UNIVERSITI PUTRA MALAYSIA

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By

SEYED HAMED MIRHOSSEINI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

December 2007

Specially Dedicated to

My Parent

Father, Mother, Brothers

and

Grandmother

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

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December 2007

Chairman: **Tan Chin Ping, PhD**

Faculty: **Food Science and Technology**

In the present study, the effect of main important factors affecting the headspace (HS) extraction efficiency of orange flavor compounds was investigated for the development of the solid phase microextraction (SPME) technique. The optimum HS-SPME conditions were established by using the diluted emulsion (1:100) including 15% NaCl, a 75 μm CAR/PDMS fiber at 45 ˚C for 15 min under stirring mode. Subsequently, the influence of different concentration levels of main beverage emulsion components namely Arabic gum (7-20% w/w), xanthan gum (0.1-0.5% w/w) and orange oil (6-14% w/w) on the physicochemical properties and release pattern of target volatile flavor compounds from orange beverage emulsion was studied using a three-factor central composite design (CCD). The main objective of this study was to determine the optimum level of the main emulsion components which led to the desirable response goals. The desirable response goals include: (1) the highest emulsion stability, viscosity, pseudoplastic behavior, turbidity,

cloudiness, electrophoretic mobility and largest magnitude of ζ-potential; (2) the least turbidity loss rate, conductivity, size index, average droplet size, polydispersity index, pH and flavor release content; and (3) the target value for density. The results indicated that the physicochemical properties of emulsion and release behavior of target volatile flavor compounds from orange beverage emulsion were significantly ($p < 0.05$) influenced by the main and interaction effects of the main beverage emulsion components. In most cases, the significant ($p < 0.05$) nonlinear regression models were fitted by the response surface analysis for describing the variation of physicochemical properties of emulsion. The response surface models exhibited high R^2 values (> 0.8) which had no indication of significant ($p > 0.05$) lack of fit in most cases, thus ensuring a satisfactory adjustment of the polynomial regression models fitted to the experimental data. The fitted models were accurately explained by the high variation of physicochemical properties of emulsion as a function of the proportion of main beverage emulsion components. In general, the predicted optimum for the orange beverage emulsion was 20% (w/w) Arabic gum, 0.3% (w/w) xanthan gum and 14% (w/w) orange oil. The results also indicated that CCD was found to be a very useful experimental design for investigating the variation of physicochemical properties of orange beverage emulsion and optimizing the proportion of beverage emulsion components leading to the desirable orange beverage emulsion. The results exhibited that independent variables had the least and most significant ($p < 0.05$) effects on the release of β-pinene and γ-terpinene, respectively. The effect of hydrocolloid concentration on volatile compound release was more pronounced with the negative effect of xanthan gum concentration on the overall release content. In the present study, the reduction in flavor release intensity may be explained by the different phenomena such as adsorption, complexation,

entrapment, hydrogen bonds and encapsulation of target flavor compounds induced by their interactions with Arabic gum, xanthan gum and other matrix constituents. Consequently, the effect of different concentrations of pectin (1.5, 3 and 4.5% w/w), carboxymethyl cellulose (CMC) (0.1, 0.3 and 0.5% w/w), glycerol (0.5, 1 and 1.5% w/w) and vegetable oil (2, 3 and 4% w/w) on the emulsion properties of the optimum beverage emulsion was investigated. The results indicated that these supplementary emulsion components (especially vegetable oil and pectin) could be used to modulate the physicochemical properties and release pattern of volatile flavor compounds from the orange beverage emulsion.

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

KESAN KOMPONEN EMULSI KE ATAS CIRI-CIRI FIZIKOKIMIA DAN SIFAT PEMBEBASAN BAHAN PERISA MERUAP DARI EMULSI MINUMAN OREN

Oleh

SEYED HAMED MIRHOSSEINI

Disember 2007

Pengerusi: **Tan Chin Ping, PhD**

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Dalam kajian ini, kesan faktor utama yang mempengaruhi keberkesanan pengekstrakan ruang tutupan untuk komponen perisa oren telah dikaji untuk pembentukan kaedah pengekstrakan fasa pepejal mikro-ruang kepala (HS-SPME) yang efisien. Keadaan optimum HS-SPME telah dikenalpasti dengan menggunakan emulsi yang dicairkan (1:100) termasuk 15% NaCl, 75μm CAR/PDMS fiber pada suhu 45°C dan dikacau selama 15 minit. Kesan kepekatan bahan emulsi utama yang berlainan, iaitu gum Arabik (7 – 20% w/w), gum xanthan (0.1 – 0.5% w/w) dan minyak oren (6 – 14% w/w), ke atas ciri-ciri fizikokima dan corak pembebasan sebatian perisa meruap dari emulsi minuman oren telah dikaji dengan menggunakan rekabentuk komposit pusat (CCD) tiga faktor. Objektif utama kajian ini adalah untuk menentukan takat optimum komponen emulsi utama yang dapat menghasilkan matlamat respon yang disukai. Ciri-ciri tersebut ialah: (1) nilai kestabilan emulsi, kelikatan, sifat pseudoplastik, kekeruhan, kemendungan, mobiliti elektroforesis yang

