PUTRA MALAYSIA

CHARACTERIZATION OF EXTRACTED CARBOXYMETHYL CELLULOSE FROM OIL PALM EMPTY FRUIT BUNCH STALK FIBERS FOR ICE CREAM APPLICATION

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FK 2018 127

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By

DZIEDA BINTI MUHAMAD PARID

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

May 2018

Dedicated to my family

For your endless love, support and encouragement

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

CHARACTERIZATION OF EXTRACTED CARBOXYMETHYL CELLULOSE (CMC) FROM OIL PALM EMPTY FRUIT BUNCH (OPEFB) STALK FIBERS FOR ICE CREAM APPLICATION

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May 2018

Chairman : Nur 'Aliaa Abd Rahman, PhD
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The current extraction of carboxymethyl cellulose (CMC) from wood has created competition with wood industries. Interest in alternative sources is critical to ensure the sustainable production of CMC. Therefore, the extraction of CMC from oil palm empty fruit bunch (OPEFB) stalk fibers was evaluated. Based on previous work, highest cellulose content was obtained from raw stalk fibers with the least amount of lignin and residual oil as compared to the empty fruit bunch (EFB) and spikelet. Based on the results, OPEFB stalks fibers is suitable to be used as the substrate to produce CMC as proven by its cellulose composition (76.45%), proven morphology images, changes of diffraction patterns and new functional group occurred throughout the extraction process. Meanwhile, extracted CMC in powder form shows a good physicochemical properties in terms of its moisture content (11.18%), densities, flowability, good water retention capacity (21.55%), approaching desired colour of CMC and good physical appearance. Extracted CMC in its aqueous form also shows a comparable viscosity behaviour as it depicts a good shear thinning characteristics at 4% and the same flow behaviour curve as shown by the commercial CMC at 1% and 4% concentration.

Prior to the production of the extracted CMC, the potential application of the extracted CMC from OPEFB stalk fibers were analysed on hard ice cream properties. Such data are useful in accessing the overall quality during the production and handling of hard ice cream, as well as in developing a desirable mouthfeel. The extracted CMC from OPEFB stalks fibers was able to increase the viscosity of liquid ice cream mixture (129.4cp) and hard ice cream produced by using the extracted CMC shows a good and comparable melting characteristics

as it shows the same melting rate with hard ice cream produced using commercial CMC, with melting resistance (60.28%), lower overrun (35.2%) and lower hardness (28.88N) which will make the hard ice cream creamier, smoother and richer taste and the hard ice cream produced using sCMC shows a viscoelastic behaviour same as the hard ice cream produced using commercial CMC.

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

PENCIRIAN KARBOKSIMETIL SELULOSA YANG DIEKSTRAK DARI SERAT TANDAN BUAH KELAPA SAWIT KOSONG UNTUK APLIKASI DI DALAM AIS KRIM

Oleh

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Pengekstrakan terkini karboksimetil selulosa (CMC) dari kayu telah mewujudkan persaingan dengan industri kayu. Kepentingan dalam sumber alternatif adalah penting untuk memastikan pengeluaran CMC yang mampan. Oleh itu, pengekstrakan CMC dari serat tandan buah kelapa sawit kosong (OPEFB) telah diuji. Berdasarkan projek sebelum ini, kandungan selulosa tertinggi diperolehi dari serat tangkai mentah dengan jumlah sisa minyak dan lignin yang paling sedikit berbanding dengan seluruh tandan buah kelapa sawit kosong (EFB) dan spikelet. Berdasarkan hasilnya, serat tangkai OPEFB sesuai untuk digunakan sebagai substrat untuk menghasilkan CMC sebagaimana terbukti dengan jumlah komposisi selulosa (76.45%), gambar morfologi yang telah terbukti, perubahan corak difraksi dan kumpulan fungsi telah terjadi sepanjang proses pengekstrakan. Sementara itu, CMC yang diekstrak dalam bentuk serbuk menunjukkan sifat fizikokimia yang baik dari segi kandungan lembapannya (11.18%), ketumpatan, pengaliran, kapasiti pengekalan air yang baik (21.55%), menghampiri warna CMC yang dikehendaki dan penampilan fizikal yang baik. CMC yang diekstrak dalam bentuk cecair juga menunjukkan sifat kelikatan yang setanding dengan CMC komersil kerana ia menggambarkan ciri-ciri penipisan ricih yang baik pada kepekatan 4% dan lengkung sifat aliran yang sama seperti yang ditunjukkan oleh CMC komersil pada kepekatan 1% dan 4%.

