

Table of Contents

	Page Nos.
Certificates.....	ii-v
Acknowledgements.....	vi-vii
Table of Contents.....	viii-xv
List of Figures.....	xvi-xx
List of Tables.....	xxi-xxiii
List of Abbreviations.....	xxiv-xxvi
Preface.....	xxvii-xxviii
Chapter 1: Introduction.....	1-18
1.1 Introduction.....	1
1.2 Energy crisis.....	2
1.3 Environmental pollution.....	3
1.4 Uneven distribution.....	4
1.5 Renewable energy technology.....	6
1.6 Biofuel policies.....	9
1.6.1 Biofuel policy of India.....	9
1.7 Global biodiesel production.....	12
1.7.1 Biodiesel production in India.....	14
1.8 Problem statement.....	15
1.9 Objectives.....	18

Chapter 2: Literature Review.....19-37

2.1 Biodiesel.....	19
2.2 Technique for biodiesel production.....	19
2.2.1 Direct use or blending.....	20
2.2.3 Microemulsion.....	20
2.2.3 Pyrolysis or thermal cracking.....	21
2.2.4 Transesterification reaction.....	21
2.3 Raw materials for biodiesel production.....	22
2.3.1 Alcohol.....	22
2.3.2 Lipid feedstocks.....	23
2.3.2 (a) First generation feedstocks.....	23
2.3.2 (b) Second generation feedstocks.....	23
2.3.2 (c) Third generation feedstocks.....	24
2.3.3 Selection of feedstocks.....	24
2.3.3 (a) Used vegetable oil.....	25
2.3.3 (b) Kusum oil.....	27
2.4 Catalyst selection.....	28
2.5 Special reference to alumina-based catalyst.....	31

Chapter 3: Materials and Methods.....38-57

3.1 Synopsis of methodology.....	38
3.2 Materials.....	38
3.3 Synthesis of heterogeneous base catalyst.....	39
3.3.1 Synthesis by co-precipitation route.....	39

3.3.2 Synthesis by physicochemical route.....	40
3.4 Catalyst characterization.....	40
3.4.1 Thermal characterization (TG-DTA).....	40
3.4.2 X-ray diffraction (XRD).....	41
3.4.3 Fourier transform infrared spectroscopy (FTIR).....	42
3.4.4 Scanning electron microscopy- Energy dispersive X-ray spectroscopy (SEM-EDX).....	43
3.4.5 Textural characterization (BET-BJH).....	44
3.4.6 Basic strength and amount determination.....	44
3.5 Biodiesel synthesis.....	45
3.5.1 Biodiesel synthesis from used vegetable oil.....	45
3.5.2 Biodiesel synthesis from kusum oil.....	46
3.5.2 (a) Degumming process.....	46
3.5.2 (b) Esterification reaction.....	47
3.5.2 (c) Transesterification reaction.....	47
3.5.3 Optimization Study.....	48
3.5.3 (a) Effect of methanol: oil molar ratio.....	48
3.5.3 (b) Effect of catalyst dose.....	48
3.5.3 (c) Effect of temperature.....	49
3.5.3 (d) Effect of stirring speed.....	49
3.5.4 Kinetics and thermodynamic studies.....	49

3.5.4	(a)	First-order		
kinetics.....		50		
3.5.4	(b)	Activation		
calculation.....		energy		
3.5.4 (c) Thermodynamic assessment.....		51		
3.5.5	Green	matrix		
formulation.....		52		
3.5.6	Reusability	and	regeneration	of
catalysts.....		53		
3.6 Biobiodiesel characterizations.....		53		
3.6.1 Nuclear magnetic resonance study.....		53		
3.6.2 Gas chromatography-mass spectroscopy (GC-MS) analysis.....		54		
3.6.3 Physicochemical characterizations.....		54		
3.6.3 (a) Acid value.....		55		
3.6.3 (b) Density.....		55		
3.6.3 (c) Kinematic viscosity.....		56		
3.6.3 (d) Calorific value.....		56		
3.6.3 (e) Flash point and fire point.....		56		
3.6.3 (f) Cloud point and pour point.....		57		
3.6.3 (g) Cetane number.....		57		

Chapter 4: Synthesis and characterization of the heterogeneous catalyst barium aluminum oxide (BaAl_2O_4) and potassium aluminum oxide ($\text{K}_2\text{Al}_2\text{O}_4$).....58-76

4.1 Introduction.....	58
4.2 Synthesis of catalyst barium aluminum oxide (BaAl_2O_4).....	58

4.3 Characterization of catalyst barium aluminum oxide (BaAl_2O_4).....	60
4.3.1 Thermal characterization (TG-DTA).....	60
4.3.2 XRD.....	60
4.3.3 FT-IR.....	62
4.3.4 HR-SEM.....	63
4.3.5 EDX.....	65
4.3.6 BET-BJH analysis.....	65
4.3.7 Basic strength and amount determination.....	66
4.4 Synthesis of catalyst potassium aluminum oxide ($\text{K}_2\text{Al}_2\text{O}_4$).....	67
4.5 Characterization of the catalyst potassium aluminum oxide ($\text{K}_2\text{Al}_2\text{O}_4$).....	68
4.5.1 Thermal characterization (TG-DTA).....	68
4.5.2 XRD.....	69
4.5.3 FT-IR.....	71
4.5.4 HR-SEM.....	72
4.5.5 EDX.....	73
4.5.6 BET-BJH analysis.....	74
4.5.7 Basic strength and amount determination.....	74
4.6. Conclusion.....	75
Chapter 5: Application of barium aluminum oxide (BaAl_2O_4) in biodiesel production from non-edible oil feedstocks (used vegetable oil and kusum oil).....	77-109
5.1	
Introduction.....	77