paling tinggi dan keupayaan zeta yang terbesar; (2) kadar kehilangan kekeruhan, kekonduksian, indeks saiz, purata saiz titisan, indeks kepoliserakan dan kandungan pembebasan perisa yang paling rendah; dan (3) nilai pH dan ketumpatan yang disasarkan. Keputusan kajian ini menunjukkan bahawa sifat fizikokimia emulsi dan corak pembebasan sebatian perisa meruap dari emulsi minuman oren dipengaruhi secara bererti ($p < 0.05$) oleh kesan utama dan interaksi komponen-komponen emulsi minuman utama. Bagi kebanyakan kes, model regresi tak linear secara bererti yang ditetapkan melalui analisis permukaan respons telah dapat menghuraikan variasi pada sifat-sifat fizikokimia emulsi. Model-model permukaan gerak balas telah menunjukkan nilai R^2 yang tinggi (> 0.8) dan dalam kebanyakan kes tidak menunjukkan padanan kurang tepat yang bererti ($p > 0.05$). Ini memastikan bahawa data eksperimen telah dapat menghasilkan pengubahsuaian model regresi polinomial yang memuaskan. Model-model yang dihasilkan didapati mampu menghuraikan variasi yang tinggi terhadap sifat fizikokimia emulsi secara tepat sebagai satu fungsi terhadap perkadaran komponen emulsi minuman yang utama. Secara keseluruhannya, keadaan optimum bagi emulsi minuman oren mengandungi 20% (w/w) gum Arabik, 0.3% (w/w) gum xanthan dan 14% (w/w) minyak oren. Keputusan kajian juga mendapati bahawa CCD merupakan satu rekabentuk eksperimen yang sesuai untuk mengaji perubahan variasi pada sifat-sifat fizikokimia emulsi minuman oren dan dapat mengoptimumkan perkadaran komponen emulsi minuman bagi menghasilkan sifat-sifat emulsi minuman oren yang disukai. Keputusan kajian ini juga menunjukkan bahawa pembolehubah tak bersandar masing-masing mempunyai kesan yang paling tak ketara dan paling ketara ($p < 0.05$) ke atas pembebasan β-pinene dan γ-terpinene. Kesan kepekatan hidrokoloid ke atas pembebasan bahan meruap adalah lebih ketara dengan korelasi negatif di antara

kandungan pembebasan keseluruhan dan kepekatan gum xanthan. Dalam kajian ini, pengurangan pembebasan sebatian perisa meruap kelihatan lebih disebabkan oleh kesan penyerapan keatas pengkompleksan yang membawa kepada pemerangkapan, pengkapsulan dan ikatan-ikatan hidrogen akibat interaksi di antara sebatian perisa dan juzuk matriks dan bukan hanya disebabkan oleh kesan kelikatan. Kesan pelbagai kepekatan pektin (1.5, 3 dan 4.5% w/w), carboxymethyl cellulose (CMC) (0.1, 0.3 dan 0.5% w/w), glycerol (0.5, 1.0 dan 1.5% w/w) dan minyak sayuran (2.0, 3.0 dan 4.0% w/w) pada tahap optimum emulsi minuman juga telah dikaji. Keputusan menunjukkan bahawa emulsi tambahan (terutama minyak sayuran dan pektin) boleh digunakan untuk modulat sifat-sifat fizikokimia dan pembebasan komponen meruap dari emulsi minuman berperisa oren.

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I certify that an Examination Committee has met on 27th December 2007 to conduct the final examination of Seyed Hamed Mirhosseini on his Doctor of Philosophy thesis entitled "INFLUENCE OF EMULSION COMPONENTS ON PHYSICOCHEMICAL PROPERTIES AND RELEASE OF THE VOLATILE FLAVOR COMPOUNDS FROM ORANGE BEVERAGE EMULSION" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the student be awarded the degree of Doctor of Philosophy.

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

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

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SEYED HAMED MIRHOSSEINI

Date: 29 December 2007

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