UNIVERSITI TEKNOLOGI MARA

CRYSTALLISATION BEHAVIOUR OF PALM OIL: APPLICATION OF FRACTAL ANALYSIS ON CRYSTAL NETWORK OF PALM OIL PRODUCTS

ZALIHA OMAR

Thesis submitted in fulfillment of the requirements for the degree of Doctor of Philosophy

Faculty of Applied Sciences

August 2006

Abstract

Refined bleached deodorised palm oil (RBDPO) was blended with 4 to 25% (w/w) fully hydrogenated palm oil (HPO). The effect of blending, crystallisation kinetics and storage temperatures on the physical, chemical, morphological and macroscopic rheological properties of these samples were studied. The microstructural level of crystal networks of the samples were also quantified via fractal geometrical methods by rheological measurement (D*f*) and image analysis (D*b*). The results showed a significant ($p<0.05$) increase in the slip melting point (SMP) from 37 to 53°C and maximum solid fat content (SFC) of 69% at 5°C and completely melted within 45 to 55°C with HPO added. However, X-ray diffraction result showed that, the pure RBDPO and HPO blends at all ratios were stabilised in the β′ polymorphic form. Crystallisation kinetics studied using the Avrami model indicated that, pure RBDPO and the blends crystallised in a rod-like growth mechanism from instantaneous nucleation (Avrami exponent n=2) at 10°C and by a disc-like growth mechanism with n = 3 from sporadic nucleation at 15 and 20°C. Blends with 4% of HPO at 15°C crystallised with a smaller crystallisation rate constant (k= 0.00432 k^{-1min}), lower nucleation activation free energy (ΔG_c,= 1.02 kJ/mol) and a shorter induction time (ι) using the Fisher-Turnbull model. During storage at 15, 20 and 25°C, there was an increase in storage modulus (G′) and hardness index (HI). At 15°C, fractal dimensions (D*f* and D*b*) of pure RBDPO by both methods respectively decreased from D*f*=2.50 to 2.41 and D*b*=1.38 to 1.22 . The same trend was observed for fractal dimension of the crystal network at 20 and 25°C. However, there was no significant ($p>0.5$) increase in the SFC of the microstructures of pure RBDPO and HPO blends. The β′ crystal of the microstructures of the blends showed better dispersion and smaller clusters than pure RBDPO. An increase in storage modulus (G′), hardness index (HI) and compression force (g) indicated that the crystal network of the blends are becoming more highly structured with increasing storage time. This implies that post-hardening had occurred without transformation of polymorphic form and is due to crystal networking after the crystallisation process.

ACKNOWLEDGEMENTS

I wish to express my deepest gratitude to my supervisor, Associate Professor Dr. Cheow Chong Seng of the Food Technology Department, Faculty of Applied Sciences, UiTM for his valuable guidance, suggestion and advise during the completion of this thesis.

My deepest appreciation goes to my co-supervisor, Principal Research Officer, Dr. Chong Chiew Let of the Lipid Technology Group, MPOB for his guidance and encouragement throughout my study and completing this project.

Appreciation and thanks are dedicated to all Physics laboratory staffs of MPOB who are directly or indirectly involved towards the completion of this project, especially Pn. Roziah, Cik Zukarinah, En. Mohd. Khairul Nizam, En. Mohd. Nizam and Pn. Noraini Muhammad for their kind cooperation. Special thanks to Dr. Norizzah of UiTM, for her kind friendship and valuable advise.

Acknowledgement is due to MPOB for financial support to attend training on fractal analysis in Guelph University, Ontario, Canada, from 3rd. October to15th November 2004. Greatest appreciation and thanks to Prof. Alejandro Marangoni, Centre of Food and Soft Materials Science, University of Guelph for the valuable guidance and making arrangement for the experiments in the laboratory during the training. Speacial thanks to Dr. Amanda, Silvana, Latifeh, Heidi, Dong Ming, Alejandra and Staffanie for their kind cooperation during the training.

I wish to express my heartfelt appreciation to my mum, sisters and brothers for their continuous encouragement and support. To my daughter Nur Hamizah, thank you for her love, understanding, patience and sacrifice.

Last but not least, I would like to express my everlasting memory to my late father Haji Omar and late sister Nur Hashimah.

May Allah bless you all.

Wassalam.

TABLE OF CONTENTS

CHAPTER 1

INTRODUCTION

1.1 Palm Oil

Palm oil is derived from the mesocarp of the fruits of the oil palm species *Elaeis guineensis* mainly of the hybrid *Tenera.* The palm oil industry worldwide has provided the fastest increase in global oils and fats supplies over the last four decades. World palm oil production increased 20-fold from a mere 2 million tonnes in 1962 to 25.0 million tonnes in 2002, growing at a rate of 7.8% p.a. or more than double that of the total world oils and fats production which has a growth rate of 3.5% p.a. during this period. Malaysia and Indonesia account for more than 80% of the world's palm oil production (Yusof et al., 2004). Palm oil has many applications with potential applications currently being explored. The uses of palm oil can be categorised into edible and non-edible purposes. One of the technological concerns of the edible oil industry is how to expand the multiple usage of palm oil. The growth of palm oil in the world oils and fats market had led to many studies, particularly into its crystallisation behaviour and nutritional value.

Palm oil is a semi-solid oil at ambient temperature. It is a mixture of low and high melting triglycerides and can be fractionated into a liquid fraction called palm olein and a solid fraction called palm stearin. Palm olein is an excellent cooking oil and palm stearin is an excellent hard stock for food applications as it is free from trans-fatty acids as compared to other hydrogenated liquid vegetable oils.

1.2 Fat Crystallisation

Fat is a material that is composed of an intimate mixture of liquid and solid fractions whose main constituents are triacylglycerols. Triacylglycerol structures and crystallisation conditions will influence crystal behaviour upon crystallisation. Crystals aggregate into larger structure that will eventually form a continuous 3-dimensional network, which is largely responsible for the solid-like properties of