



Alkaline industrial by-product effects on plant growth in acidic-contaminated soil systems
by Joel Thomas Mehlenbacher

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Land Rehabilitation

Montana State University

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Abstract:

In some regions of the United States the near proximity of alkaline industrial byproducts to acidic-contaminated landscapes in concert with low acquisition cost, make these by-products an attractive option for soil remediation projects. The objective of this research was to determine whether acidic-contaminated soil systems amended with alkaline industrial by-products enable plant growth equivalent to that attained with a commercial grade mixture of CaCO_3 and CaO . In addition, it was determined whether an alkaline by-product dosage threshold existed, above which plant growth was impaired.

Three types of cement kiln dust (CKD), three types of lime kiln dust (LKD), and two other alkaline by-products (Dicalcium Silicate, Carbide Lime) were evaluated in this investigation. These alkaline by-products, and the standard treatment composed of a commercial grade CaCO_3/CaO mixture, were applied to metalliferous tailings (pH 1.8), and metal contaminated soil (pH 5.0) and plant growth was evaluated.

All alkaline products produced a desired soil pH (7.0 - 8.4) in the root zone during plant growth tests. Following a 111 day plant growth period with Basin Wildrye and Redtop all alkaline industrial by-products tested had plant growth equal to-or greater than- the CaCO_3/CaO mixture. This was the case in tailings and the contaminated soil for above ground plant biomass, plant height, root biomass, root depth, and number of roots at the 5 cm and 10 cm soil depths.

When alkaline by-products, including the CaCO_3/CaO mixture, were added to the Plant Growth Center soil, the greater the application rate the less was plant growth. Across the alkaline product dosage range of 0 % to 12 % (soil dry weight basis) the loss in aboveground plant biomass was 65 % for Basin Wildrye and 88 % for Redtop.

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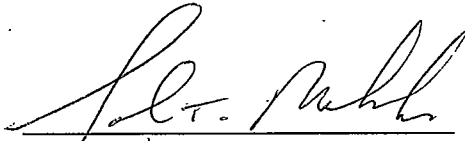
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TABLE OF CONTENTS

LIST OF TABLES.....	viii
LIST OF FIGURES.....	xxxiii
ABSTRACT.....	xxxiv
1. INTRODUCTION.....	1
Alkaline Industrial By-Products.....	2
Sources of Contamination.....	3
Efforts to Characterize Kiln Dust Physicochemical Traits.....	5
Kiln Dust use in the Soil - Plant System.....	6
Alkaline Industrial By-product Chemical Enrichment.....	9
Vanadium.....	10
Aluminum.....	11
Barium.....	12
Boron.....	12
Selenium.....	13
Zinc.....	14
Arsenic, Cadmium, Chromium, Copper, Lead, Manganese, Nickel.....	14
Objectives of Research.....	15
2. METHODS AND MATERIALS.....	17
Tailings, Contaminated Soil, Plant Growth Center Soil Bulk Sample Collection.....	17
Alkaline Industrial By-Products Bulk Sample Collection.....	17
CaCO ₃ /CaO.....	18
Greymont LKD.....	18
Tacoma LKD.....	18
MT Limestone LKD.....	19
Holnam (CH ₄) CKD.....	19
Holnam (Coal, Coke) CKD.....	19
Ash Grove CKD.....	20
Dicalcium Silicate.....	20
Carbide Lime.....	21
Analytical Methods for Soil and Alkaline Industrial By-Products.....	21
Plant Species.....	23
Soil Treatments for Tailings, Contaminated Soil and Plant Growth Center Soil.....	24
Soil Treatments for The Alkaline Product Dosage Sequence Plant Growth Tests.....	25
Plant Growth Conditions.....	26

TABLE OF CONTENTS - CONTINUED

Plant Growth Measurements.....	27
Statistical Analyses.....	28
Data Quality Control.....	28
 3. RESULTS AND DISCUSSION.....	 30
Physicochemical Characteristics of Tailings and Contaminated Soil.....	30
Opportunity Tailings.....	30
Contaminated Soil.....	32
Physicochemical Characteristics of Alkaline Industrial By-Products, Calcium Carbonate and Calcium Oxide.....	32
Alkaline Industrial By-Product Soluble Salt Content – Salinity and Sodicity.....	36
Alkaline Industrial By-Product – Chemical Enrichment.....	39
Effect of Alkaline Industrial By-Products on Plant Growth in Acidic Contaminated Soil Systems.....	46
Soil pH Control During The Plant Growth Period.....	46
Effect Of Alkaline Industrial By-Products On Initial 14 Days of Plant Growth.....	48
Effect of Alkaline Industrial By-Products After 111 Days of Plant Growth.....	52
Above Ground Plant Biomass.....	53
Above Ground Height.....	58
Below Ground Plant Biomass.....	59
Maximum Root Depth.....	60
Number of Roots.....	61
Effect Of Increasing Application Rate Of Alkaline Products On Plant Growth.....	62
Soil pH Control During the Plant Growth Period.....	63
Effect Of Increasing Alkaline Amendment Application Rate During The Initial 14 Day Plant Growth Period.....	64
Effect Of Increasing Alkaline Amendment Application Rates After a 90 Day Plant Growth Period.....	66
Correlation Of Soil pH And Electrical Conductivity To Increasing Amendment Application Rate And Loss In Plant Biomass.....	78
Conclusion.....	83
 REFERENCES CITED.....	 86
 APPENDICES.....	 93

