CONTENTS

SECTIONS

PAGE NO.

LIST OF TABLES LIST OF FIGURES ABSTRACT

CHAPTER I	(GENERAL INTRODUCTION	1
	1.0	Introduction to coal	2-7
	1.1	Major types/forms of coal found in Margherita	7-8
	1.2	Coal resources and its distribution in NER India	8-9
	1.3	Present scenario of coal utilization	10-12
	1.4	Geological settings of Ledo opencast mining area	13-14
	1.5	Generation of acid mine drainage (AMD)	14-16
	1.6	Environmental consequences of AMD	16-20
	1.7	Objectives of the study	20-21
	1.8	Arrangement of chapters	21-26
	Referen	nces	27-37
CHAPTER II	I	Methods and materials	38
	2.0	Introduction	39
	2.1	Study area`	39-43
	2.2	Collection of different types samples	43
	2.2.1	Collection of coal, OB, soil and sediment	43-44
	2.2.2	Collection of mine water/mine affected	
		water samples	44
	2.3	Analysis of mine water/ mine	
		affected water	44
	2.3.1	Physico-chemical analyses of mine water /	
		Mine affected water	44
	2.3.2	Chemical analyses of mine water / mine	
		affected water	45
	2.3.2.1	ICP-OES analyses of mine water / mine	
		affected water	45
	2.3.2.2	Ion-Chromatographic analyses of mine water/	
		mine affected water	45-46
	2.4	Analytical characterizations of coal, OB,	
		Soil and sediment	46

[Seasonal variability study of acid mine drainage Formation of Ledo high-sulphur coal mine area,	
Referen		54-55
Doforan	drainage water	53
2.4.13	Study of cytotoxicity assays for acid mine	
2.4.12	Aqueous leaching of coal and overburden samples	53
2.4.11	Atomic absorption spectroscopic (AAS) analyses of coal ,OB, soil, and sediment	52-53
2 4 1 1	energy dispersed spectroscopic (HRTEM - EDS) analyses of coal, and OB	52
2.4.10	microscopy - energy dispersed spectroscopic (FESEM- EDS) analyses of coal, OB, soil, and sediment High resolution transmission electron microscopy-	52
2.4.9	OB, soil and sediment Field emission scanning electron	51-52
2.4.8	and sediment Mössbauer spectroscopic analyses of coal,	51
2.4.7	Raman spectroscopic analyses of coal, soil,	51
2.4.6	Fourier transform spectroscopic (FTIR)	50-51
2.4.5	in coal, OB, soil, and sediment X-Ray Diffraction (XRD) analyses of coal,	50 51
2.4.4	Determination of carbon, hydrogen, and nitrogen	
2.4.3	Proximate analyses of coal, OB, soil, and sediment	50
2.4.2.4	Determination of organic sulphur	50
2.4.2.3	Calculation of pyritic sulphur	50
2.4.2.1	Determination of sulphate sulphur	47-48
2.1.2	sulfur in coals	46
242	soil and sediment Determination of total and forms of	46
2.4.1	Chemical analyses of coal, OB,	

CHAPTER III	Seasonal variability study of acid mine drainage Formation of Ledo high-sulphur coal mine area,	
	Northeastern coalfield	56
3.0	Introduction	57-58
3.1	Methods and materials	58
3.1.1	Sampling and sampling area	58
3.2	Analysis of mine water/mine affected water	
	Samples / other solid samples	59
3.3	Cytotoxicity assays in AMD affected water	59-60
3.4	Polycyclic aromatic hydrocarbon (PAH) analyses	
	of AMD water	60
3.5	Results and discussions	60

