APPENDIX F

RAW DATA FOR pH, CONDUCTIVITY, TOTAL ORGANIC CARBON (TOC), UV/VIS SPECTROSCOPY, POTENTIOMETRIC TITRATIONS, AND MASS SPECTROMETRY

Table F-1. Raw data from pH and conductivity replicates

pH					
	Replicate #1	*Replicate #2*	*Replicate #3*	*Average*	*St. Dev (+/-)*
Sulfuric acid	1.356	1.366	1.372	1.365	0.008
Ionic	1.432	1.437	1.442	1.437	0.005
Sulfonated limonene	1.394	1.390	1.390	1.391	0.002
Polymer	11.421	11.441	11.405	11.422	0.018
Sodium silicate	11.320	11.280	11.350	11.317	0.035

Conductivity					
	Replicate #1	*Replicate #2*	*Replicate #3*	*Average*	*St. Dev (+/-)*
Sulfuric acid	15.50	15.33	15.74	15.523	0.206
Ionic	13.59	13.76	13.53	13.627	0.119
Sulfonated limonene	14.30	14.90	15.02	14.740	0.386
Polymer	12.10	12.71	11.42	12.077	0.645
Sodium silicate	11.88	12.20	11.90	11.993	0.179

Table F-2. Raw data from TOC autosampler

Injection Volume: 0.2 mL

Sample	*Raw Data*	*ppm*	***Average***	***St Dev. (+/-)***
SL500FIL	633569	14.6399		
SL500FIL	634375	14.6585		
SL500FIL	641511	14.8234	**14.70727**	**0.101003**
DI Wash	41341	0.9553		
I500FIL	3542746	81.8624		
I500FIL	3504580	80.9805		
I500FIL	3690199	85.2696	**82.70417**	**2.265066**
DI Wash	101944	2.3556		
DI Wash	102584	2.3704		
DI Wash	85604	1.9781		
DI Wash	92008	2.1260		
SL500UNF	1386969	32.0488		
SL500UNF	1405452	32.4759		
SL500UNF	1457914	33.6881	**32.73760**	**0.850407**
DI Wash	59353	1.3714		
DI Wash	55425	1.2807		
DI Wash	45809	1.0505		
DI Wash	52319	1.2089		
DI Wash	47771	1.1039		
I500UNF	3621734	83.6876		
I500UNF	3662877	84.6383	**84.16295**	**0.672246**
I500UNF**	3135289	72.4473		
Wash	85948	1.9860		
Wash	75966	1.7554		
Wash	75420	1.7427		
Wash	74116	1.7126		
Wash	72706	1.6800		
DI Wash	51205	1.1832		
DI Wash	46232	1.0683		
DI Wash	40892	0.9449		

** Result was discarded due to sample being retained in the instrument.
All samples were diluted 1:500 and deionized water rinses were made after each set of analyses.

SL= Sulfonated Limonene UNF= Unfiltered sample

I= Ionic Stabilizer FIL= Filtered sample

Table F-3. Raw data from TOC boat sampler

Injection Volume: 0.04 mL

Sample ID	*Raw Data*	*ppm Carbon*	***Average***	***St. Dev. (+/-)***
Polymer 1	2471919	255.3619		
Polymer 2	2205561	227.8457		
Polymer 3	2233280	230.7093	**237.97230**	**15.12774578**
Enzyme 1	365487	37.7567		
Enzyme 2	347814	35.9310		
Enzyme 3	382107	39.4736		
Enzyme 4	342730	35.4058	**37.14178**	**1.852487147**

Note: The enzyme sample was diluted 1:10,000.

Table F-4. Raw data from sulfate analysis

	Raw Data	*Concentration (mg/L)*	***Average***	***St. Dev. (+/-)***
Sulfuric acid	8138690	208.3817271		
	8224902	210.5974043		
	8023471	205.4205603	**208.1332**	**2.59735276**
Ionic stabilizer	4864650	124.2378052		
	4828890	123.3187612		
	4902345	125.2065793	**124.2544**	**0.94401818**
Sulfonated limonene	5151009	131.5973272		
	5033456	128.5761758		
	5213248	133.1968903	**131.1235**	**2.34652083**

Note: All samples were diluted 1:10,000.

