ESTERIFICATION OF FREE FATTY ACID IN USED COOKING OIL USING SULPHONATED HYPERCROSSLINKED EXCHANGE RESIN AS CATALYST

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Master

UNIVERSITI MALAYSIA PAHANG

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Faculty of Chemical and Natural Resources Engineering

UNIVERSITI MALAYSIA PAHANG

JUNE 2017

ACKNOWLEDGEMENTS

In the name of Allah S.W. T. the Most Beneficent and the Most Merciful. The deepest sense of gratitude to the Almighty for the strength and ability to complete my study successfully. Infinite thanks we brace upon Him for the blessing, protecting and guiding me throughout this period and for always sent me so many good people in finishing my study.

I would like to take this opportunity to extent my deepest gratitude to all the parties involved in this research. First of all, I would like to express my special appreciation to my supervisor, Dr. Sumaiya Bt Zainal Abidin for her continuous support, guidance, encouragement, knowledge, patience, and advices throughout my master's journey. I also owe my sincere gratitude and warm thanks to my co-supervisor, Dr. Norhayati Bt Abdullah for her help throughout the work. I have learned a lot from both of them and without their help, I could not have finished my study successfully.

Special thanks to all the technical staffs from the Laboratory of Faculty of Chemical and Natural Resources Engineering who have helped me for the lab work anytime I ask for help. This work would not have been possible without their help and support. I would also like to express my appreciation to all my friends especially to Nur Nabillah Bt Mohd Arif and Norazimah Bt Harun for their supports, trust, and insightful suggestions throughout the entire year. Thanks for the enjoyable journey which made the life as a master's student easier.

The deepest gratitude goes to my beloved mother, Mrs. Azizul Bt Aris, my brothers and sister, Shahidan, Samsul, and Ashikin for their support, love, and prayers. Thank you for being by my side through thick and thin. Never to be left my faith pray for my late father, Allahyarham Mr. Roslan Bin Ariffin, may be peace upon His blessing.

Lastly, my deepest gratitude to UMP and Ministry of High Education (MOHE), Malaysia for the financial support throughout this research work via Graduate Research Scheme (GRS 1403159), RDU 130311 and MyMaster sponsorship.

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

N_{acid}	Acid number
A	Volume of KOH, volume of the titration solvent
B	Volume corresponding to the blank titration
W	Weight of the sample
M	Concentration of KOH
N	Normality of the titration solution
AR_i	Area of the ith component
A_i	Area of the ith component
A_{is}	Area of the internal standard
CR_i	Concentration ratio of the ith component
C_i	Concentration of the ith component
C_{is}	Concentration of the internal standard
RF_i	Response factor of the ith component
AR_i	Area ratio of the ith component
CR_i	Concentration ratio of the ith component
C_{ij}	Concentration ratio of the ith component of jth sample
AR_{ij}	Area ratio of the ith component of the sample jth
RF_i	Response factor of the ith component
V	Volume of water
m_{H_2O}	Mass of water
ρ_{H_2O}	Density of water
ρ_L	Density of liquid sample
m_L	Mass of liquid sample
m_c	Mass of catalyst
v	Volume of the sample
C_{NaOH}	Concentration of NaOH
C_{HCl}	Concentration of HCl
V_{HCl}	Volume of HCl
V_{NaOH}	Volume of NaOH
$C_{NaOH\ adsorbed}$	Concentration of NaOH adsorbed

$C_{NaOH\ initial}$	Initial concentration of NaOH
$C_{NaOH\ final}$	Final concentration of NaOH
q_{NaOH}	Sodium adsorption capacity
$V_{initial}$	Initial volume
C_{FFA}	Conversion of FFA
C_{FO}	Initial concentration of FFA
C_F	Concentration of FFA at any time
Mw_{tri}	Molecular weight of oil
Mw_{FFA}	Molecular weight of free fatty acid
Mw_{water}	Molecular weight of water

LIST OF ABBREVIATIONS

BET	Brunauer-Emmelt-Teller
BSI	British Standard Institution
FAME	Fatty acid methyl ester
FESEM	Field emission scanning electron microscopy
FESEM	Field emission gun-scanning electron microscope
EDX	Energy dispersion X-ray
FFA	Free fatty acids
FID	Flame ionisation detector
FTIR	Fourier transform-infra red
GC-MS	Gas chromatography-mass spectrometry
IEC	Ion exchange capacity
IER	Ion exchange resin
KBr	Potassium bromide
NA	Not available
NAD	Non-aqueous dispersion
OVAAT	One variable at a time
PP	Precipitation polymerisation
PSD	Particle size distribution
RF	Response factor
RPM	Revolution per minute
SEM	Scanning electron microscopy
SHER	Sulphonated hypercrosslinked exchange resin
SPE	Solid phase extraction
SUCO	Simulated used cooking oil
TGA	Thermogravimetric
UCO	Used Cooking Oil
XRF	X-ray Fluorescence