TABLE OF CONTENTS - CONTINUED

APPENDIX A: ANALYSIS OF VARIANCE RESULTS.....	94
APPENDIX B: RAW DATA.....	216
APPENDIX C: DATA QUALITY CONTROL.....	247

LIST OF TABLES

Table	Page
1) Analytical testing methods for soil and alkaline industrial by-products.....	22
2) Characteristics of plant species utilized in this research (Munshower, 1998).....	23
3) Alkaline amendment application rates for tailings, contaminated soil, and Plant Growth Center soil.....	23
4) Alkaline amendment application rates for variable dosage rate research using the Plant Growth Center soil.....	25
5) Plant growth measurements.....	27
6) Physicochemical characteristics of tailings and contaminated soil.....	31
7) Physicochemical characteristics of alkaline industrial by-products and commercial grade lime.....	33
8) Toxicity Characteristic Leaching Procedure for Alkaline Industrial By-Products and Commercial Grade Lime.....	36
9) pH, salinity and sodicity characteristics of alkaline industrial by-products and commercial grade lime.....	37
10) Electrical conductivity of Plant Growth Center potting soil as a function of alkaline amendment application rate.....	38
11) Water soluble metal concentrations of Alkaline Industrial By-Products and Commercial Grade Lime.....	40
12) Total metal concentration of Alkaline Industrial By-Products and Commercial Grade Lime.....	41
12) Total metal concentration of Alkaline Industrial By-Products and Commercial Grade Lime - Continued.....	41
13) Water soluble concentrations of aluminum (Al) following a 111 day plant growth period in tailings, contaminated soil, and Plant Growth Center soil amended with Dicalcium Silicate.....	42

LIST OF TABLES - Continued

Table	Page
14) Total and water soluble concentrations of vanadium (V) following a 111 day plant growth period in tailings, contaminated soil, and Plant Growth Center soil amended with Greymont LKD.....	43
15) Soil pH in pots containing amended tailings, contaminated soil, and Plant Growth Center soil prior to seeding and during the plant growth period.....	45
16) Effect of different alkaline products on growth of Basin Wildrye during the initial 14 days.....	49
17) Effect of different alkaline products on growth of Redtop during the initial 14 days.....	50
18) Effect of different alkaline products on growth of Basin Wildrye 111 days after seeding.....	56
19) Effect of different alkaline products on growth of Redtop 111 days after seeding.....	57
20) Plant Growth Center soil pH ¹ prior to seeding and during the plant growth period as a function of increasing application rates of alkaline amendments.....	65
21) Effect of increasing alkaline product application rate on growth of Basin Wildrye 14 days after emergence.....	67
21) Effect of increasing alkaline product application rate on growth of Basin Wildrye 14 days after emergence - Continued.....	68
22) Effect of increasing alkaline product application rate on growth of Redtop 14 days after emergence.....	69
22) Effect of increasing alkaline product application rate on growth of Redtop 14 days after emergence - Continued.....	70
23) Effect of increasing alkaline product application rate on Basin Wildrye following a 90 day growth period.....	71

LIST OF TABLES - Continued

Table	Page
23) Effect of increasing alkaline product application rate on Basin Wildrye following a 90 day growth period - Continued.....	72
24) Effect of increasing alkaline product application rate on growth of Redtop 90 days after emergence.....	73
24) Effect of increasing alkaline product application rate on growth of Redtop 90 days after emergence - Continued.....	74
25) Soil pH following a 90 day plant growth period for Basin Wildrye as a function of different alkaline amendments and application rates.....	78
26) Soil pH following a 90 day plant growth period for Redtop as a function of different alkaline amendments and application rates.....	80
27) Linear correlation coefficient (r) between above ground plant production and soil pH at time of harvest.....	81
28) Linear correlation coefficient (r) between above ground plant production and soil Electrical Conductivity at time of harvest.....	82
29) Number of days required for emergence in tailings: Basin Wildrye.....	95
30) Number of days required for emergence in contaminated soil: Basin Wildrye.....	95
31) Number of days required for emergence in Plant Growth Center soil: Basin Wildrye.....	96
32) Number of days required for emergence in tailings: Redtop.....	96
33) Number of days required for emergence in contaminated soil: Redtop.....	97
34) Number of days required for emergence in Plant Growth Center soil: Redtop.....	97

LIST OF TABLES - Continued

Table	Page
35) Number of seedlings emerged of 15 seeds in tailings: Basin Wildrye.....	98
36) Number of seedlings emerged of 15 seeds in contaminated soil: Basin Wildrye.....	98
37) Number of seedlings emerged of 15 seeds in Plant Growth Center soil: Basin Wildrye.....	99
38) Number of seedlings emerged of 15 seeds in tailings: Redtop.....	99
39) Number of seedlings emerged of 15 seeds in contaminated soil: Redtop.....	100
40) Number of seedlings emerged of 15 seeds in Plant Growth Center soil: Redtop.....	100
41) Shoot height in tailings: Basin Wildrye.....	101
42) Shoot height in contaminated soil: Basin Wildrye.....	101
43) Shoot height in Plant Growth Center soil: Basin Wildrye.....	102
44) Shoot height in tailings: Redtop.....	102
45) Shoot height in contaminated soil: Redtop.....	103
46) Shoot height in Plant Growth Center soil: Redtop.....	103
47) Measured pH in tailings: Basin Wildrye.....	104

LIST OF TABLES - Continued

Table	Page
48) Measured pH in contaminated soil: Basin Wildrye.....	104
49) Measured pH in Plant Growth Center soil: Basin Wildrye.....	105
50) Measured pH in tailings: Redtop.....	105
51) Measured pH in contaminated soil: Redtop.....	106
52) Measured pH in Plant Growth Center soil: Redtop.....	106
53) Aboveground biomass in tailings: Basin Wildrye.....	107
54) Aboveground biomass in contaminated soil: Basin Wildrye.....	107
55) Aboveground biomass in Plant Growth Center soil: Basin Wildrye.....	108
56) Aboveground biomass in tailings: Redtop.....	108
57) Aboveground biomass in contaminated soil: Redtop.....	109
58) Aboveground biomass in Plant Growth Center soil: Redtop.....	109
59) Belowground biomass in tailings: Basin Wildrye.....	110
60) Belowground biomass in contaminated soil: Basin Wildrye.....	110

