FORMATION OF AROMA COMPOUNDS BY DEGRADATION OF BETA-CAROTENE FROM CRUDE PALM OIL AND OIL PALM WASTES

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Thesis submitted in fulfilment of the requirements
for the award of the degree of
Master of Science in Industrial Chemistry

Faculty of Industrial Sciences & Technology
UNIVERSITI MALAYSIA PAHANG

JULY 2016

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LIST OF SYMBOLS

α	Alpha
β	Beta
°C	Celcius
cm	Centimetre
eV	Electron voltage
γ	Gamma
g	Gram
hr	Hour
K	Kelvin
kg	Kilogram
L	Litre
m/z	Mass-to-charge ratio
MPa	Megapascal
µL	Microliter
mg	Milligram
mL	Millilitre
mm	Millimetre
min	Minute
M	Molarity
nm	Nanometer
ppm	Parts per million
%	Percentage
s	Second

LIST OF ABBREVIATIONS

CO_2	Carbon dioxide
CPO	Crude palm oil
DCM	Dichloromethane
DHA	Dihydroactinidiolide
EFB	Empty fruit bunch
GC-FID	Gas chromatography-flame ionization detector
GC-MS	Gas chromatography-mass spectrometry
HPLC	High performance liquid chromatography
IPA	Isopropanol
LC-MS	Liquid chromatography-mass spectrometry
NaOH	Sodium hydroxide
Na_2SO_4	Sodium sulfate
OPB	Oil palm biomass
OPF	Oil palm fronds
OPT	Oil palm trunk
OPW	Oil palm wastes
PFAD	Palm fatty acid distillate
PKS	Palm kernel shells
POME	Palm oil mill effluent
PPF	Palm pressed fiber
SFE	Supercritical fluid extraction
TLC	Thin layer chromatography
UV-Vis	Ultraviolet-visible

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ABSTRACT

Aroma compounds such as α-ionone, β-ionone, β-damascenone and other important compounds can be produced via degradation of carotenoids. Extraction of natural carotenoid-derived aroma compounds directly from natural sources is expensive and uneconomical because isolation of single pure natural aroma compounds directly from plant sources are difficult and involves tedious work as it is available in a very low yield. Therefore, this research aims to produce aroma compounds by degradation of β-carotene from crude palm oil (CPO) and oil palm wastes (OPW). Palm carotene extraction process was performed by using soxhlet adsorption. The extraction process mainly aimed at extracting β-carotene high in concentration and yield. Based on the optimization of this extraction technique, soxhlet adsorption recovered highest concentration of β-carotene at 1:4 ratio of CPO: HP-20 for 1 hour isopropanol (IPA) extraction time. The concentration of β-carotene extracted was determined by using HPLC and UV-Vis standard calibration curve. CPO showed highest concentration of β-carotene under the optimised conditions which was 3770 ppm, followed by palm pressed fiber (PPF), with concentration of 1397 ppm and empty fruit bunch (EFB) yielded lowest concentration of 687 ppm. As for production of aroma compounds, optimization of degradation reaction conducted using commercial β-carotene in order to achieve optimum degradation conditions. Different sonication time, reaction time and reaction temperature was studied to ascertain the optimization of thermal degradation reaction. Effect of sonication, light and amount of sample also catalyst was studied to optimize the oxidative degradation. Based on the results obtained, the optimum condition for thermal degradation is 1 hour sonication, 5 hours of reaction and 120-130 °C. Whereas for oxidative degradation vial must be completely covered, sealed and kept at room temperature. The extracted palm carotene further was degraded using thermal and oxidative degradation under optimized reaction conditions. The oxidative degradation using extracted carotene failed to produce any aroma compounds. However, thermal degradation of extracted β-carotene from both CPO and OPW produced aroma compounds such as β-ionone, dihydroactinidiolide (DHA), D-limonene, β-ionone epoxide and 3-oxo-β-ionone. The major aroma compound with highest composition produced is DHA (45.91 %) followed by β-ionone which was analysed using GC-MS and GC-FID. Overall, this research signifies that, recovery of β-carotene by soxhlet adsorption from CPO is higher in concentration compared to OPW. Thermal degradation method is a more advantageous technique for production of aroma compounds compared to oxidative degradation. The results obtained prove that production of aroma compounds by degradation of β-carotene from CPO and OPW is viable.

