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APPLICATION OF DIFFERENTIAL SCANNING CALORIMETRIC METHOD FOR ASSESSING AND MONITORING VARIOUS PHYSICAL AND OXIDATIVE PROPERTIES OF VEGETABLE OILS

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By

TAN CHIN PING

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June 2001

UPM

To my family
Dad, Mom, Grandma, Sister, and Brother

Here is the endeavor of my absences

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

APPLICATION OF DIFFERENTIAL SCANNING CALORIMETRIC METHOD FOR ASSESSING AND MONITORING VARIOUS PHYSICAL AND OXIDATIVE PROPERTIES OF VEGETABLE OILS

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June 2001

Chairman: **Professor Yaakob Bin Che Man, Ph.D.**

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Differential scanning calorimetry (DSC) can be a powerful instrumental technique for analyzing oils and fats systems but has tended not to be well understood and used in the field of oils and fats. The main purpose of this project is to develop various techniques based on DSC to study the physical and chemical properties of vegetable oils. High-performance liquid chromatography (HPLC), gas-liquid chromatography (GLC), oxidative stability instrument (OSI), and various standard chemical analyses were used in this investigation to complement the DSC methods.

This work is a systematic study of vegetable oils' melting and crystallization profiles by using DSC. The investigation began with the successful comparison of the DSC thermal curves of 17 different vegetable oils. Thorough investigations in this work were also directed towards obtaining basic information about the relationship between thermal profiles and chemical compositions of 17

different vegetable oils. Thereafter, the effects of DSC scanning rate variation were studied. Scanning rates were found to affect melting/crystallization profile, melting (and/or crystallization) offset (and/or onset) and peak temperatures, and peak enthalpies of all vegetable oils.

In this study, DSC was utilized to monitor the oxidation of heated oils during deep-fat frying and microwave heating. A statistical comparative study was carried out on the DSC and standard chemical methods. The results revealed that there is good correlation ($P < 0.01$) between the DSC method and other standard chemical methods. In another study, a new calorimetric technique was developed to determine three important quality indices in deep-fat frying industry namely, total polar compounds (TPC), free fatty acid (FFA) content and iodine value (IV) of heated oils using the DSC cooling profiles. The studies have shown that all DSC methods developed were comparable to the standard American Oil Chemists' Society (AOCS) methods.

A simple and efficient DSC technique to determine the oxidative stability of vegetable oils was described. The isothermal DSC technique for direct determination of the oxidative stability of vegetable oils has been built and a comparative study to OSI was demonstrated. The results indicated that there is good correlation ($P < 0.01$) between the DSC oxidative induction time (T_o) and OSI values. Isothermal calorimetry was then employed as a general analytical method where the Arrhenius kinetic data for the lipid oxidation of vegetable oils were obtained by measurement of the DSC T_o at various temperatures. The present

study also developed a simple method for measuring the antioxidant activity in $RBDPO_O$ using isothermal DSC technique.

Generally, this project concluded that DSC appears to be a useful method in determining various physical and chemical parameters of vegetable oils, and it may have the potential to replace the laborious, time- and chemical-consuming standard methods. The various methods developed here can be applied in the oils and fats industry.

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

PENGGUNAAN KAEDAH PERMETERAN KALORI PENGIMBASAN KEBEZAAN (DSC) DALAM PENILAIAN DAN PENELITIAN PELBAGAI CIRI FIZIKAL AND OKSIDATIF MINYAK SAYUR-SAYURAN

Oleh

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Jun 2001

Pengerusi: **Profesor Yaakob Bin Che Man, Ph.D.**

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Permeteran kalori pengimbasan kebezaan (DSC) mampu bertindak sebagai satu teknik peralatan yang berkebolehan dalam menganalisa sistem minyak and lemak. Walau bagaimanapun, dalam bidang penyelidikan minyak and lemak, penggunaan dan pemahaman alat ini masih berada di tahap yang kurang memuaskan. Tujuan utama projek ini adalah untuk mengembangkan pelbagai teknik peralatan yang berasaskan DSC dalam kajian pelbagai ciri fizikal dan kimia minyak sayur-sayuran. Kromatografi cecair berprestasi-tinggi (HPLC), kromatografi gas-cecair (GLC), alat pengukuran kestabilan oksidatif (OSI) dan pelbagai analisa kimia piawai telah digunakan untuk melengkapi kaedah-kaedah DSC.