Table F-5. UV/Visible spectrometer results for sulfonated limonene

λ	*Millipore*	*Sulfonated Limonene 1:200*[a]			*Average*	*Absorbance*
190	0	2.545	2.554	2.470	2.523	2.523
195	0.004	1.881	1.895	1.890	1.889	1.885
200	0.017	1.611	1.628	1.624	1.621	1.604
205	0.038	1.402	1.419	1.415	1.412	1.374
210	0.062	1.237	1.251	1.248	1.245	1.183
215	0.079	1.115	1.128	1.124	1.122	1.043
220	0.091	1.056	1.067	1.065	1.063	0.972
225	0.102	0.999	1.010	1.008	1.006	0.904
230	0.115	0.939	0.950	0.948	0.946	0.831
235	0.126	0.904	0.915	0.913	0.911	0.785
240	0.134	0.884	0.894	0.893	0.890	0.756
245	0.140	0.882	0.891	0.890	0.888	0.748
250	0.145	0.894	0.902	0.902	0.899	0.754
255	0.150	0.917	0.924	0.923	0.921	0.771
260	0.156	0.933	0.941	0.939	0.938	0.782
265	0.162	0.928	0.936	0.934	0.933	0.771
270	0.168	0.895	0.902	0.900	0.899	0.731
275	0.173	0.837	0.844	0.842	0.841	0.668
280	0.178	0.790	0.798	0.795	0.794	0.616
285	0.182	0.751	0.759	0.756	0.755	0.573
290	0.186	0.711	0.719	0.717	0.716	0.530
300	0.190	0.664	0.671	0.669	0.668	0.478
310	0.193	0.623	0.631	0.628	0.627	0.434
320	0.196	0.578	0.586	0.583	0.582	0.386
330	0.197	0.577	0.573	0.569	0.573	0.376
340	0.222	0.532	0.537	0.535	0.535	0.313
350	0.224	0.502	0.506	0.503	0.504	0.280
360	0.225	0.476	0.480	0.478	0.478	0.253
370	0.226	0.454	0.459	0.456	0.456	0.230
380	0.227	0.436	0.440	0.437	0.438	0.211
390	0.228	0.420	0.425	0.422	0.422	0.194
400	0.232	0.409	0.413	0.410	0.411	0.179
410	0.234	0.398	0.402	0.399	0.400	0.166
420	0.234	0.389	0.394	0.391	0.391	0.157
430	0.236	0.381	0.384	0.381	0.382	0.146
440	0.238	0.374	0.378	0.375	0.376	0.138
450	0.240	0.368	0.371	0.368	0.369	0.129

Table F-5. UV/Visible spectrometer results for sulfonated limonene (continued)

λ	*Millipore*	*Sulfonated Limonene 1:200*[a]			*Average*	*Absorbance*
460	0.241	0.363	0.365	0.362	0.363	0.122
470	0.242	0.358	0.360	0.359	0.359	0.117
480	0.244	0.353	0.356	0.353	0.354	0.110
490	0.245	0.349	0.352	0.349	0.350	0.105
500	0.247	0.347	0.348	0.346	0.347	0.100
510	0.248	0.343	0.346	0.343	0.344	0.096
520	0.250	0.340	0.343	0.340	0.341	0.091
530	0.251	0.337	0.340	0.337	0.338	0.087
540	0.252	0.335	0.338	0.335	0.336	0.084
550	0.254	0.333	0.336	0.333	0.334	0.080
560	0.254	0.330	0.334	0.332	0.332	0.078
570	0.256	0.328	0.332	0.330	0.330	0.074
580	0.257	0.327	0.330	0.328	0.328	0.071
590	0.258	0.325	0.328	0.326	0.326	0.068
600	0.258	0.323	0.326	0.325	0.325	0.067
610	0.258	0.322	0.324	0.323	0.323	0.065
620	0.259	0.320	0.323	0.322	0.322	0.063
630	0.260	0.318	0.322	0.320	0.320	0.060
640	0.259	0.316	0.320	0.317	0.318	0.059
650	0.259	0.315	0.319	0.316	0.317	0.058
660	0.260	0.314	0.318	0.315	0.316	0.056
670	0.261	0.312	0.317	0.314	0.314	0.053
680	0.261	0.311	0.316	0.313	0.313	0.052
690	0.262	0.310	0.314	0.311	0.312	0.050
700	0.263	0.310	0.313	0.310	0.311	0.048
710	0.263	0.308	0.313	0.310	0.310	0.047
720	0.263	0.307	0.312	0.309	0.309	0.046
730	0.264	0.307	0.311	0.308	0.309	0.045
740	0.265	0.306	0.310	0.307	0.308	0.043
750	0.265	0.306	0.309	0.306	0.307	0.042
800	0.266	0.304	0.307	0.306	0.306	0.040

[a] Dilutions were made from sulfonated limonene stock solution (3% limonene; 97% sulfuric acid by volume).

