TABLE OF CONTENT

Contents		Page No.
ABSTRACT		i–viii
LIST OF FIGURES		ix–xviii
LIST OF TABLES		xix–xxi
LIST OF SYMBOLS AND ABBREVIATIONS		xxii–xxvi
CHAPTER 1: INTRODUCTION		1-1–1-29
1.1	Background	1-1
1.2	Layered double hydroxides (LDHs) and its structure	1-3
1.3	Synthetic procedures of LDHs	1-4
1.3.1	Co-precipitation method	1-5
1.3.2	Sonochemical method	1-6
1.3.3	Urea method	1-7
1.3.4	Sol-gel method	1-7
1.3.5	Hydrothermal method	1-7
1.3.6	Ion exchange method	1-8
1.3.7	Reconstruction method	1-8
1.3.8	Induced hydrolysis method	1-8
1.4	Properties of LDHs	1-9
1.5	Applications of LDHs	1-10
1.5.1	Waste water treatment	1-10
1.5.1.1	Organic dyes and their adverse effect on environment	1-10
1.5.1.2	Phenolic compounds and their adverse effect on environment	1-12
1.5.1.3	Methods for treatments of organic pollutants	1-12
1.5.1.3.1	Adsorption	1-13
1.5.1.3.2	Photocatalysis	1-17

1.5.2	Organic transformations	1-23
1.5.2.1	Nitro-aldol condensation reaction	1-23
1.5.2.2	Microwave assisted organic reaction	1-27
1.6	Objectives of the research work	1-29
REFERENCES		1-30–1-44
CHAPTER 2: MATERIALS AND METHODS		2-1–2-19
2.1	Materials	2-1
2.2	Synthetic procedures	2-1
2.2.1	Synthesis of NiMgAl LDHs with variable composition	2-1
2.2.2	Synthesis of CuMgAl LDHs	2-2
2.2.3	Synthesis of NiAl LDHs using sonochemical method	2-3
2.2.4	Synthesis of ZnFe LDH	2-3
2.2.5	Synthesis of M^{2+}MgAl (M^{2+} = Ni^{2+} or Co^{2+}) LDHs and their derived mixed oxides	2-4
2.2.6	Synthesis of NiMgAl LDHs derived mixed oxides	2-4
2.3	Characterization techniques	2-5
2.3.1	Powder X-ray diffraction (PXRD)	2-5
2.3.2	Inductively coupled plasma optical emission spectroscopy (ICP-OES)	2-5
2.3.3	Atomic absorption spectroscopy (AAS)	2-6
2.3.4	Fourier transform infrared (FTIR) spectroscopy	2-5
2.3.5	Thermogravimetric analysis (TGA)	2-6
2.3.6	Scanning electron microscopy (SEM)	2-6
2.3.7	Transmission electron microscopy (TEM)	2-7
2.3.8	Brunauer–Emmett–Teller (BET) analysis	2-7
2.3.9	Ultraviolet-visible (UV-visible) spectroscopy	2-7
2.3.10	Diffused reflectance ultraviolet-visible (DRUV-vis) spectroscopy	2-7
2.3.11	Raman spectroscopy	2-8

2.3.12	Nuclear magnetic resonance (NMR) spectroscopy	2-8
2.4	Typical procedures of catalytic reactions	2-8
2.4.1	Adsorption study of organic dye pollutants	2-8
2.4.1.1	Adsorption of methyl orange (MO) over various ternary NiMgAl LDHs	2-8
2.4.1.2	Adsorption of various anionic and cationic dyes over CuMgAl LDHs	2-9
2.4.1.3	Adsorption of congo red (CR) over NiAl LDHs	2-10
2.4.1.4	Adsorption kinetics study	2-10
2.4.1.5	Adsorption isotherm study	2-11
2.4.1.6	Adsorption thermodynamics study	2-12
2.4.2	Photocatlytic degradation of organic polllutants	2-13
2.4.2.1	Photocatalytic degradation of phenol and its compounds using ZnFe LDH	2-13
2.4.2.2	Photocatalytic degradation of methylene blue and rhodamine B using ZnFe LDH	2-14
2.4.2.3	Reaction kinetics study	2-14
2.4.3	Nitro-aldol condensation reaction	2-15
2.4.3.1	Nitro-aldol condensation reaction under mild condition	2-15
2.4.3.2	Nitro-aldol condensation reaction under microwave condition	2-15
2.5	Computational details	2-16
REFERENCES		2-17–2-19
CHAPTER 3: BINARY AND TERNARY LAYERED DOUBLE HYDROXIDES (LDHs) FOR ADSORPTIVE REMOVAL OF VARIOUS ORGANIC DYE POLLUTANTS		3-1–3-72
3.1	Prologue	3-2
Section 3A: Efficient removal of anionic organic dye pollutants from aqueous solution by NiMgAl layered double hydroxides		3-3–3-25