Kajian ini telah dimulakan dengan melakukan satu penyelidikan yang sistematik ke atas profil-profil peleburan dan pembekuan minyak sayur-sayuran dengan penggunaan DSC. Penyelidikan ini telah berjaya membuat perbandingan kelok-kelok termal DSC

bagi 17 minyak sayur-sayuran yang berlainan. Kajian yang menyeluruh ini juga telah memperolehi maklumat asas hubungan di antara profil terma dan komposisi kimia 17 minyak sayur-sayuran yang berlainan.Tambahan lagi, kesan penggunaan kadar imbasan DSC yang berbeza telah dikaji. Kadar imbasan didapati mempengaruhi profil peleburan/pembekuan, suhu permulaan (dan/atau pengakhiran) takat peleburan (dan/atau pembekuan), suhu puncak-puncak kelok terma dan entalpi-entalpi kelok terma bagi kesemua minyak sayur-sayuran yang telah digunakan.

Dalam kajian ini, DSC telah digunakan untuk meneliti pengoksidaan minyak yang dipanaskan secara penggorengan dan pemanasan gelombang mikro. Satu kajian perbandingan secara statistik telah dilakukan di antara kaedah-kaedah kimia piawai dan DSC. Keputusan-keputusan kajian telah menunjukkan bahawa terdapat korelasi ($P <$ 0.01) yang baik di antara kaedah DSC dan kaedah-kaedah kimia piawai. Dalam kajian yang lain, satu teknik kalorimetrik yang baru telah dibina bagi penentuan secara serentak tiga indeks kualiti yang utama dalam industri penggorengan. Ia melibatkan penentuan jumlah sebatian polar (TPC), kandungan asid lemak bebas (FFA) dan nilai iodin (IV) dalam minyak yang telah dipanaskan dengan menggunakan profil-profil pembekuan DSC. Kajian ini telah menunjukkan bahawa kesemua kaedah DSC adalah setanding dengan kaedah-kaedah piawai *American Oil Chemists' Society* (AOCS).

Satu teknik DSC yang mudah dan cekap telah diperihalkan untuk menentukan tahap kestabilan pengoksidaan minyak sayur-sayuran. Teknik DSC secara isoterma telah dibina bagi menentukan tahap kestabilan pengoksidaan minyak sayur-sayuran secara langsung dan perbandingan dengan kaedah OSI telah dijalankan. Keputusan-keputusan ini telah menunjukkan bahawa terdapat korelasi ($P <$0.01) yang baik di antara masa

induksi pengoksidaan DSC (T_o) dan nilai OSI. Analisa kalorimetrik secara isoterma kemudiannya telah digunakan sebagai satu kaedah penganalisaan umum di mana data kinetik *Arrhenius* bagi pengoksidaan lipid dalam minyak sayur-sayuran telah diperolehi melalui pengukuran nilai T_o pada beberapa suhu yang berbeza. Projek ini juga telah membina satu kaedah yang mudah bagi mengukur aktiviti antioksidan dalam RBDPO$_O$ dengan menggunakan teknik DSC secara isoterma.

Secara umum, penyelidikan ini menyimpulkan bahawa DSC merupakan satu kaedah peralatan yang bermakna dalam penentuan pelbagai parameter fizikal dan kimia minyak sayur-sayuran. Ia berpotensi untuk menggantikan kaedah-kaedah piawai yang memerlukan langkah-langkah yang rumit, memakan masa dan melibatkan penggunaan bahan kimia yang banyak. Pelbagai kaedah yang telah dikembangkan dalam projek ini boleh digunakan dalam industri minyak dan lemak.

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A sense of joy and appreciation comes with the completion of this journey. It is funny how life turns you in a certain direction. The wind blows and you end up on a new path, searching for who you will become. It is certainly true that no man is an island. When I decided to fulfill my dreams and venture down the path towards a doctorate, I never knew I would meet such incredible people. The last three years of my life have been rewarding, challenging, and inspirational. As I have extended myself in this journey, I have been blessed with the friendship, guidance, and support of many people, from scientific to personal. There is no possible way to thank everyone who has helped me along the journey, so I'll start by expressing my eternal gratitude to everyone.

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At last this journey has come to an end and another begins.....

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