

SYNTHETIC RADICAL ADDITION  
OF METHYLACRYLIC ACID TO METHYL OLEATE

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## Catalytic radical addition of methyl isobutyl ketone to methyl oleate.



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CATALYTIC RADICAL ADDITIONS  
OF METHYL ISOBUTYL KETONE TO METHYL OLEATE

BY

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**PENGAKUAN DAN PENGESAHAN LAPORAN PROJEK PENYELIDIKAN**

Adalah ini di akui bahawa laporan penyelidikan bertajuk: Catalytic Radical Addition of Methyl Isobutyl Ketone to Methyl Oleate oleh Ong Yik Seng, No. Matrik UK 6805 telah diperiksa dan semua pembetulan yang disarankan telah dilakukan. Laporan ini dikemukakan kepada Jabatan Sains Kimia sebagai memenuhi sebahagian daripada keperluan memperolehi Ijazah Sarjana Muda Sains – Kimia Analisis dan Persekutaran. Fakulti Sains dan Teknologi, Kolej Universiti Sains dan Teknologi Malaysia.

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AOCS	American Oil Chemists' Society
Co(NO <sub>3</sub> ) <sub>2</sub>	Cobalt (II) Nitrate
Co(OAc) <sub>2</sub>	Cobalt (II) Acetate
DSC	Differential Scanning Calorimetry
FTIR	Fourier Transform-Infrared
GC-MS	Gas Chromatography-Mass Spectroscopy
IR	Infrared
IV	Iodine Value
KBr	Potassium Bromide
KI	Potassium Iodide
MEOL	Methyl Oleate
MIBK	Methyl Isobutyl Ketone
Mn(OAc) <sub>2</sub>	Manganese (II) Acetate
Mn(OAc) <sub>3</sub>	Manganese (III) Acetate
Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	Sodium Thiosulphate
N <sub>2</sub>	Nitrogen
O <sub>2</sub>	Oxygen
rpm	round per minute
TGA	Thermogravimetry analysis
UV-vis	Ultraviolet-visible

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## **ABSTRACT**

Palm oil is one of the major commodities of Malaysia. Since it comprise of high degree in unsaturated fatty acid such as Oleic acid (40%) and Linoleic acid (10%), it is very susceptible to experience oxidations which resulted in the formation of undesirable toxic compounds. This in turns, make the application of palm oil in the food and nonfood industries such as lubricants and hydraulic fluids to be limited. Transesterification process in transforming triacylglycerol to methyl esters could also reduce the viscosity and it would not be suitable for the lubrication purpose. Hence, addition reactions of Methyl Isobutyl Ketone (MIBK) to methyl oleate (MEOL) were suggested in order to improve the oxidation stability of the unsaturated methyl esters and furthermore increasing the viscosity of the MEOL. It has been reported that,  $Mn(OAc)_3$  was found to be a good compound for promoting radical addition of enolizable compounds to unsaturated fatty acid methyl esters. However, due to its stoichiometric requirements and the cost effectiveness, this kind of catalyst is not attractive to the industries. Hence, a new breed of combination catalyst,  $Mn(OAc)_2$  with Co(II) under  $O_2$  atmosphere, was being used to study it's effectiveness against the regular catalyst of  $Mn(OAc)_3$  in this type addition reactions. The resulted ketonyl products were then characterized using iodine value test, infrared, UV-Vis spectroscopic techniques, TGA and viscosity. The GC-MS techniques were used for structure elucidation of the products. Results in the analyses of ketonyl products made confirmed that the new combination catalyst was more effective than using  $Mn(OAc)_3$  alone as a radical promoter.

## **TINDAK BALAS PENAMBAHAN RADIKAL BERMANGKIN METIL ISOBUTIL KETON KEPADA METIL OLEATE**

### **ABSTRAK**

Minyak sawit merupakan salah satu barang dagangan utama Malaysia. Oleh kerana ia mengadungi darjah asid lemak tak tepu yang tinggi seperti asid oleik (40%) dan asid Linoleik (10%), maka mudah mengalami pengoksidaan dan membentuk sebatian toksik. Oleh itu, kegunaan minyak sawit dalam industri makanan dan bukan makanan adalah amat terhad dalam penghasilan minyak pelincir dan cecair hidrolik. Penukaran triasilglicerol kepada metil ester melalui proses transesterifikasi akan menurunkan kelikatannya dan menyebabkan tidak sesuai digunakan dalam industri pelinciran. Oleh itu, proses penambahan Metil Isobutil Keton kepada Metil Oleate dicadangkan untuk meningkatkan kestabilan pengoksidaan metil ester tak tepu serta juga meningkatkan kelikatan Metil Oleate. Menurut kajian yang telah dilaporkan,  $Mn(OAc)_3$  didapati berkesan untuk menjanakan radikal dari sebatian enolan seterusnya bertindak balas dengan metil ester tak tepu. Walaubagaimanapun,  $Mn(OAc)_3$  adalah mahal dan stoikiometri diperlukan dalam proses penambahan, maka, ia tidak menarik minat pihak industri. Dalam pada itu, satu sistem mangkin baru iaitu gabungan antara  $Mn(OAc)_2$  dengan Co(II) dalam kehadiran  $O_2$  dicadangkan untuk membandingkan dengan keberkesanan  $Mn(OAc)_3$  dalam tindak balas penambahan radikal ini. Produk ketonil yang terhasil telah dianalisis dan diuji dengan ujian nilai iodin, spektroskopi infra-merah, spektroskopi UV-Vis, termogravimetri analisis (TGA) dan ujian kelikatan. Manakala kaedah GC-MS digunakan untuk memisahkan serta menentukan jenis ketonil produk yang terhasil. Keseluruhananya, analisis terhadap produk ketonil telah membuktikan bahawa sistem gabungan mangkin tersebut adalah lebih berkesan dalam pemangkinan radikal berbanding  $Mn(OAc)_3$  sahaja.