LIST OF TABLES - Continued

Table	Page
61) Belowground biomass in Plant Growth Center soil: Basin Wildrye.....	111
62) Belowground biomass in tailings: Redtop.....	111
63) Belowground biomass in contaminated soil: Redtop.....	112
64) Belowground biomass in Plant Growth Center soil: Redtop.....	112
65) Aboveground height in tailings: Basin Wildrye.....	113
66) Aboveground height in contaminated soil: Basin Wildrye.....	113
67) Aboveground height in Plant Growth Center soil: Basin Wildrye.....	114
68) Aboveground height in tailings: Redtop.....	114
69) Aboveground height in contaminated soil: Redtop.....	115
70) Aboveground height in Plant Growth Center soil: Redtop.....	115
71) Number of roots (5 cm) in tailings: Basin Wildrye.....	116
72) Number of roots (5 cm) in contaminated soil: Basin Wildrye.....	116
73) Number of roots (5 cm) in Plant Growth Center soil: Basin Wildrye.....	117

LIST OF TABLES - Continued

Table	Page
74) Number of roots (5 cm) in tailings: Redtop.....	117
75) Number of roots (5 cm) in contaminated soil: Redtop.....	118
76) Number of roots (5 cm) in Plant Growth Center soil: Redtop.....	118
77) Number of roots (10 cm) in tailings: Basin Wildrye.....	119
78) Number of roots (10 cm) in contaminated soil: Basin Wildrye.....	119
79) Number of roots (10 cm) in Plant Growth Center soil: Basin Wildrye.....	120
80) Number of roots (10 cm) in tailings: Redtop.....	120
81) Number of roots (10 cm) in contaminated soil: Redtop.....	121
82) Number of roots (10 cm) in Plant Growth Center soil: Redtop.....	121
83) Number of days required for emergence (Greymont): Basin Wildrye.....	122
84) Number of days required for emergence (Tacoma): Basin Wildrye.....	122
85) Number of days required for emergence (MT Limestone): Basin Wildrye.....	123
86) Number of days required for emergence (Holnam CH ₄): Basin Wildrye.....	123

LIST OF TABLES - Continued

Table	Page
87) Number of days required for emergence (Holnam coal,coke): Basin Wildrye.....	124
88) Number of days required for emergence (Ash Grove): Basin Wildrye.....	124
89) Number of days required for emergence (Dicalcium Silicate): Basin Wildrye.....	125
90) Number of days required for emergence (Carbide Lime): Basin Wildrye.....	125
91) Number of days required for emergence (CaCO ₃ /CaO): Basin Wildrye.....	126
92) Number of days required for emergence (Greymont): Redtop.....	126
93) Number of days required for emergence (Tacoma): Redtop.....	127
94) Number of days required for emergence (MT Limestone): Redtop.....	127
95) Number of days required for emergence (Holnam CH ₄): Redtop.....	128
96) Number of days required for emergence (Holnam coal,coke): Redtop.....	128
97) Number of days required for emergence (Ash Grove): Redtop.....	129
98) Number of days required for emergence (Dicalcium Silicate): Redtop.....	129
99) Number of days required for emergence (Carbide Lime): Redtop.....	130

LIST OF TABLES - Continued

Table	Page
100) Number of days required for emergence (CaCO_3/CaO): Redtop.....	130
101) Number of seedlings emerged of 15 seeds (Greymont): Basin Wildrye.....	131
102) Number of seedlings emerged of 15 seeds (Tacoma): Basin Wildrye.....	131
103) Number of seedlings emerged of 15 seeds (MT Limestone): Basin Wildrye.....	132
104) Number of seedlings emerged of 15 seeds (Holnam CH_4): Basin Wildrye.....	132
105) Number of seedlings emerged of 15 seeds (Holnam coal, coke): Basin Wildrye.....	133
106) Number of seedlings emerged of 15 seeds (Ash Grove): Basin Wildrye.....	133
107) Number of seedlings emerged of 15 seeds (Dicalcium Silicate): Basin Wildrye.....	134
108) Number of seedlings emerged of 15 seeds (Carbide Lime): Basin Wildrye.....	134
109) Number of seedlings emerged of 15 seeds (CaCO_3/CaO): Basin Wildrye.....	135
110) Number of seedlings emerged of 15 seeds (Greymont): Redtop.....	135
111) Number of seedlings emerged of 15 seeds (Tacoma): Redtop.....	136
112) Number of seedlings emerged of 15 seeds (MT Limestone): Redtop.....	136

LIST OF TABLES - Continued

Table	Page
113) Number of seedlings emerged of 15 seeds (Holnam CH ₄): Redtop.....	137
114) Number of seedlings emerged of 15 seeds (Holnam coal,coke): Redtop.....	137
115) Number of seedlings emerged of 15 seeds (Ash Grove): Redtop.....	138
116) Number of seedlings emerged of 15 seeds (Dicalcium Silicate): Redtop.....	138
117) Number of seedlings emerged of 15 seeds (Carbide Lime): Redtop.....	139
118) Number of seedlings emerged of 15 seeds (CaCO ₃ /CaO): Redtop.....	139
119) Shoot height (Greymont): Basin Wildrye.....	140
120) Shoot height (Tacoma): Basin Wildrye.....	140
121) Shoot height (MT Limestone): Basin Wildrye.....	141
122) Shoot height (Holnam CH ₄): Basin Wildrye.....	141
123) Shoot height (Holnam coal,coke): Basin Wildrye.....	142
124) Shoot height (Ash Grove): Basin Wildrye.....	142
125) Shoot height (Dicalcium Silicate): Basin Wildrye.....	143

LIST OF TABLES - Continued

Table	Page
126) Shoot height (Carbide Lime): Basin Wildrye.....	143
127) Shoot height (CaCO_3/CaO): Basin Wildrye.....	144
128) Shoot height (Greymont): Redtop.....	144
129) Shoot height (Tacoma): Redtop.....	145
130) Shoot height (MT Limestone): Redtop.....	145
131) Shoot height (Holnam CH_4): Redtop.....	146
132) Shoot height (Holnam coal, coke): Redtop.....	146
133) Shoot height (Ash Grove): Redtop.....	147
134) Shoot height (Dicalcium Silicate): Redtop.....	147
135) Shoot height (Carbide Lime): Redtop.....	148
136) Shoot height (CaCO_3/CaO): Redtop.....	148
137) Aboveground biomass (Greymont): Basin Wildrye.....	149
138) Aboveground biomass (Tacoma): Basin Wildrye.....	149