Table F-6. UV/Visible spectrometer results for the ionic stabilizer

λ	*Millipore*	*Ionic Stabilizer 1:200*			*Average*	*Absorbance*
190	0.000	2.720	2.686	2.686	2.697	2.697
195	0.004	2.787	2.760	2.756	2.768	2.764
200	0.017	3.017	0.300	2.990	2.102	2.085
205	0.038	2.696	0.268	2.673	1.879	1.841
210	0.062	1.213	1.198	1.194	1.202	1.140
215	0.079	0.938	0.922	0.918	0.926	0.847
220	0.091	0.975	0.960	0.957	0.964	0.873
225	0.102	1.092	1.077	1.075	1.081	0.979
230	0.115	1.212	1.199	1.199	1.203	1.088
235	0.126	1.196	1.186	1.187	1.190	1.064
240	0.134	0.966	0.956	0.959	0.960	0.826
245	0.140	0.611	0.599	0.601	0.604	0.464
250	0.145	0.470	0.461	0.464	0.465	0.320
255	0.150	0.453	0.445	0.447	0.448	0.298
260	0.156	0.468	0.460	0.461	0.463	0.307
265	0.162	0.493	0.485	0.485	0.488	0.326
270	0.168	0.515	0.507	0.507	0.510	0.342
275	0.173	0.530	0.523	0.522	0.525	0.352
280	0.178	0.523	0.515	0.514	0.517	0.339
285	0.182	0.499	0.491	0.491	0.494	0.312
290	0.186	0.427	0.419	0.418	0.421	0.235
300	0.190	0.357	0.349	0.348	0.351	0.161
310	0.193	0.336	0.328	0.328	0.331	0.138
320	0.196	0.316	0.308	0.308	0.311	0.115
330	0.197	0.332	0.325	0.323	0.327	0.130
340	0.222	0.323	0.316	0.314	0.318	0.096
350	0.224	0.315	0.308	0.306	0.310	0.086
360	0.225	0.309	0.302	0.299	0.303	0.078
370	0.226	0.302	0.296	0.293	0.297	0.071
380	0.227	0.298	0.291	0.288	0.292	0.065
390	0.228	0.294	0.288	0.284	0.289	0.061
400	0.232	0.292	0.286	0.282	0.287	0.055
410	0.234	0.291	0.284	0.280	0.285	0.051
420	0.234	0.289	0.283	0.278	0.283	0.049
430	0.236	0.287	0.282	0.277	0.282	0.046
440	0.238	0.286	0.281	0.276	0.281	0.043
450	0.240	0.285	0.280	0.275	0.280	0.040

Table F-6. UV/Visible spectrometer results for the ionic stabilizer (continued)

λ	*Millipore*	*Ionic Stabilizer 1:200*			*Average*	*Absorbance*
460	0.241	0.284	0.279	0.274	0.279	0.038
470	0.242	0.284	0.279	0.273	0.279	0.037
480	0.244	0.283	0.279	0.273	0.278	0.034
490	0.245	0.282	0.278	0.272	0.277	0.032
500	0.247	0.282	0.278	0.272	0.277	0.030
510	0.248	0.282	0.278	0.272	0.277	0.029
520	0.250	0.283	0.279	0.273	0.278	0.028
530	0.251	0.283	0.279	0.273	0.278	0.027
540	0.252	0.283	0.279	0.273	0.278	0.026
550	0.254	0.283	0.279	0.273	0.278	0.024
560	0.254	0.283	0.279	0.273	0.278	0.024
570	0.256	0.283	0.279	0.273	0.278	0.022
580	0.257	0.283	0.279	0.273	0.278	0.021
590	0.258	0.283	0.279	0.274	0.279	0.021
600	0.258	0.284	0.280	0.275	0.280	0.022
610	0.258	0.284	0.280	0.275	0.280	0.022
620	0.259	0.284	0.280	0.275	0.280	0.021
630	0.260	0.284	0.280	0.275	0.280	0.020
640	0.259	0.283	0.280	0.275	0.279	0.020
650	0.259	0.283	0.280	0.275	0.279	0.020
660	0.260	0.283	0.280	0.275	0.279	0.019
670	0.261	0.283	0.280	0.275	0.279	0.018
680	0.261	0.283	0.280	0.275	0.279	0.018
690	0.262	0.283	0.280	0.275	0.279	0.017
700	0.263	0.283	0.280	0.275	0.279	0.016
710	0.263	0.283	0.280	0.275	0.279	0.016
720	0.263	0.283	0.280	0.275	0.279	0.016
730	0.264	0.283	0.280	0.275	0.279	0.015
740	0.265	0.283	0.280	0.275	0.279	0.014
750	0.265	0.283	0.280	0.275	0.279	0.014
800	0.266	0.283	0.280	0.275	0.279	0.013