CMC yang diekstrak dari serat tangkai OPEFB dianalisis pada sifat ais krim. Data sedemikian berguna dalam mengakses kualiti keseluruhan semasa pengeluaran dan pengendalian ais krim, serta sifat ais krim dalam mulut yang wajar. CMC yang diekstrak daripada serat tangkai OPEFB dapat meningkatkan kelikatan campuran ais krim (129.4cp) dan ais krim yang dihasilkan dengan menggunakan CMC yang diekstrak memberikan ciri pencairan yang baik dan

setanding dengan ais krim yang dihasilkan dengan CMC komersil kerana ia menunjukkan kadar lebur yang sama dengan rintangan lebur (60.28%), overrun lebih rendah (35.2%) dan kekerasan (28.88N) yang akan menjadikan ais krim mempunyai rasa yang lebih bagus dan enak. Tambahan lagi, ais krim yang dihasilkan menggunakan sCMC menunjukkan sifat viscoelastik yang sama seperti ais krim yang dihasilkan menggunakan CMC komersial.

ACKNOWLEDGEMENT

Alhamdulillah, thank you Allah SWT for giving me endless strength and courage to complete my project. First of all, I would like to express my sincere gratitude to my main supervisor and supervisory committee, Dr Nur 'Aliaa Abd Rahman, Dr Azhari Samsu Baharuddin and Dr Mohd Afandi P Mohammed for their guidance, enthusiasm, encouragement, support and inspiring knowledge in this project.

Furthermore, special thanks to my university, Universiti Putra Malaysia for providing me with a comfortable environment and equipment needed to carry out my MSc project. I also would like to take this opportunity to express my uncountable thanks to the lab technicians from the Department of Process and Food Engineering for their help throughout the experimental work in indicating the equipment and devices that I required and in making the setup for the processes. A special appreciation also goes to my fellow Ice Cream Research Group: Siti Zubaidah, Amiruddin and others for their joyful and memorable friendship moments. Not to forget, my seniors, Dr Halimaton, Dr Nadia, Loo Xiang, Sobah and Termizi who always helped me in handling experiments and giving advice based on their precious experiences, thank you so much for lending your time and willingness. The appreciation is beyond words.

Up and foremost, my indebtedness goes to my dearest parents, Hj Muhamad Parid and Hjh Norehan, my lovely husband, Syed Fuad and not to forget my supportive siblings, Dzuan, Dzuady, Dziela and their entire family, for their faith, understanding, endless support and motivation and most importantly their prayers.

Thank you for the love and strength.

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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LIST OF ABBREVIATIONS

ADF	acid detergent fibre
ADL	acid detergent lignin
C	carbon
CI	Carr index
CMC	Carboxymethyl cellulose
COMM1	CMC from Warisnove, Pahang, Malaysia
COMM2	CMC from R&M Chemicals, Petaling Jaya, Malaysia
CrI	crystallinity index
EFB	Empty fruit bunches
FFB	Fresh fruit bunches
FELCRA	Federal Land Consolidation and Rehabilitation Authority
FELDA	Federal Land Development Authority
FTIR	Fourier Transmission Infrared
G	guaicyls
H	ρ-hydroxyphenyls
H	hydrogen
HR	Hausner ratio
MCC	Microcrystalline cellulose
MPOB	Malaysian Palm Oil Board
N	nitrogen
$NaClO_2$	sodium chlorite

NaOH	sodium hydroxide
NDF	Neutral detergent fibre
OPEFB	Oil palm empty fruit bunch
O	oxygen
PTFE	Polytetraflouroethylene
S	syringyls
sCMC	CMC extracted from OPEFB stalk fibers
SEM	Scanning Electron Microscopy
WI	whiteness index
WRC	water retention capacity
XRD	X-ray diffractometer
YI	yellowness index
V_{gas}	volume of gas
V_{liquid}	volume of liquid
p_{tapped}	tapped density
p_{bulk}	bulk density
ΔE	total color difference

