

**PREPARATION AND CHARACTERISATION OF MgO NANOPARTICLE
INCORPORATED BIORESIN USING PALM OIL**

**GAN WEI TENG
(SUPERVISOR: PM DR MD MAKSUDUR RAHMAN KHAN)**

**BACHELOR OF CHEMICAL ENGINEERING
UNIVERSITI MALAYSIA PAHANG**

**PREPARATION AND CHARACTERISATION OF MgO NANOPARTICLE
INCORPORATED BIORESIN USING PALM OIL**

GAN WEI TENG
(SUPERVISOR: PM DR MD MAKSUDUR RAHMAN KHAN)

Thesis is submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering

Faculty of Chemical & Natural Resources Engineering
UNIVERSITI MALAYSIA PAHANG

JANUARY 2017

SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering.

Signature :
Name of main supervisor : PM DR MD MAKSUDUR RAHMAN KHAN
Position : ASSOCIATE PROFESSOR
Date : 10 JANUARY 2017

STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

Signature :
Name : GAN WEI TENG
ID Number : KA13052
Date : 10 JANUARY 2017

DEDICATION

*Highest gratitude to
my supervisor, my family members and my friends
for all your care, support and trust on me.*

*Special dedication to
Faculty of Chemical Engineering and Natural Resources of
University Malaysia Pahang
on providing all the related environment
and appropriate equipment
on finishing my research.*

ACKNOWLEDGEMENT

First and foremost, I would like to dedicate the most sincere gratitude to my supervisor, PM Dr Maksudur Rahman Khan for his guidance throughout the whole year of my research by for giving insightful comments and suggestions. He had always encouraged me and never failed to share his knowledge. I would also like to thank to Ong Huei Ruey, a PHD student of my supervisor who gives me hands on guidance during my research. He is always helpful and kindly shares his experience with me. I would like to thank you for your never ending support during my tenure as research student under your guidance, for giving insightful comments and suggestions.

Next, I would like to thank the Faculty of Chemical and Natural Resources of University Malaysia Pahang in providing me a superb environment of study and learning throughout the research. I am also indebted to the staff of chemical laboratory, Mr Syahrul, Mr Hairul, Mr Hafiz, Mr Najib, Mr Zulhabri and other technical staff who help me in many ways.

Last but not least, I would also like to express my gratitude towards my family members and friends for their support and encouragement which helps me in completing this project.

TABLE OF CONTENTS

SUPERVISOR’S DECLARATION II

STUDENT’S DECLARATION III

DEDICATION IV

ACKNOWLEDGEMENT V

ABSTRACT VI

ABSTRAK VII

LIST OF TABLES XI

LIST OF FIGURES XII

LIST OF SYMBOLS XIII

LIST OF ABBREVIATIONS XIV

CHAPTER 1 INTRODUCTION 1

 1.1 Background 1

 1.2 Motivation 2

 1.3 Problem statement 3

 1.4 Objectives 3

 1.5 Scopes of study 4

CHAPTER 2 LITERATURE REVIEW 5

 2.1 Introduction to alkyd resin 5

 2.2 Classification of alkyd 6

 2.3 Raw materials 9

 2.3.1 Oils 9

 2.3.2 Polybasic acids 10

 2.3.3 Polyols 11

 2.4 Manufacture of alkyd resins 14

2.4.1	Monoglyceride process	14
2.4.2	Fatty acid process.....	15
2.4.3	Alcoholysis	16
2.4.4	Polyesterification	16
2.4.5	Catalysts used in alkyd resin synthesis	24
2.4.6	Incorporation of Nanoparticles	25
2.5	Summary	27
CHAPTER 3 METHODOLOGY		28
3.1	Materials.....	28
3.2	Research algorithms	29
3.3	Preparation of MgO nano sols.....	30
3.4	Preparation of alkyd resins.....	30
3.5	Instrumentation and Characterisation of MgO nano sols.....	32
3.5.1	X-ray powder diffraction (XRD)	32
3.6	Instrumentation and Characterisation of Monoglyceride formation	32
3.6.1	High performance liquid chromatography (HPLC).....	32
3.7	Characterisation of alkyd resin.....	33
3.7.1	Acid value (AV).....	33
3.7.2	Average degree of polymerisation	33
3.7.3	Fourier Transform Infrared (FTIR).....	34
3.7.4	Nuclear magnetic resonance analysis (NMR).....	34
3.7.5	Antimicrobial test.....	35
CHAPTER 4 RESULT AND DISCUSSION.....		36
4.1	Preparation and characterisation of MgO sol.....	36
4.1.1	X-ray diffraction technique (XRD).....	37