Note: Ionic stabilizer was modified using ammonium hydroxide and extracting to remove siloxane component.

Table F-7. UV/Visible spectrometer results for the polymer stabilizer

λ	*Millipore*	*Polymer 1:25*		*Average*	*Absorbance*	*Polymer (conc.)*			*Average*	*Absorbance*
190	-0.008	2.401	2.380	2.391	2.399			3.311	16.555	16.563
195	0.000	2.165	2.122	2.144	2.144			3.737	18.685	18.685
200	0.009	1.929	1.864	1.897	1.888			3.775	18.875	18.866
210	0.010	0.964	0.941	0.953	0.943			2.270	11.350	11.340
215	0.012	0.593	0.588	0.591	0.579			1.854	9.270	9.258
220	0.012	0.370	0.376	0.373	0.361			1.563	7.815	7.803
230	0.013	0.280	0.286	0.283	0.270			1.167	5.835	5.822
240	0.013	0.236	0.242	0.239	0.226			0.957	4.785	4.772
250	0.013	0.203	0.209	0.206	0.193			0.797	3.985	3.972
260	0.012	0.157	0.162	0.160	0.148			0.591	2.955	2.943
270	0.012	0.109	0.115	0.112	0.100			0.386	1.930	1.918
280	0.011	0.071	0.076	0.074	0.063			0.225	1.125	1.114
290	0.009	0.045	0.051	0.048	0.039	0.899	0.879	0.884	0.887	0.878
300	0.007	0.030	0.035	0.033	0.026	0.535	0.522	0.524	0.527	0.520
310	0.003	0.020	0.026	0.023	0.020	0.361	0.349	0.350	0.353	0.350
320	0.000	0.013	0.019	0.016	0.016	0.278	0.263	0.265	0.269	0.269
325	-0.002	0.010	0.017	0.014	0.016	0.251	0.241	0.240	0.244	0.246
326	0.020	0.032	0.036	0.034	0.014	0.230	0.217	0.219	0.222	0.202
328	0.020	0.032	0.036	0.034	0.014	0.215	0.202	0.206	0.208	0.188
330	0.018	0.031	0.034	0.033	0.015	0.202	0.191	0.194	0.196	0.178
335	0.014	0.029	0.031	0.030	0.016	0.192	0.181	0.184	0.186	0.172
340	0.012	0.027	0.029	0.028	0.016	0.184	0.171	0.174	0.176	0.164
350	0.009	0.024	0.025	0.025	0.016	0.174	0.163	0.166	0.168	0.159
360	0.007	0.020	0.022	0.021	0.014	0.169	0.159	0.162	0.163	0.156
370	0.005	0.017	0.019	0.018	0.013	0.164	0.155	0.158	0.159	0.154
380	0.003	0.015	0.017	0.016	0.013	0.162	0.151	0.154	0.156	0.153
390	0.004	0.013	0.018	0.016	0.012	0.159	0.147	0.150	0.152	0.148
400	0.004	0.015	0.016	0.016	0.012	0.156	0.144	0.147	0.149	0.145
410	0.003	0.013	0.015	0.014	0.011	0.149	0.141	0.143	0.144	0.141
420	0.002	0.012	0.015	0.014	0.012	0.146	0.138	0.140	0.141	0.139
430	0.002	0.012	0.014	0.013	0.011	0.143	0.135	0.136	0.138	0.136
440	0.001	0.010	0.013	0.012	0.011	0.141	0.134	0.135	0.137	0.136
450	0.001	0.010	0.013	0.012	0.011	0.140	0.132	0.133	0.135	0.134
460	0.001	0.009	0.013	0.011	0.010	0.139	0.129	0.129	0.132	0.131
470	0.001	0.009	0.013	0.011	0.010	0.135	0.127	0.127	0.130	0.129
480	0.001	0.009	0.013	0.011	0.010	0.133	0.125	0.127	0.128	0.127
490	0.001	0.009	0.013	0.011	0.010	0.132	0.123	0.126	0.127	0.126
500	0.001	0.009	0.013	0.011	0.010	0.129	0.121	0.125	0.125	0.124