ABSTRAK

Sebatian aroma seperti α-ionone, β-ionone, β-damascenone dan sebatian penting yang lain boleh dihasilkan melalui proses degradasi karotenoid. Pengekstrakan semulajadi karotenoid yang mengahsilkan sebatian aroma secara langsung daripada sumber semula jadi adalah mahal dan tidak berbaloi. Pengasingan tunggal tulen sebatian aroma asli terus daripada sumber tumbuh-tumbuhan adalah sukar dan didapati dalam hasil yang sangat rendah. Oleh itu, kajian ini bertujuan untuk menghasilkan sebatian aroma melalui degradasi β-karotena daripada minyak sawit mentah (MSM) dan bahan buangan kelapa sawit (OPW). Proses pengekstrakan karotena sawit dijalankan melalui teknik penjerapan soxhlet. Proses pengekstrakan bertujuan untuk mengekstrak β-karotena yang tinggi dalam kepekatan dan hasil. Berdasarkan pengoptimuman teknik pengekstrakan ini, penjerapan soxhlet menghasilkan kepekatan tertinggi β-karotena dalam nisbah 1: 4 untuk MSM: HP-20 dalam tempoh 1 jam masa pengekstrakan untuk menghasilkan isopropanol (IPA) . Kepekatan β-karotena yang diekstrak ditentukan dengan menggunakan HPLC dan keluk penentukuran standard UV-Vis. MSM menunjukkan kepekatan tertinggi β-karotena di bawah keadaan optimum pada 3770 ppm, diikuti oleh gentian kelapa ditekan (PPF), dengan kepekatan 1397 ppm dan tandan buah kosong (EFB) menghasilkan kepekatan terendah pada 687 ppm. Bagi pengeluaran sebatian aroma, pengoptimuman reaksi degradasi dijalankan dengan menggunakan komersial β-karotena untuk mencapai tahap optimum degradasi. Kesan sonikasi, cahaya dan pemangkin telah dikaji untuk mengenal pasti degradasi oksidatif. Berdasarkan keputusan yang diperolehi, keadaan optimum untuk degradasi haba adalah 1 jam sonikasi, 5 jam untuk menghasilkan tindak balas dan 120-130 ° C, manakala bagi degradasi oksidatif, botol mesti ditutup sepenuhnya, dimeterai dan disimpan pada suhu bilik. Karotena sawit yang diekstrak didegradasi menggunakan degradasi terma dan oksidatif di bawah keadaan tindak balas yang dioptimumkan. Degradasi oksidatif menggunakan karotena yang diekstrak, gagal menghasilkan sebatian aroma.. Walau bagaimanapun, penurunan haba diekstrak β-karotena daripada kedua-dua MSM dan OPW menghasilkan sebatian aroma seperti β-ionone, dihydroactinidiolide (DHA), D-limonene, β-ionone epoksida dan 3-oxo-β-ionone. Sebatian aroma utama dengan komposisi tertinggi yang terhasil adalah DHA (45,91%) diikuti dengan β-ionone yang dianalisa menggunakan GC-MS dan GC-FID. Secara keseluruhan, kajian ini menandakan bahawa, pemulihan β-karotena melalui penjerapan soxhlet dari MSM lebih tinggi dalam kepekatan berbanding OPW.Kaedah degradasi haba adalah satu teknik yang lebih berfaedah untuk pengeluaran sebatian aroma berbanding degradasi oksidatif. Keputusan yang diperolehi membuktikan bahawa pengeluaran sebatian aroma oleh degradasi β-karotena daripada MSM dan OPW lebih produktif.

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