Physico-chemical parameters of acid mine	
water collected from different sites	60-66
Types of sulphur in raw coals	66-67
Potentially hazardous elements (PHEs) in	
mine water samples	68-74
Ions present in AMD water	75-84
Study of cytotoxicity assay of AMD water	84-85
PAHs contents in AMD waters of coalfield	85-87
nces	88-91
Aqueous leaching of high sulphur coal	
and overburden from Ledo colliery and	
their physico-chemical and elemental analysis	92
Introduction	93-95
Experimental section	96
Sampling and aqueous leaching	96-97
Physico-chemical analyses of aqueous leach	ates 97
Chemical analyses of aqueous leachates	97
Results and discussions	97
Physico-chemical characteristics of	57
aqueous leachates	07_00
Mine water characteristics and comparison)[-))
with aqueous leachates	99-101
Ion- chromatographic analyses of	<i>))</i> 101
aqueous leachates	101-112
Elemental analyses (ICP-OES and	101 112
AAS analyses)	113-120
XRD and FTIR analyses of leached coals	120-125
Observation from field-emission-	120 125
scanning-electron microsconic	
(FE-SEM) analyses	125-128
nces	129-134
Environmental assessment and	127 131
nano-mineralogical characterization	
of raw coal, overburden, and sediment from	
Ledo coal mining acid drainage area	135
Introduction	136-137
Methods and materials	138
Experimental sections	138
Geochemical analyses of coal OB soil	100
and sediment	138
Chemical analyses of coal, OB, soil.	100
and sediments	138
Ion chromatographic analyses of mine /	100
mine affected water	139
Results and discussions	139
	Physico-chemical parameters of acid mine water collected from different sites Types of sulphur in raw coals Potentially hazardous elements (PHEs) in mine water samples Ions present in AMD water Study of cytotoxicity assay of AMD water PAHs contents in AMD waters of coalfield nces Aqueous leaching of high sulphur coal and overburden from Ledo colliery and their physico-chemical and elemental analysis Introduction Experimental section Sampling and aqueous leaching Physico-chemical analyses of aqueous leach Chemical analyses of aqueous leachates Results and discussions Physico-chemical characteristics of aqueous leachates Mine water characteristics and comparison with aqueous leachates Ion- chromatographic analyses of aqueous leachates Elemental analyses (ICP-OES and AAS analyses) XRD and FTIR analyses of leached coals Observation from field-emission- scanning-electron microscopic (FE-SEM) analyses nces Environmental assessment and nano-mineralogical characterization of raw coal, overburden, and sediment from Ledo coal mining acid drainage area Introduction Methods and materials Experimental sections Geochemical analyses of coal, OB, soil, and sediment Chemical analyses of coal, OB, soil, and sediments Ion chromatographic analyses of mine / mine affected water Results and discussions

	5.3.1.	Geochemical characteristics coal mine water	139-140
	5.3.2	pH of soil, OB, and sediment	140-142
	5.3.3	Chemical analyses of coal, soil, OB,	
		and sediment	142-144
	5.3.4	Ion-chromatographic analyses of mine	
		water/ mine affected water	145-147
	5.3.5	XRD mineralogy of coal, OB,	
		soil, and sediment	147-151
	5.3.6	FTIR analyses of coal, OB, soil, and sediment	151-156
	5.3.7	Raman analysis of coal, overburden,	
		soil, and sediment	156-159
	5.3.8	Mössbauer spectroscopic analyses of coal, OB	,
		soil, and sediment	160-161
	5.3.9	Observation from EF-SEM and HR-TEM	
		analyses of coal and OB	162-167
	5.3.10	Mobility of elements in CMD water	168
	Referen	ices	169-173
CHAPTER VI		Remediation of mine waters /acid	
		leachates by using nano-sized limestone	174
	6.0	Introduction	175-176
	6.1	Experimental sections	176
	6.1.1	Samples	176
	6.1.2	Remedial method `1	76-177
	6.2	Lab-scale remediation study for neutralization	
		of AMD by nano-limestone	178-186
	Referen	ices	187-188
CHAPTER VII	Sur	nmary and recommendations	189-194
PUBLICATION	S]	-VI