LIST OF TABLES - Continued

Table	Page
139) Aboveground biomass (MT Limestone): Basin Wildrye.....	150
140) Aboveground biomass (Holnam CH ₄): Basin Wildrye.....	150
141) Aboveground biomass (Holnam coal,coke): Basin Wildrye.....	151
142) Aboveground biomass (Ash Grove): Basin Wildrye.....	151
143) Aboveground biomass (Dicalcium Silicate): Basin Wildrye.....	152
144) Aboveground biomass (Carbide Lime): Basin Wildrye.....	152
145) Aboveground biomass (CaCO ₃ /CaO): Basin Wildrye.....	153
146) Aboveground biomass (Greymont): Redtop.....	153
147) Aboveground biomass (Tacoma): Redtop.....	154
148) Aboveground biomass (MT Limestone): Redtop.....	154
149) Aboveground biomass (Holnam CH ₄): Redtop.....	155
150) Aboveground biomass (Holnam coal,coke): Redtop.....	155
151) Aboveground biomass (Ash Grove): Redtop.....	156

LIST OF TABLES - Continued

Table	Page
152) Aboveground biomass (Dicalcium Silicate): Redtop.....	156
153) Aboveground biomass (Carbide Lime): Redtop.....	157
154) Aboveground biomass (CaCO_3/CaO): Redtop.....	157
155) Belowground biomass (Greymont): Basin Wildrye.....	158
156) Belowground biomass (Tacoma): Basin Wildrye.....	158
157) Belowground biomass (MT Limestone): Basin Wildrye.....	159
158) Belowground biomass (Holnam CH_4): Basin Wildrye.....	159
159) Belowground biomass (Holnam coal, coke): Basin Wildrye.....	160
160) Belowground biomass (Ash Grove): Basin Wildrye.....	160
161) Belowground biomass (Dicalcium Silicate): Basin Wildrye.....	161
162) Belowground biomass (Carbide Lime): Basin Wildrye.....	161
163) Belowground biomass (CaCO_3/CaO): Basin Wildrye.....	162
164) Belowground biomass (Greymont): Redtop.....	162

LIST OF TABLES - Continued

Table	Page
165) Belowground biomass (Tacoma): Redtop.....	163
166) Belowground biomass (MT Limestone): Redtop.....	163
167) Belowground biomass (Holnam CH ₄): Redtop.....	164
168) Belowground biomass (Holnam coal,coke): Redtop.....	164
169) Belowground biomass (Ash Grove): Redtop.....	165
170) Belowground biomass (Dicalcium Silicate): Redtop.....	165
171) Belowground biomass (Carbide Lime): Redtop.....	166
172) Belowground biomass (CaCO ₃ /CaO): Redtop.....	166
173) Aboveground height (Greymont): Basin Wildrye.....	167
174) Aboveground height (Tacoma): Basin Wildrye.....	167
175) Aboveground height (MT Limestone): Basin Wildrye.....	168
176) Aboveground height (Holnam CH ₄): Basin Wildrye.....	168
177) Aboveground height (Holnam coal,coke): Basin Wildrye.....	169

LIST OF TABLES - Continued

Table	Page
178) Aboveground height (Ash Grove): Basin Wildrye.....	169
179) Aboveground height (Dicalcium Silicate): Basin Wildrye.....	170
180) Aboveground height (Carbide Lime): Basin Wildrye.....	170
181) Aboveground height (CaCO_3/CaO): Basin Wildrye.....	171
182) Aboveground height (Greymont): Redtop.....	171
183) Aboveground height (Tacoma): Redtop.....	172
184) Aboveground height (MT Limestone): Redtop.....	172
185) Aboveground height (Holnam CH_4): Redtop.....	173
186) Aboveground height (Holnam coal,coke): Redtop.....	173
187) Aboveground height (Ash Grove): Redtop.....	174
188) Aboveground height (Dicalcium Silicate): Redtop.....	174
189) Aboveground height (Carbide Lime): Redtop.....	175
190) Aboveground height (CaCO_3/CaO): Redtop.....	175

LIST OF TABLES - Continued

Table	Page
191) Number of roots (5 cm) (Greymont): Basin Wildrye.....	176
192) Number of roots (5 cm) (Tacoma): Basin Wildrye.....	176
193) Number of roots (5 cm) (MT Limestone): Basin Wildrye.....	177
194) Number of roots (5 cm) (Holnam CH ₄): Basin Wildrye.....	177
195) Number of roots (5 cm) (Holnam coal,coke): Basin Wildrye.....	178
196) Number of roots (5 cm) (Ash Grove): Basin Wildrye.....	178
197) Number of roots (5 cm) (Dicalcium Silicate): Basin Wildrye.....	179
198) Number of roots (5 cm) (Carbide Lime): Basin Wildrye.....	179
199) Number of roots (5 cm) (CaCO ₃ /CaO): Basin Wildrye.....	180
200) Number of roots (5 cm) (Greymont): Redtop.....	180
201) Number of roots (5 cm) (Tacoma): Redtop.....	181
202) Number of roots (5 cm) (MT Limestone): Redtop.....	181
203) Number of roots (5 cm) (Holnam CH ₄): Redtop.....	182