LIST OF SYMBOLS

˚C	degree celcius
g/L	gram per litre
%	percentage
rev/min	revolutions per minute
β	beta
O_2	oxygen
CO_2	carbon dioxide
H_2O	water
h	hour
min	minutes
µm	micrometer
mm	milimeter
Pa	Pascal
U/g	unit per gram

CHAPTER 1

INTRODUCTION

1.1 Research Background

Southeast Asian countries like Malaysia generated abundance of biomass leftover particularly produced from the mill itself including the plantation area whilst oil palm is considered as the main crop in the country. Palm oil industry contribute a few types of solid biomass and the oil palm empty fruit bunch (OPEFB) is considered the largest solid waste produced. During the threshing process, the fresh fruit bunches (FFB) are rolled and threshed in rotating steel drums to separate the fruits from the stalks and this process generates the oil palm empty fruit bunches (OPEFB). The two main parts of OPEFB are stalk and spikelet, where Xiang et al. (2016) reported that the raw stalk fibers yielded the highest cellulose content and had the lowest lignin content. Likewise, the raw stalk fibers contained the lowest residual oil content when compared to the OPEFB as a whole and spikelets (Yunos et al. 2015; Xiang et al. 2016). Spikelets was surrounded by the fruitlets, which were the main source of oil and make it comprised of a higher amount of residual oil. Residual oil on the oil palm fibres need to be omitted because it can influence the derivation of cellulose production, thus it must go through a supplemental pre-treatment step.

The attempts to transform OPEFB into value-added products have gained wide attention because it is one of the most produced biomasses that come from oil palm refineries since it was known to have a rich source of cellulose. Cellulose must be transformed into its derivatives to be used in the food industry, such as carboxymethyl cellulose (CMC). CMC is a linear, water soluble, long-chain, and an anionic polysachharide artificial modified cellulose and it can be described as a white to cream color, tasteless, odorless, and free-flowing powder (Adinugraha et al., 2005) that is widely applicable for foods and pharmaceuticals (Tasaso, 2015). Extraction of CMC from oil palm fibers has been favourable given that Asian countries such as Malaysia generates abundance of oil palm biomass. OPEFB which is made up of lignocellulosic components will be used as the raw material to produce CMC.

The extracted cellulose; CMC act as a stabilizers and functional ingredients in hard ice cream. The solubility of these cellulose derivatives in cold and hot water causes the modification of rheological properties and produces structure and texture improvements of hard ice cream since hard ice cream is prepared in two phases which are during ice cream mix preparation and freezing process. Only small amount of CMC can be utilized to achieved its credit functionality to hard ice cream texture and condition. Besides, 0.5% is the highest amount that can be used in order to utilize CMC in hard ice cream. This study will also focus on the potential application of extracted CMC on hard ice cream attributes. Such data are useful in accessing the overall quality during the production and handling of hard ice cream, as well as in developing a desirable mouthfeel. The texture, which can be defined as the physical and mouthfeel characteristics of a food or drink, is very unique in hard ice cream due to the fact that it contains all three state of matter: solid (found in the ice crystals and fat globules), liquid (found in the sugar solution), and gas (found in the air bubbles). The desired texture is essential for customer satisfaction and can be evaluated through a variety of viscoelastic and mechanical properties, which include its viscosity behaviour, hardness, compressive strength, and stress relaxation. Mechanical tests for each of these properties were developed in order to compare the textural characteristics of regular hard ice creams due to its different composition (Casarotto, 2015).

No report on utilizing the raw OPEFB stalk fibers to produce carboxymethyl cellulose (CMC) is currently available. Main objective is to create a complete link of using palm oil mill by-product to produce valuable food enhancer and application of it. Thus, it is an essential to have a proper study to recover the lignocellulosic components for the conversion of cellulose derivatives. This study also will help the palm industry to provide further information about the oil palm biomass, which is important for future bioconversion processes. The production of CMC from raw OPEFB stalk fibers was conducted by using a chemical extraction method, prior to proceeding with the physicochemical and rheological analyses of the extracted CMC. Comparison was made between the CMC produced in this study with those commercially available. The current research provided the first step towards the feasibility of clean OPEFB stalk fibers as a source to produce CMC. This will promote the usage of raw material and may boost the economic value of hard ice cream product in Malaysia since it utilizes the palm oil by-product abundance.