LIST OF TABLES

TABLE NOCAPTION

PAGE NO

Table 1.1	Coal resources in NER of India	9
Table1.2	Consumption of coal in different industries (in million tons)	12
Table 2.1	Different sample points around Ledo colliery	41
Table 3.1	Physico-chemical parameters for sample	
	set S1, S2, S3, and S4	62
Table 3.2	Co-relation among physico-chemical parameters	
	of mine water in different seasons	65
Table 3.3	Forms of sulphur in coal in monsoon and in	
	non-monsoon seasons	67
Table 3.4	ICP-OES analysis of mine water samples (S1, S2)	
	in monsoon season	69
Table 3.5	ICP-OES analysis of mine water samples (S3, S4) in	
	non-monsoon season	72
Table 3.6	Ion-chromatographic analyses of cations	
	in mine water during monsoon and	
	non-monsoon seasons	76
Table 3.7	Ion-chromatographic analyses of anions in	
	mine water during monsoon and	
	non-monsoon seasons	83
Table 3.8	PAHs contents in AMD Water from Ledo	
	Colliery (µg/l)	87
Table 4.1	Physical analyses of aqueous leachates	
	at room temperature at different leaching times	100
Table 4.2	Geochemical analyses of mine water samples	101
Table 5.1	Geochemical analysis of water samples	140
Table 5.2	Determination of pH of soil, OB, and sediment	
	of Ledo colliery and nearby Tirap colliery	141
Table 5.3	Classification of soil pH range	142
Table 5.4	Proximate analysis of coal (wt %)	143
Table 5.5	CHNS and forms of sulphur analysis of coal (wt %)	143
Table 5.6	Proximate analysis of OB, soil and sediment (wt %)	144
Table 5.7	CHNS analysis of soil, sediment and overburden (wt %)	144
Table 5.8	Analysis of cations in coal mine water samples (mg/l)	146
Table 5.9	Analysis of anions in coal mine water (mg/l)	146
Table 5.10	Ion chromatographic analyses of coal, OB,	
	soil and sediment (mg/l)	147
Table 5.11	Assignments of d-values for the mineral phases	151
Table 5.12	Mössbauer parameters of Fe-components	
	observed in the samples	161
Table 6.1	Change in pH with volume of mine	
	water during neutralization	180

Table 6.2	Change in TDS with volume of mine	
	water during neutralization	180
Table 6.3	Change in EC with volume of mine water	
	during neutralization	181
Table 6.4	Percentage of elemental absorption in treated AMD	182

LIST OF FIGURES

FIGURE NO. CAPTION

PAGE NO.

Figure 1.1(a)	Anthracite	3
Figure1.1(b)	Bituminous	3
Figure1.1(c)	Subbituminous	3
Figure1.1(d)	Lignite	3
Figure 1.2	Sector wise coal consumption (2016 -2017)	12
Figure 1.3	Satellite picture of Ledo open cast mine	14
Figure 2.1.	Showing the location of Ledo colliery under study	40
Figure 2.2	Sampling sites of fresh mining [A], AMD water [B],	
	seepage water [C], and sediment with Fe(OH)3	
	stain [D] in Ledo colliery	42
Figure 2.3	Sampling sites of OB dump [E], Ledopani [F],	
	Kachanallah [G], and seepage water in Ledo colliery	
	in monsoon season [H]	43
Figure 2.4.1	Flowchart for determination of sulphate sulphur	47
Figure 2.4.2	Flowchart for determination of pyritic sulphur	49
Figure 2.4.3	Leaching apparatus used for stirring of experimental	
	samples	53
Figure 3.1	Seasonal variation of pH of mine and mine affected water	63
Figure 3.2	Seasonal variation of EC of mine and mine affected water	63
Figure 3.3	Seasonal variation of TDS of mine and mine affected water	64
Figure 3.4	Forms of sulphur in coal in monsoon and in non-monsoon	
	seasons	67
Figure 3.5	Elemental concentration of mine water samples in monsoon	-
	season (S1)	70
Figure 3.6	Elemental concentration of mine water samples in monsoon	71
E '	season (S2)	/1
Figure 5.7	Elemental concentration of mine water samples in	72
Eiguna 2.9	Flamental concentration of mine water complex in	15
Figure 5.6	non monsoon season (SA)	73
Figure 3.0	Cationic concentration of mine water in monsoon	15
I iguic 5.7	season (S1)	77
Figure 3.10	Cationic concentration of mine water in monsoon	,,
1 iguie 5.10	season (S2)	77
Figure 3.11	Cationic concentration of mine water in non-monsoon	
8	season (S3)	78
Figure 3.12	Cationic concentration of mine water in non-monsoon	
C	season (S4)	78
Figure 3.13	Anionic concentration of mine water in monsoon	
-	season (S1)	79