LIST OF TABLES - Continued

Table	Page
204) Number of roots (5 cm) (Holnam coal,coke): Redtop.....	182
205) Number of roots (5 cm) (Ash Grove): Redtop.....	183
206) Number of roots (5 cm) (Dicalcium Silicate): Redtop.....	183
207) Number of roots (5 cm) (Carbide Lime): Redtop.....	184
208) Number of roots (5 cm) (CaCO ₃ /CaO): Redtop.....	184
209) Measured pH (Greymont): Basin Wildrye.....	185
210) Measured pH (Tacoma): Basin Wildrye.....	185
211) Measured pH (MT Limestone): Basin Wildrye.....	186
212) Measured pH (Holnam CH ₄): Basin Wildrye.....	186
213) Measured pH (Holnam coal,coke): Basin Wildrye.....	187
214) Measured pH (Ash Grove): Basin Wildrye.....	187
215) Measured pH (Dicalcium Silicate): Basin Wildrye.....	188
216) Measured pH (Carbide Lime): Basin Wildrye.....	188

LIST OF TABLES - Continued

Table	Page
217) Measured pH (CaCO_3/CaO): Basin Wildrye.....	189
218) Measured pH (Greymont): Redtop.....	189
219) Measured pH (Tacoma): Redtop.....	190
220) Measured pH (MT Limestone): Redtop.....	190
221) Measured pH (Holnam CH_4): Redtop.....	191
222) Measured pH (Holnam coal, coke): Redtop.....	191
223) Measured pH (Ash Grove): Redtop.....	192
224) Measured pH (Dicalcium Silicate): Redtop.....	192
225) Measured pH (Carbide Lime): Redtop.....	193
226) Measured pH (CaCO_3/CaO): Redtop.....	193
227) Measured soil pH vs. aboveground biomass (Greymont): Basin Wildrye.....	194
228) Measured soil pH vs. aboveground biomass (Tacoma): Basin Wildrye.....	195
229) Measured soil pH vs. aboveground biomass (MT Limestone): Basin Wildrye.....	195

LIST OF TABLES - Continued

Table	Page
230) Measured soil pH vs. aboveground biomass (Holnam CH ₄): Basin Wildrye.....	196
231) Measured soil pH vs. aboveground biomass (Holnam coal,coke): Basin Wildrye.....	196
232) Measured soil pH vs. aboveground biomass (Ash Grove): Basin Wildrye.....	197
233) Measured soil pH vs. aboveground biomass (Dicalcium Silicate): Basin Wildrye.....	197
234) Measured soil pH vs. aboveground biomass (Carbide Lime): Basin Wildrye.....	198
235) Measured soil pH vs. aboveground biomass (CaCO ₃ /CaO): Basin Wildrye.....	198
236) Measured soil pH vs. aboveground biomass (Greymont): Redtop.....	199
237) Measured soil pH vs. aboveground biomass (Tacoma): Redtop.....	199
238) Measured soil pH vs. aboveground biomass (MT Limestone): Redtop.....	200
239) Measured soil pH vs. aboveground biomass (Holnam CH ₄): Redtop.....	200
240) Measured soil pH vs. aboveground biomass (Holnam coal,coke): Redtop.....	201
241) Measured soil pH vs. aboveground biomass (Ash Grove): Redtop.....	201
242) Measured soil pH vs. aboveground biomass (Dicalcium Silicate): Redtop.....	202

LIST OF TABLES - Continued

Table	Page
243) Measured soil pH vs. aboveground biomass (Carbide Lime): Redtop.....	202
244) Measured soil pH vs. aboveground biomass (CaCO ₃ /CaO): Redtop.....	203
245) Measured soil EC vs. aboveground biomass (Greymont): Basin Wildrye.....	203
246) Measured soil EC vs. aboveground biomass (Tacoma): Basin Wildrye.....	204
247) Measured soil EC vs. aboveground biomass (MT Limestone): Basin Wildrye.....	204
248) Measured soil EC vs. aboveground biomass (Holnam CH ₄): Basin Wildrye.....	205
249) Measured soil EC vs. aboveground biomass (Holnam coal,coke): Basin Wildrye.....	205
250) Measured soil EC vs. aboveground biomass (Ash Grove): Basin Wildrye.....	206
251) Measured soil EC vs. aboveground biomass (Dicalcium Silicate): Basin Wildrye.....	206
252) Measured soil EC vs. aboveground biomass (Carbide Lime): Basin Wildrye.....	207
253) Measured soil EC vs. aboveground biomass (CaCO ₃ /CaO): Basin Wildrye.....	207
254) Measured soil EC vs. aboveground biomass (Greymont): Redtop.....	208
255) Measured soil EC vs. aboveground biomass (Tacoma): Redtop.....	208

LIST OF TABLES - Continued

Table	Page
256) Measured soil EC vs. aboveground biomass (MT Limestone): Redtop.....	209
257) Measured soil EC vs. aboveground biomass (Holnam CH ₄): Redtop.....	209
258) Measured soil EC vs. aboveground biomass (Holnam coal,coke): Redtop.....	210
259) Measured soil EC vs. aboveground biomass (Ash Grove): Redtop.....	210
260) Measured soil EC vs. aboveground biomass (Dicalcium Silicate): Redtop.....	211
261) Measured soil EC vs. aboveground biomass (Carbide Lime): Redtop.....	211
262) Measured soil EC vs. aboveground biomass (CaCO ₃ /CaO): Redtop.....	212
263) Measured soil pH vs. aboveground biomass (all amendments): Basin Wildrye.....	212
264) Measured soil pH vs. aboveground biomass (all amendments): Redtop.....	213
265) Aboveground biomass vs. application rate (all amendments): Basin Wildrye.....	213
266) Aboveground biomass vs. application rate (all amendments): Redtop.....	214
267) Measured soil pH vs. application rate (all amendments): Basin Wildrye.....	214
268) Measured soil pH vs. application rate (all amendments): Redtop.....	215