1.2 Problem Statement

Development of products that transform waste materials to beneficial products is of great importance and need to be prioritized. During oil collection in the palm oil mills, excessive waste are generated after the threshing process. One of the abundance is the oil palm empty fruit bunch (OPEFB). Although there are plenty of studies were done to acquire cellulose derivatives from oil palm fibers, all projects generally emphasized on the whole bunch of OPEFB, without specific study on particular parts of OPEFB for instance stalk and spikelet. It is often utilized as a whole but researchers have found that raw stalks fibers comprised the highest percentage of cellulose and the lowest lignin content compared to OPEFB as a whole and spikelet fibres. On top of that, lack studies emphasized on the potential application of the extracted CMC have been tested on the hard ice cream quality in terms of its textural properties and mechanical behaviour.

1.3 Objectives of the Studies

The main objective of this thesis work is to characterize carboxymethyl cellulose (CMC) derived from oil palm empty fruit bunch (OPEFB) stalks fibers for hard ice cream application. To be specific, this research aims:

1. To characterize physicochemical properties of carboxymethyl cellulose (CMC) extracted from oil palm empty fruit bunches (OPEFB) stalks fibers;
2. To assess the potential application of extracted CMC by analysing the textural properties and mechanical behaviour of hard ice cream.

1.4 Scope of Research

This study is principally concerned about the feasibility study of utilizing the oil palm empty fruit bunch (OPEFB) stalk fibers as the material to be extracted as carboxymethyl cellulose (CMC). The research continues with the potential application of the extracted CMC on hard ice cream properties. During this research, an in depth study has been performed in studying the characteristics of the material throughout the extraction process that involved lignocellulosic properties, FTIR analysis, XRD analysis and microstructural analysis. Then, the extracted CMC were further analyse on its properties; physicochemical properties and viscosity properties. On the other hand, the potential application of the extracted CMC has been tested on hard ice cream properties that involved the textural properties and mechanical behaviour and the performance were compared with the hard ice cream prepared using commercial CMC. The potential application of the extracted CMC from OPEFB stalk fibers were analysed on hard ice cream properties including viscosity of ice cream mix, melting characteristics, overrun, hardness test and mechanical compression. The properties were compared with ice cream prepared using commercial CMC. Some limitation occurred for mechanical test of hard ice cream samples by using the actual texture analyser from commercial brand. This was because when the test was done at room temperature, the hard ice cream did not hold its structure and melted faster. Hence, a toy-brick miniature tester was designed to study the compression behaviour of hard ice cream produced using the extracted CMC in a small freezer to maintain the hard ice cream temperature and shape. Lego Mindstorms EV3 consists of microprocessors, motors, and sensors that can be programmed to perform basic tasks on repeat. It involves a capable software to manage any essential movement that will assist the features of the toy-brick.

Matlab programme was used to navigate a simple up, down, up, move sequence on a loop.

1.5 Thesis Structure

There are 5 chapters in this thesis. In Chapter 1, a brief introduction of the overall research was written together with objectives of the study and the problem statement of the research. In Chapter 2, extensive literature review was written covering current available knowledge on the oil palm biomass, carboxymethyl cellulose (CMC) and hard ice cream properties. In Chapter 3, the overall experimental framework was explained on how the materials were prepared, method used on extracting carboxymethyl cellulose (CMC), characterization of materials throughout the extraction process, analysis on CMC properties and the procedure on determining the hard ice cream properties. The objectives of the research were further explained in Chapter 4. First objective was well intricate in which to produce and characterize the extracted carboxymethyl cellulose (CMC) extracted from oil palm empty fruit bunch (OPEFB) stalk fibers. Meanwhile, second objective was elaborated in which to study the potential application of the extracted CMC from OPEFB stalk fibers on hard ice cream properties. The hard ice cream properties were compared with hard ice cream prepared using commercial CMC. Finally, in Chapter 5, final conclusions and some of recommendations were mentioned. Appendix and references used in this entire study was listed at the back of the thesis.

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