Figure 3.14	Anionic concentration of mine water in monsoon season (S2)	80
Figure 3.15	Anionic concentration of mine water in non- monsoon season (S3)	81
Figure 3.16	Anionic concentration of mine water in non- monsoon	01
I iguie 5.10	season (S4)	82
Figure 3.17	Water samples from mining sites induces decline in	02
1 iguie 3.17	percentage viability of (A)Normal rat muscle (L6)	
	human carcinoma viz. (B)Pancreatic (MIAPaCa2).	
	(C) Lung (A459) and (D) Liver (HenG2) cell lines	85
Figure 4.1	Schematic diagram showing the actual sampling	00
i iguio ili	locations in around Ledo colliery	96
Figure 4.2	Change in concentration (in ppm) of cations in	20
1.8010	aqueous leachates of Ledo coal (LC-20A) with	
	leaching time at 25 C	102
Figure 4.3	Change in concentration (in ppm) of cations in	
	aqueous leachates of Ledo coal (LC-60A)	
	with leaching time at 25° C	102
Figure 4.4	Change in concentration (in ppm) of cations in	
	aqueous leachates of Ledo overburden with	
	leaching time (LOB-15A) at 25° C	103
Figure 4.5	Change in concentration of cations (in ppm) in	
U	aqueous leachates of Ledo overburden (LOB-15B)	
	with leaching time at 25° C	103
Figure 4.6	Change in concentration of cations (in ppm) in	
C	aqueous leachates of Ledo coal (LC-20A)	
	with leaching time at 45° C	104
Figure 4.7	Change in concentration of cations (in ppm) in aqueous	
0	leachates of Ledo coal (LC-60A) with leaching	
	time at 45° C	104
Figure 4.8	Change in concentration of cations (in ppm) in aqueous	
	leachates of Ledo coal (LC-20A) with leaching	
	time at 65° C	105
Figure 4.9	Change in concentration of cations in aqueous leachates	
	of Ledo coal (LC-60A) with leaching time at 65 C	105
Figure 4.10	Change in concentration (in ppm) of anions in aqueous	
	leachates of Ledo coal (LC-20A) with leaching	
	time at 25 C	106
Figure 4.11	Change in concentration (in ppm) of anions in aqueous	
	leachates of Ledo coal (LC-60A) with leaching	
	time at 25° C	107
Figure 4.12	Change in concentration (in ppm) of anions in aqueous	
	leachates of Ledo overburden (LOB-15A) with leaching	
	time at 25° C	107

Figure 4.13	Change in concentration (in ppm) of anions in aqueous leachates of Ledo overburden (LOB-15B) with leaching	100
Eiguno 4 14	time at 25° C Change in concentration (in nnm) of onions in equation	108
rigule 4.14	Change in concentration (in ppin) of anions in aqueous leachest of L ado coal (L $C(20A)$) with leaching	
	time at 15 C	100
Figure 4 15	Change in concentration (in ppm) of anions in aqueous	107
1 iguie 4.15	leachates of Ledo, coal (LC-60A) with leaching	
	time at 45 C	109
Figure 4.16	Change in concentration (in ppm) of anions in aqueous	107
0	leachates of Ledo overburden (LOB-15A) with leaching	
	time at 45 C	110
Figure 4.17:	Change in concentration (in ppm) of anions in aqueous	
C	leachates of Ledo overburden (LOB-15B) with	
	leaching time at 45 C	110
Figure 4.18	Change in concentration (in ppm) of anions in aqueous	
	Leachates of Ledo coal (LC-20A) with leaching	
	time at 65 C	111
Figure 4.19	Change in concentration (in ppm) of anions in aqueous	
	leachates of Ledo coal (LC-60A) with leaching	
	time at 65 C	111
Figure 4.20	Change in concentration (in ppm) of anions in aqueous	
	Leachates of Ledo OB (LOB-15A) with leaching	
D ' 4.01	time at 65 C	112
Figure 4.21	Change in concentration (in ppm) of anions in aqueous	
	time at 65 C	110
Eiguna 1 22	Change in elemental concentration (in nnm) in equation	112
Figure 4.22	langhetes of Lado cost (LC 20A) with leasting	
	time at 25 C	114
Figure 4 23	Change in elemental concentration (in ppm) in aqueous	114
1 iguie 4.25	leachates of Ledo coal (LC-60A) with leaching	
	time at 25 C	115
Figure 4.24	Change in elemental concentration (in ppm) in aqueous	
0	Leachates of Ledo coal (LC-20A) with leaching	
	time at 45 C	116
Figure 4.25	Change in elemental concentration (in ppm) in aqueous	
C	Leachates of Ledo coal (LC-60A) with leaching	
	time at 45 C	117
Figure 4.26	Change in elemental concentration (in ppm) in aqueous	
	leachates of Ledo coal (LC-20A) with leaching	
	time at 65 C	118
Figure 4.27	Change in elemental concentration (in ppm) in aqueous	
	leachates of Ledo coal (LC-60A) with leaching	
	time at 65 C	118