LIST OF TABLES - Continued

Table	Page
269) Number of days required for emergence in tailings, contaminated soil and Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	217
270) Germination (number of seedlings emerged/15 seeds, 14 day post-emergence) in tailings, contaminated soil and Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	218
271) Survival (number of seedlings survived/emerged, 14 day post-emergence) in tailings, contaminated soil and Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	219
272) Shoot height (height from soil to longest leaf (mm), 14 day post-emergence) in tailings, contaminated soil and Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	220
273) Measured pH (s.u., 70 day growth) in tailings, contaminated soil and Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	221
274) Aboveground height (height from soil to longest leaf (mm), 111 day growth) in tailings, contaminated soil and Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	222
275) Maximum root depth (length of longest root (mm), 111 day growth) in tailings, contaminated soil and Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	223
276) Root distribution (number of roots at 5 cm depth, 111 day growth)) in tailings, contaminated soil and Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	224

LIST OF TABLES - Continued

Table	Page
277) Root distribution (number of roots at 10 cm depth, 111 day growth)) in tailings, contaminated soil and Plant Growth Center soil amended with alkaline industrial by-products.....	225
278) Aboveground biomass (weight of aboveground production (g), 111 day growth) in tailings, contaminated soil and Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	226
279) Belowground biomass (weight of belowground production (g), 111 day growth) in tailings, contaminated soil and Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	227
280) Measured pH (s.u., 111 day growth) in tailings, contaminated soil and Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	228
281) Number of days required for emergence in Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	229
281) Number of days required for emergence in Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime - Continued.....	230
282) Germination (number of seedlings emerged/15 seeds, 14 day post-emergence) in Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	231
282) Germination (number of seedlings emerged/15 seeds, 14 day post-emergence) in Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime - Continued.....	232

LIST OF TABLES - Continued

Table	Page
283) Survival (number of seedlings survived/emerged, 14 day post-emergence) in Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	233
283) Survival (number of seedlings survived/emerged, 14 day post-emergence) in Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime - Continued.....	234
284) Shoot height (height from soil to longest leaf (mm), 14 day post-emergence) in Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	235
284) Shoot height (height from soil to longest leaf (mm), 14 day post-emergence) in Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime - Continued.....	236
285) Aboveground height (height from soil to longest leaf (mm), 90 day growth) Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	237
285) Aboveground height (height from soil to longest leaf (mm), 90 day growth) Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime - Continued.....	238
286) Root distribution (number of roots at 5 cm depth, 90 day growth) in Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	239
286) Root distribution (number of roots at 5 cm depth, 90 day growth) in Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime - Continued.....	240

LIST OF TABLES - Continued

Table	Page
287) Aboveground biomass (weight of aboveground production (g), 90 day growth) Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	241
287) Aboveground biomass (weight of aboveground production (g), 90 day growth) Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime - Continued.....	242
288) Belowground biomass (weight of belowground production (g), 90 day growth) Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	243
288) Belowground biomass (weight of belowground production (g), 90 day growth) Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime - Continued.....	244
289) Measured pH (s.u., 90 day growth) in Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime.....	245
289) Measured pH (s.u., 90 day growth) in Plant Growth Center soil amended with alkaline industrial by-products and commercial grade lime - Continued.....	246

LIST OF FIGURES

Figure	Page
1) Photos of Basin Wildrye (top) and Redtop (bottom) after a 111 day plant growth period in tailings as a function of different alkaline amendments.....	54
2) Photos of Basin Wildrye (top) and Redtop (bottom) after 111 day plant growth period in contaminated soil as a function of different alkaline amendments.....	55
3) Loss in above ground biomass, Basin Wildrye (top) and Redtop (bottom), with increasing application of alkaline amendments. Each point is a mean of 45 observations (9 alkaline products, 5 replications and error bars represent plus or minus one standard deviation.....	77
4) Loss in above ground biomass, Basin Wildrye (top) and Redtop (bottom), in relation to increased soil pH, electrical conductivity and alkaline amendment application rate.....	79

Abstract

In some regions of the United States the near proximity of alkaline industrial by-products to acidic-contaminated landscapes in concert with low acquisition cost, make these by-products an attractive option for soil remediation projects. The objective of this research was to determine whether acidic-contaminated soil systems amended with alkaline industrial by-products enable plant growth equivalent to that attained with a commercial grade mixture of CaCO_3 and CaO . In addition, it was determined whether an alkaline by-product dosage threshold existed, above which plant growth was impaired.

Three types of cement kiln dust (CKD), three types of lime kiln dust (LKD), and two other alkaline by-products (Dicalcium Silicate, Carbide Lime) were evaluated in this investigation. These alkaline by-products, and the standard treatment composed of a commercial grade CaCO_3/CaO mixture, were applied to metalliferous tailings (pH 1.8), and metal contaminated soil (pH 5.0) and plant growth was evaluated.

All alkaline products produced a desired soil pH (7.0 - 8.4) in the root zone during plant growth tests. Following a 111 day plant growth period with Basin Wildrye and Redtop all alkaline industrial by-products tested had plant growth equal to-or greater than- the CaCO_3/CaO mixture. This was the case in tailings and the contaminated soil for above ground plant biomass, plant height, root biomass, root depth, and number of roots at the 5 cm and 10 cm soil depths.

When alkaline by-products, including the CaCO_3/CaO mixture, were added to the Plant Growth Center soil, the greater the application rate the less was plant growth. Across the alkaline product dosage range of 0 % to 12 % (soil dry weight basis) the loss in aboveground plant biomass was 65 % for Basin Wildrye and 88 % for Redtop.

CHAPTER 1

INTRODUCTION

History has many lessons; not the least of which points to the importance of foresight. Mining practices that occurred in the United States prior to Federal and State legislation utilized all existing resources to extract the relevant mineral often without regard or respect for the land which harbored it. The advent of the industrial revolution in the United States brought with it the ability to strip the earth of resources crucial to the development, if not the integrity, of this nation.

The Butte-Anaconda region of Montana has a long history of mining, primarily hard rock mining. The processing required to extract and purify the relevant mineral produces a waste stream of material enriched in metals, primarily, Al, Cd, Cu, Pb, Zn, the metalloid As, and metal sulfides. The final processed ore is sent to a smelting operation where further impurities are volatilized. The waste or "mill tailings" are slurried into an impoundment where the excess water is extracted. Upon exposure to O_2 and H_2O , the metal sulfides present in the tailings produce hydrogen ions acidifying the soil and mobilizing metals that contaminate water resources and deter any vegetative establishment.