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ABSTRAK

Proses pengesteran adalah proses rawatan yang digunakan untuk mengurangkan asid lemak bebas yang terkandung di dalam minyak berasid. Kebiasaanya, asid lemak bebas yang terkandung di dalam minyak akan menyebabkan pembentukkan sabun dan mengurangkan jumlah asid lemak metil ester yang dihasilkan. Tujuan utama kajian ini adalah untuk mengkaji proses pengesteran asid lemak bebas yang terkandung di dalam minyak masak terpakai buatan menggunakan hypercrosslik resin yang disulfonatkan (SHER) sebagai pemangkin. Kajian ini dibahagikan kepada dua peringkat kajian yang penting. Peringkat pertama membincangkan proses penyediaan SHER melalui teknik sebaran kering diikuti dengan proses sulfonasi. Kesan kepekatan monomer/co-monomer, kepekatan crosslinker, nisbah pemangkin asid Lewis dan reagen sulfonasi ke atas ciri-ciri fizikokimia SHER telah dikaji untuk menghasilkan pemangkin yang terbaik. Pada peringkat ini, pemangkin yang terbaik telah berjaya dihasilkan dengan menggunakan 80% stirena (St), 20% vinilbenzyl klorida (VBC), 1wt% ethylina glikol dimethacrylate (EGDMA), 1:1 nisbah molar $FeCl_3$: CH_2Cl dan 4wt% asid sulfurik. SHER yang dihasilkan mempunyai liang permukaan yang tinggi dengan luas permukaan sebanyak 836 m^2 g^{-1}. Ia juga mempunyai kebolehan untuk bertahan sehingga suhu ke 398°C dan mempunyai keasidan sebanyak 5.1 mmol g^{-1}. Peringkat kedua memfokuskan prestasi SHER di dalam proses pengesteran asid lemak bebas menggunakan minyak masak terpakai buatan sebagai bahan mentah. Proses pengesteran telah dijalankan di dalam sistem kelompok dan beberapa parameter seperti muatan pemangkin, suhu dan nisbah molar telah dikaji. SHER telah berjaya memangkin tindak balas tersebut dan telah mencapai 97% penukaran asid lemak bebas pada kadar kacauan 150 rpm, 5wt% muatan pemangkin, 60°C suhu tindak balas dan 12:1 nisbah molar metanol (MeOH) kepada minyak masak terpakai buatan. Semasa proses guna semula dijalankan, lebih kurang 40% aktiviti pemangkin telah berkurang selepas digunakan sebanyak lima kali. Pengurangan ini adalah disebabkan oleh liang pemangkin telah tersumbat semasa tindak balas berlaku dan telah menghalang reaktan untuk memasuki kawasan yang aktif. Keberkesanan SHER telah dibandingkan dengan beberapa komersial pemangkin (Diaion RCP145H, PK228LH and SK1BH) dan eksperimen tersebut telah dijalankan pada keadaan tindak balas yang sama. Daripada keputusan yang diperoleh, SHER telah menunjukkan penukaran dan kadar tindak balas yang paling tinggi berbanding dengan pemangkin-pemangkin lain. Prestasi SHER yang cemerlang adalah disebabkan luas permukaan, isipadu liang, kandungan sulfur dan keasidan yang tinggi.

ABSTRACT

Esterification reaction is a pretreatment method used to reduce the free fatty acids (FFA) content in acidified oil. Normally, the presence of high FFA content contributes to saponification reaction and thus, decreases the yield of fatty acid methyl ester (FAME) produced. The usage of ion exchange resin (IER) as a catalyst has been widely used to reduce the FFA content because they can catalyse the esterification reaction under mild conditions. However, current IER are having low acidic acid sites, moderate surface area and low thermal stability. This study focuses on the esterification process of FFA in simulated used cooking oil (SUCO) using self-synthesised sulphonated hypercrosslinked exchange resin (SHER) as catalyst. This study was divided into two stages. The first stage focused on the synthesis and characterisation of SHER, prepared via non-aqueous dispersion (NAD) technique followed by sulphonation process. The effect of monomer/co-monomer concentration, crosslinker concentration, Lewis acid catalyst ratio, and sulphonation reagent on the physicochemical characterisation of the resin were investigated to produce the best catalyst beads. From this stage, the best SHER beads has successfully developed at the condition of 80% of styrene (St), 20% of vinylbenzyl chloride (VBC), 1wt% of ethylene glycol dimethacrylate (EGDMA), 1:1 of molar ratio of $FeCl_3$: CH_2Cl, and 4wt% of sulphuric acid. The newly developed SHER possesses a high surface porosity with approximately 836 m^2 g^{-1} surface area. It has the ability to withstand high temperature up to 398°C with the acidity values at 5.1 mmol g^{-1}. The second stage concentrated on the performance of SHER in the esterification of FFA using SUCO as a feedstock. The esterification reaction was carried out in a batch system and the effect of various parameters such as catalyst loading, temperature, and molar ratio were investigated. SHER had successfully catalysed the reaction and achieved 97% of FFA conversion at 150 rpm, 5wt% of catalyst loading, 60°C, and 12:1 of methanol (MeOH) to SUCO molar ratio. During reusability study, the catalyst activity decreased for about 40% after five cycles of reaction. The decrease in FFA conversion was due to the pore blockage during the reaction and hence, blocked the reactants from accessing the active sites. The performance of SHER was compared with selected commercial resins (i.e., Diaion RCP145H, Diaion PK228LH, and Diaion SK1BH) and the experiments were conducted at the same reaction conditions. SHER showed the highest conversion and reaction rate compared to other catalysts. The excellence performance of SHER was due to the high amount of surface area, pore volume, sulphur content, and acidity owned by the SHER.

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