4.2	Preparation and characterisation of MgO incorporated alkyd resin.....	37
4.2.1	Effect of different catalytic systems on Alcoholysis	38
4.2.2	Formation of alkyd resin.....	43
CHAPTER 5	CONCLUSION AND RECOMMENDATION	52
5.1	Conclusion.....	52
5.2	Recommendation.....	52
REFERENCE.....		54
APPENDIX.....		59

LIST OF TABLES

Table 2.1 : Properties of alkyd resin based on oil length (Koleske,1995).....	6
Table 2.2 : Classification of Alkyd Resins Based on Length of Oil (Aghaie et al.,2012)..	7
Table 2.3 Polybasic acids commonly used to synthesis alkyd resin.....	10
Table 2.4 : Polyols commonly used to synthesis alkyd resin	12
Table 2.5 : Optimization of reaction parameters of alkyd preparation by alcoholysis- polyesterification process through fusion method	19
Table 4.1 : Antimicrobial activity of MgO incorporated alkyd resin	50

LIST OF FIGURES

Figure 2.1 : Properties to be expected from the alkyd of different oil length and iodine value (Patton,1962).....	8
Figure 2.2 : Reaction pathway of two step method (alcoholysis followed by polyesterification) (Ong et al.,2015).....	14
Figure 2.3 : Process diagram of alkyd resin preparation by fusion method (Nanvae, Yahya & Gan, 2013).....	18
Figure 2.4 : Process diagram of alkyd resin preparation by solvent method (Sonntag, 1979).....	23
Figure 3.1 : Flowchart of alkyd resin preparation.....	29
Figure 3.2 : Process diagram of alkyd resin preparation by fusion method (Nanvae et al.,2013).....	31
Figure 4.1 : MgO nano sol at different concentration.....	36
Figure 4.2 : XRD of Magnesium Oxide.....	37
Figure 4.3 : Effect of different catalytic systems on alcoholysis at 240°C.....	38
Figure 4.4 : Effect of different MgO catalysed systems on selectivity of MG at 240°C..	40
Figure 4.5 : Effect of different MgO catalysed systems on TG conversion at 240°C.....	40
Figure 4.6 : Effect of different MgO catalysed systems on MG yield at 240°C.....	41
Figure 4.7 : Selectivity versus TG conversion at 240°C.....	41
Figure 4.8 : Plot of acid value and DP value against reaction time for different catalytic systems during polyesterification at 240°C. Table of initial reaction rates was shown inside the figure.....	43
Figure 4.9 : Effect of temperature on 0.04wt% MgO catalysed system.....	45
Figure 4.10 : FTIR spectra of oil, NaOH catalysed alkyd resin and MgO incorporated alkyd resin.....	46
Figure 4.11 : (a) ¹ H NMR and (b) ¹³ C NMR spectra of MgO catalysed alkyd resin.....	48

LIST OF SYMBOLS

D	Average crystalline size
λ	CuK α radiation
β	Full width at half maximum (FWHM)
θ	Braggs angle
X	Conversion of triglycerides
S	Selectivity of monoglycerides
P	Extent of reaction
C_o	Initial acid value
C_t	Acid value at time t
W_{oil}	Weight of oil
W_{alkyd}	Weight of finished alkyd
W_{H2O}	Weight of water evolved during esterification

LIST OF ABBREVIATIONS

AV	Acid value
DG	Diglycerides
DP	Degree of polymerisation
FTIR	Fourier Transform Infrared
HPLC	High performance liquid chromatography
MG	Monoglycerides
NMR	Nuclear magnetic resonance
TG	Triglycerides
XRD	X-ray diffraction