The waste impoundments cover many square kilometers and pose a health risk to both humans (U.S. EPA 1998) and wildlife (U.S. EPA, 1999). In addition to waste impoundments, many tens of square kilometers has been impacted by stack emissions emanating from the smelting process. It is assumed that dry deposition of metals and sulfates have decreased the pH of the soil impacting all but deeply rooted vegetation;

subsequently, erosion of the topsoil can be severe in these areas. The result is the largest Environmental Protection Agency (EPA) Superfund site in the country. Remedial strategies based upon the best available technology consist of a thin lime layer underlying a 46 cm coversoil cap and/or an in situ treatment of soil with a 60 %/40 % mixture of commercial grade CaCO_3/CaO respectively (RRU *et al*, 1987). The rationale depicted is establishing a suitable plant root zone and prevention of upward migration of contaminants and acidity into the coversoil and establishing a suitable plant root zone by raising the pH of the soil to a range of 9 - 11 s.u. to precipitate metals of concern after which plant growth can be established when pH falls below 8.5 s.u.. Regions in the state of Montana that require this type of reclamation are frequently limited by financial resources. The scale of the site and frequently, the lack of a responsible party, create a cost prohibitive scenario.

Alkaline Industrial By-Products

Due to absence of a market, many types of alkaline industrial by-products have historically been landfilled in the United States. Cement kiln dust (CKD) and lime kiln dust (LKD) are two of the most common alkaline by-products, but there are many others. In some regions of the United States, the near proximity of these alkaline by-products to acidic-contaminated landscapes in concert with low acquisition cost, make these by-products an attractive option for land reclamation projects. The U.S. EPA estimated that in 1990 the

cement industry produced approximately 14 million tons (12.7 metric tons) of cement kiln dust from 111 plants in 38 states (EPA, 1999). The industry disposed of 3.6 million metric tons of CKD in 1995 (EPA, 1999). Lime kiln dust, a by-product of calcium oxide production, is produced at 114 plants in 32 states (USGS, 1999).

Sources of Contamination

These alkaline products are in the strictest sense a waste stream and liability of the manufacturer. They are discarded due to impurities that are introduced via:

- i) the type of fuel used in the kiln process
- ii) the limestone ore body chemistry (or dolomite in the case of Dicalcium Silicate)
- iii) chemical inputs required for the final product
- iv) the degree of recirculation of the dust within the kiln

The type of fuel utilized in the kiln process can be coal, coke, petroleum, heat oil, natural gas or a combination of fuels. All fuel except natural gas leave an ash residue with an inherent metal and salt content. This "fly ash" becomes part of the kiln dust and directly affects the utility of the kiln dust for reclamation projects. Natural gas, which does not introduce new contaminants into the kiln can have the potential to concentrate the kiln dust relative to the burning of coal and coke. The introduction of coal and coke into the kiln is accomplished through forced air due to the extreme temperatures present. This input effectively dilutes the kiln dust several magnitudes by increasing the amount of kiln dust

created. A natural gas-fired kiln will not require this forced air input and relative to a coal and coke fueled kiln will have a increased concentration of contaminants (Holnam, 2000).

Companies that use kilns to produce burnt lime (CaO) typically input the highest grade of limestone available. Therefore, ore bodies associated with this industry are typically 95 % or greater limestone resulting in a low potential to introduce impurities into the LKD. Cement production in a kiln requires additional constituents to reach the desired output. The manufacturing of cement requires iron, aluminum and silica inputs in addition to limestone. When the ore body does not contain these materials, they must be added to the kiln. This has the potential to increase the metal and salt content of the resulting kiln dust compared to lime production.

Kiln dust, although impure, contains amounts of the original materials and can be recirculated into the kiln for continued processing. This results in highly enriched kiln dust but overall, the amount of kiln dust produced decreases which is beneficial to the manufacturer. A study by the Portland Cement Association (1992) chemically analyzed CKD at most cement manufacturing facilities in the United States. The only samples of CKD that failed to pass the Toxicity Characteristic Leaching Procedure (TCLP) test, which is used to determine whether a material is a hazardous waste, were those from kilns where the kiln dust was recirculated. The study concluded the single most important parameter in determining the level of trace metals in CKD is the degree of recirculation of the CKD in the kiln system.

Efforts to Characterize Kiln Dust Physicochemical Traits

Haynes and Kramer (1982) analyzed kiln dust samples from 102 cement plants in the United States. Concentrations of aluminum (Al), chloride (Cl^-), fluoride (F^-), sulfate (SO_4^{2-}), strontium (Sr), and titanium (Ti) were consistently greater than 500 mg/kg. Lead (Pb) concentrations ranged as high as 2500 mg/kg and 8000 mg/kg for zinc (Zn) while median values were 148 and 167 mg/kg, respectively. They determined CKD was not a hazardous waste as defined by the Resource Conservation and Recovery Act (RCRA).

The Portland Cement Association (1992) sampled kiln dust from 79 plants in the United States. All but two samples passed the TCLP test used to identify a hazardous waste. Total concentrations of 12 metals were determined and enrichment was greatest for barium (Ba) (mean 280 mg/kg) followed by chromium (Cr), nickel (Ni), arsenic (As), and Pb.