3A.1	Results and discussion	3-3
3A.1.1	Characterization of LDH samples	3-3
3A.1.2	Effect of contact time, adsorbent dosage, initial dye concentration and solution pH on adsorption of methyl orange (MO) onto LDH adsorbents	3-11
3A.1.3	Adsorption kinetics	3-14
3A.1.4	Adsorption isotherm	3-17
3A.1.5	Possible mechanism of adsorption	3-20
Section 3B: Preferential adsorption of various anionic and cationic dyes from aqueous solution over ternary CuMgAl LDH		3-26–3-45
3B.1	Results and discussion	3-26
3B.1.1	Characterization	3-26
3B.1.2	Adsorption of methyl orange (MO) over various LDH adsorbents	3-30
3B.1.3	Preferential adsorption of various anionic and cationic dyes over CuMgAl4 LDH	3-32
3B.1.4	Influence of contact time, adsorbent amount, solution pH and dye concentrations	3-33
3B.1.5	Adsorption kinetics	3-37
3B.1.6	Adsorption isotherm	3-39
3B.1.7	Selective adsorption of dye from a mixture of anionic and cationic dyes	3-44
Section 3C: Adsorptive removal of congo red from aqueous solution by sonochemically synthesized NiAl layered double hydroxide		3-46–3-62
3C.1	Results and discussion	3-46
3C.1.1	Characterization of the adsorbents	3-46
3C.1.2	Adsorption experiments	3-49
3C.1.3	Effect of contact time, adsorbent amount, initial dye	3-50

concentration and solution pH on adsorption of CR
3C.1.4 Adsorption isotherms 3-53
3C.1.5 Adsorption kinetics 3-55
3C.1.6 Adsorption thermodynamics study 3-56
3C.1.7 Adsorption mechanism 3-58
REFERENCES 3-63–3-72

CHAPTER 4: PHOTOCATALYTIC DEGRADATION OF ORGANIC POLLUTANTS BY USING ZnFe LAYERED DOUBLE HYDROXIDE (LDH) 4-1–4-44

4.1 Prologue 4-2
4.2 Results and Discussion 4-3
4.2.1 Characterization of ZnFe LDH 4-3
4.2.2 Catalytic activity of ZnFe LDH 4-9

Section 4A: Photocatalytic Degradation of phenol and its derivatives using ZnFe Layered Double Hydroxide (LDH) 4-9–4-26

4A.1 Photocatalytic degradation of phenol and its compounds 4-9
4A.1.1 Optimization study 4-13
4A. 1.2 Reaction kinetics 4-15
4A.1.3 Proposed mechanism and degradation pathways 4-18
4A.2 DFT investigation of Phenol + ˙OH reaction 4-20
4A.2.1 Electronic structures and vibrational frequency analysis 4-20
4A.3 Recyclability of ZnFe LDH 4-25

Section 4B: Photocatalytic degradation of methylene blue and rhodamine B using ZnFe layered double hydroxide 4-27–4-37

4B.1 Photocatalytic degradation of MB and RhB 4-27
4B.1.1 Degradation of MB and RhB under UV light irradiation 4-27
4B.1.2 Degradation of MB and RhB under visible light irradiation 4-29

4B.1.3	Optimization study	4-30
4B.1.4	Reaction kinetics	4-33
4B.1.5	Proposed degradation mechanism	4-34
4B.2	Reusability test of the photocatalyst	4-36
REFERENCES		4-38–4-44
CHAPTER 5: LAYERED DOUBLE HYDROXIDE (LDHs) DERIVED MIXED OXIDES FOR SOLVENT FREE NITRO-ALDOL CONDENSATION REACTION		5-1–5-34
5.1	Prologue	5-1
Section 5A: Synthesis of high surface area mixed metal oxide from NiMgAl LDH precursor for nitro-aldol condensation reaction		5-3–5-16
5A.1	Results and discussion	5-3
5A.1.1	Characterization	5-3
5A.1.2	Catalytic performance	5-8
Section 5B: Effect of Ni on structural properties of NiMgAl-LDH derived mixed oxides and their catalytic activity for nitro-aldol condensation reaction		5-17–5-28
5B.1	Results and discussion	5-17
5B.1.1	Characterization	5-17
5B.1.2	Catalytic performance	5-22
REFERENCES		5-29–5-34
CHAPTER 6: CONCLUSIONS AND FUTURE SCOPES		6-1–6-6
6.1	Concluding remarks and outlook	6-1
6.2	Future scopes	6-6
LIST OF PUBLICATIONS		xxvii
LIST OF CONFERENCES ATTENDED		xxviii
APPENDIX		A-1–A-5