Figure 4.28	Change in elemental concentration (in ppm) in aqueous leachates of Ledo coal (LC-20A) leaching for 1 hour	110
F ' (A A	at 90 C	119
Figure 4.29	Change in elemental concentration (in ppm) in aqueous	
	Leachates of Ledo coal (LC-60A) leaching for 1 hour	100
E' 4.20	at 90 C	120
Figure 4.30 \mathbf{F}	XRD pattern of leached coal and OB of Ledo colliery	121
Figure 4.31 \mathbf{F}	XRD graph for raw coal and OB of Ledo colliery	122
Figure 4.32	FT-IR spectra for raw coal and OB of Ledo colliery	123
Figure 4.33	FT-IR spectra for the leached coal and OB of the	101
	Ledo colliery	124
Figure 4.34:	SEM-EDS micrographs of raw coal samples LC-20A (a),	
	LC-60A (b) and OB samples LOB-15A (c), LOB-15B (d)	126
Figure 4.35	SEM-EDS of leached coal LCL-20A (a), LCL-60A (b)	
	and OB samples LOBL-15A (c), LOBL-15B (d)	
	(LCL: leached coal, LOBL: leached overburden)	127
Figure 5.1	XRD of coal samples of Ledo and Tirap collieries	148
Figure 5.2	XRD of OB samples of Ledo and Tirap collieries	149
Figure 5.3	XRD of soil and sediment samples of Ledo and	
	Tirap collieries	150
Figure 5.4	FTIR spectra of coal samples of Ledo and Tirap collieries	153
Figure 5.5	FTIR spectra of OB samples of Ledo and Tirap collieries	154
Figure 5.6	FTIR spectra of soil and sediment samples of Ledo and	
	Tirap collieries	155
Figure 5.7	Raman spectra of coal samples of Ledo and	
	Tirap collieries	157
Figure 5.8	Raman spectra of OB samples of Ledo and	
	Tirap collieries	158
Figure 5.9	Raman spectra of soil and sediment of Ledo and	
	Tirap collieries	159
Figure 5.10	FESEM image showing Organic Matter "Inside	
	or below" of the pyrite (LC- 20A sample)	
	(secondary electron image)	162
Figure 5.11	HR-TEM images of jarosite (a) and amorphous	
-	and crystalline mixed carbonaceous matter contain	
	hazardous elements (b) (LOB-15B)	163
Figure 5.12	FESEM of coal sample (LC-20A sample)	
-	(secondary electron image)	164
Figure 5.13	TEM image of singular nano-hematite (a) and	
-	FESEM image of Pickeringite in LC-20A (b)	165
Figure 5.14	Kaolinite (a), $NaCl + KCl$ (b) and gypsum (c)	
-	in LOB-15A sample (secondary electron image)	167
Figure 6.1	Photograph of the lab-scale AMD remediation process	177
Figure 6.2	Variation of elemental concentration in treated AMD	
-	from untreated AMD	181

Figure 6.3	Percentage of absorption of elements in treated AMD	183
Figure 6.4	HR-TEM image of limestone showing the nano	
	Particles / structure	184
Figure 6.5	XRD graph of limestone used for remediation of AMD	185