Dollhopf (1996a, 1996b, 1997a, 1997b) and Dollhopf and Juntunen (1995) analyzed both CKD and LKD from several facilities located in the northwest United States. All CKD and LKD evaluated had high calcium carbonate equivalence and contained desired compounds (CaCO_3 , $\text{Ca}(\text{OH})_2$, CaO) for neutralization of acidic-metalliferous mine waste. Some kiln dusts formed a notable quantity of coarse particles (>0.25 mm diameter) due to having been weathered in outdoor storage areas, a physical trait that is not desired for neutralization of acidic soil, but most materials were fine textured. Both LKD and CKD contained enriched levels of metals compared to concentrations present in natural soils in the United States. However, copper (Cu), manganese (Mn), Pb, Zn, and other metals were very enriched in some CKD samples. In addition, sodium adsorption ratio (SAR) in CKD

was typically very high (20 - 60) which could exacerbate a preexisting sodic condition in a soil. The soluble salt content in LKD was low but this trait was not analyzed in CKD. These investigators found both LKD and CKD were capable of neutralizing acidic-metalliferous mine waste, but did not have the opportunity to evaluate these industrial by-products with plant growth tests.

May (1999) evaluated whether representative trace metal data in CKD could be obtained from a single grab sample. More than 20,000 samples were collected from two cement kilns. Results indicated that variation between samples was sufficiently large that multiple samples should be collected to accurately determine chemical traits of CKD.

Kiln Dust use in the Soil - Plant System

Redente and Richard (1998) conducted plant (Redtop, *Agrostis alba*) growth tests in the greenhouse in acidic waste rock from the Summitville Superfund Site amended with LKD, quicklime (CaO), and limestone (CaCO₃). They found limestone supported significantly greater plant shoot and root biomass compared to LKD. This result was attributed to the high pH produced in the root zone when LKD was applied compared to that attained with limestone. However, the authors did not allow the LKD to attain a suitable pH <8.5 for plant establishment.

Gitt and Dollhopf (1991) treated acidic coal waste with agriculture grade limestone and CKD at a field site in central Montana that was seeded to a mixture of grasses and forbs. They found both amendments neutralized the acidic nature of the coal waste, but limestone treatment resulted in significantly more (3 fold) plant production compared to treatment with CKD.

Dollhopf and McDaniel (1997) studied alkaline industrial by-products (Dicalcium Silicate and Flux Bar Residue) produced from a kiln during the manufacture of magnesium from dolomite. Bench top column test indicated these alkaline by-products effectively neutralized acidic mine wastes and precipitated metals of concern. However, when these by-products were used to treat acidic wastes, above ground plant (*Agropyron intermedium*, Intermediate wheatgrass) production attained with a mixture of agriculture grade limestone and a commercially produced calcium hydroxide was 20-fold that produced with Dicalcium Silicate, and no plant growth was attained with Flux Bar Residue. Mine waste application rates for Dicalcium Silicate and Flux Bar Residue were very high, 190.6 t/1000t and 129 t/1000t respectively.

ARCO Environmental Remediation, L.L.C (1999) reported LKD had been used on eight sites in the Upper Clark Fork River Basin in Montana where the landscape had become acidic and contaminated with metals due to historical overland flooding containing mine waste and/or fallout from smelter emissions. These authors indicated vegetation measurements had not been completed on all sites, but some locations exhibited notable plant establishment and growth following treatment with LKD.

Winking and Dollhopf (2000) amended acidic-metalliferous tailings with LKD at an application rate of 9.8 % (dry weight basis), Dicalcium Silicate at an application rate of 9.5 %, and a mixture of commercial grade limestone with calcium hydroxide at a 8.5 % application rate. All amendments successfully neutralized the acid conditions in the tailings. Above and below ground growth of *Thinopyrum intermedium* (Intermediate wheatgrass, var. Tegmar) and *Elymus trachycaulum* (Slender wheatgrass, var. Pryor) were the same for tailings amended with the commercial grade limestone/calcium oxide mixture and LKD, but Dicalcium Silicate amended tailings had significantly less plant growth.

Investigators reported loss in plant growth when soils were treated with CKD. Saravanan and Appavu (1998) measured decreased root length, shoot length and seedling vigor index for crops grown in soils treated with CKD. Investigators determined plants grown in a solution treated with CKD had a decreased mitotic index which was related to plant chromosome damage and it was suggested CKD acted as a mutagen to the plant system (Ignacimuthu and Muraleytharan 1994, Kaushik 1996).

Numerous investigators reported deposition, i.e. dusting, of CKD onto plant leaves impaired growth of various plant species (Chitralekha and Dhakshinamoorthy 1998, Rao and Narayanan 1998, Durge and Phadnawis 1994, Durge and Phadnawis 1998, Hegazy 1996, Prasad and Inamdar 1990, Uma and Ramana 1994, Uma and Ramana 1993). This result may be a function of the caustic ($\text{pH} > 9$ s.u.) nature of kiln dusts. It is likely that any caustic amendment (i.e. CKD, LKD, CaO or $\text{Ca}(\text{OH})_2$) transferred by wind onto adjacent lands could cause impaired plant growth.

Gutenmann et al. (1994) found enriched levels of selenium in CKD resulted in significantly higher concentrations of this element in plant parts when the soil had been treated with CKD compared to commercial grade CaCO_3 and CaO .

Investigators reported LKD and CKD improved plant growth when applied to soils at rates comparable to fertilizer application or in combination with sewage sludge treatments (Simpson and Stopes 1991, Christie et al. 2001, Lafond and Simard 1999, Luo and Christie 1997).

Alkaline Industrial By-product Chemical Enrichment

Alkaline industrial by-products often contained metal contaminants at concentrations that may produce phytotoxic responses in plant growth. However, phytotoxic concentrations may be mitigated when applied to soils at an application rate of 2 - 10 % of the soil mass which facilitates dilution of the amendment metal chemistry, and the change in amendment pH from a range of 9.6 - 13.7 to the soil pH of 7.0 - 8.4 results in decreased contaminant solubility in the soil solution.

The water soluble metal content of an alkaline by-product at its pH, which ranged from 9.6 to 13.7, may be significantly different compared to an amended soil where the final pH will be in a target range of 7.0 - 8.4. It was predicted that water soluble metal concentration in alkaline by-products would be at lower concentration in an amended soil (pH 7.0 - 8.4) compared to its water soluble metal concentration at pH 9.6 - 13.7. As will be