5.2 Biodiesel synthesis from used vegetable oil using BaAl ₂ O ₄	77
5.2.1 Optimization of biodiesel production parameters.....	77
5.2.1 (a) Effect of methanol to oil molar ratio.....	78
5.2.1 (b) Effect of catalyst dose.....	79
5.2.1 (c) Effect of reaction temperature.....	81
5.2.1 (d) Effect of stirring speed.....	81
5.2.2 Reusability.....	83
5.2.3 Kinetics and thermodynamic study.....	84
5.2.3 (a) First-order kinetics.....	84
5.2.3 (b) Activation energy calculation.....	86
5.2.3 (c) Thermodynamic study.....	87
5.2.4 Green matrix formulation.....	88
5.2.5 Characterization of synthesized biodiesel.....	88
5.2.5 (a) NMR characterization (¹ H and ¹³ C).....	88
5.2.5 (b) Compositional analysis of FAME by GC-MS.....	91

5.2.5 (c) Evaluation of fuel properties.....	92
5.3 Biodiesel synthesis from kusum oil using BaAl ₂ O ₄ catalyst.....	93
5.3.1 Optimization of biodiesel production parameters.....	93
5.3.1 (a) Effect of methanol to oil molar ratio.....	93
5.3.1 (b) Effect of catalyst dose.....	94
5.3.1 (c) Effect of reaction temperature.....	96
5.3.1 (d) Effect of stirring speed.....	97
5.3.2 Reusability.....	98
5.3.3 Kinetics and thermodynamic study.....	99
5.3.3 (a) First-order kinetics.....	100
5.3.3 (b) Activation energy calculation.....	101
5.3.3(c) Thermodynamic assessment.....	102
5.3.4 Green matrix formulation.....	103
5.3.5 Characterization of synthesized biodiesel.....	103
5.3.5 (a) NMR characterization (¹ H and ¹³ C).....	103

5.3.5 (b) Compositional analysis of FAME by GC-MS.....	106
5.3.5 (c) Evaluation of fuel properties.....	107
5.4 Conclusions.....	108
Chapter 6: Application of potassium aluminum oxide ($K_2Al_2O_4$) in biodiesel production from non-edible oil feedstocks (used vegetable oil and kusum oil).....	110-141
6.1 Introduction.....	110
6.2 Biodiesel synthesis from used vegetable oil using $K_2Al_2O_4$ catalyst.....	110
6.2.1 Optimization of biodiesel production parameters.....	110
6.2.1 (a) Effect of methanol to oil molar ratio.....	111
6.2.1 (b) Effect of catalyst dose.....	112
6.2.1 (c) Effect of reaction temperature.....	114
6.2.1 (d) Effect of stirring speed.....	114
6.2.2 Reusability.....	115
6.2.3 Kinetics and thermodynamic study.....	116
6.2.3 (a) First-order kinetics.....	117

6.2.3	(b)	Activation	energy
calculation.....		118	
6.2.3 (c) Thermodynamic study.....118			
6.2.4		Green	matrix
formulation.....		120	
6.2.5		Characterization	of synthesized
biodiesel.....		120	
6.2.5	(a)	NMR characterization	(¹ H and ¹³ C).....120
6.2.5	(b)	Compositional analysis of FAME by GC-MS.....123	
6.2.5 (c) Evaluation of fuel properties.....124			
6.3	Biodiesel synthesis from kusum oil using K ₂ Al ₂ O ₄ catalyst.....125		
6.3.1	Optimization of biodiesel production parameters.....125		
6.3.1	(a) Effect of methanol to oil molar ratio.....125		
6.3.1	(b) Effect of catalyst dose.....127		
6.3.1	(c) Effect of reaction temperature.....127		
6.3.1	(d) Effect of stirring speed.....129		
6.3.2 Reusability.....130			
6.3.3	Kinetics and thermodynamic study.....131		

6.3.3	(a)	First-order
kinetics.....		132
6.3.3	(b)	Activation
calculation.....		energy
6.3.3 (c) Thermodynamic assessment.....		133
6.3.4	Green	matrix
formulation.....		135
6.3.5	Characterization	of
biodiesel.....		synthesized
6.3.5 (a)	NMR characterization	(¹ H and
¹³ C).....		135
6.3.5(b)	Compositional analysis	of FAME by GC-
MS.....		138
6.3.5 (c)	Evaluation	of
properties.....		fuel
6.4		
Conclusions.....		140
Chapter 7: Summary.....		142-152
References.....		153-172
List of publications and conferences.....		173-174