Table F-7. UV/Visible spectrometer results for the polymer stabilizer (continued)

λ	*Millipore*	*Polymer 1:25*		*Average*	*Absorbance*	*Polymer (conc.)*			*Average*	*Absorbance*
510	0.001	0.009	0.013	0.011	0.010	0.127	0.120	0.124	0.124	0.123
520	0.001	0.009	0.013	0.011	0.010	0.124	0.118	0.122	0.121	0.120
530	0.001	0.009	0.013	0.011	0.010	0.122	0.116	0.120	0.119	0.118
540	0.002	0.008	0.012	0.010	0.008	0.122	0.114	0.118	0.118	0.116
550	0.002	0.008	0.012	0.010	0.008	0.12	0.113	0.117	0.117	0.115
560	0.002	0.008	0.012	0.010	0.008	0.119	0.111	0.114	0.115	0.113
570	0.002	0.008	0.012	0.010	0.008	0.117	0.109	0.113	0.113	0.111
580	0.002	0.008	0.012	0.010	0.008	0.116	0.108	0.112	0.112	0.110
590	0.002	0.008	0.012	0.010	0.008	0.115	0.106	0.110	0.110	0.108
600	0.002	0.008	0.012	0.010	0.008	0.110	0.102	0.109	0.107	0.105
610	0.002	0.008	0.011	0.010	0.008	0.110	0.101	0.108	0.106	0.104
620	0.002	0.007	0.010	0.009	0.007	0.108	0.100	0.107	0.105	0.103
630	0.001	0.007	0.010	0.009	0.008	0.106	0.098	0.105	0.103	0.102
640	0.000	0.006	0.009	0.008	0.008	0.107	0.097	0.104	0.103	0.103
650	-0.001	0.005	0.009	0.007	0.008	0.107	0.097	0.103	0.102	0.103
660	-0.001	0.005	0.009	0.007	0.008	0.103	0.094	0.100	0.099	0.100
670	-0.001	0.005	0.009	0.007	0.008	0.101	0.091	0.099	0.097	0.098
680	-0.001	0.005	0.009	0.007	0.008	0.100	0.090	0.098	0.096	0.097
690	-0.002	0.005	0.009	0.007	0.009	0.100	0.090	0.098	0.096	0.098
700	-0.002	0.005	0.008	0.007	0.009	0.099	0.089	0.098	0.095	0.097
710	-0.002	0.005	0.008	0.007	0.009	0.097	0.088	0.097	0.094	0.096
720	-0.002	0.005	0.008	0.007	0.009	0.096	0.088	0.096	0.093	0.095
730	-0.002	0.004	0.007	0.006	0.008	0.096	0.088	0.096	0.093	0.095
735	-0.002	0.005	0.008	0.007	0.009	0.095	0.087	0.095	0.092	0.094
740	-0.002	0.005	0.008	0.007	0.009	0.093	0.085	0.093	0.090	0.092
745	-0.002	0.005	0.008	0.007	0.009	0.091	0.083	0.091	0.088	0.090
750	-0.002	0.005	0.008	0.007	0.009	0.097	0.088	0.097	0.094	0.096
760	-0.002	0.004	0.007	0.006	0.008	0.096	0.088	0.096	0.093	0.095
770	-0.002	0.004	0.007	0.006	0.008	0.096	0.088	0.096	0.093	0.095
780	-0.002	0.004	0.007	0.006	0.008	0.095	0.087	0.095	0.092	0.094
790	-0.002	0.004	0.007	0.006	0.008	0.093	0.085	0.093	0.090	0.092
800	-0.003	0.004	0.007	0.006	0.009	0.091	0.083	0.091	0.088	0.091

Note: Absorbance was determined by subtracting the Millipore absorbance from the sample measurements

Table F-8. UV/Visible spectrometer results for the enzyme stabilizer (1:10,000) from wavelengths 190-750 nm

λ	*Millipore*	*Enzyme 1:10,000*			*Average*	*Absorbance*
190	0.000	1.182	1.069	1.068	1.106	1.106
192	0.008	1.274	1.151	1.149	1.191	1.183
195	0.016	1.242	1.117	1.125	1.161	1.145
197	0.023	1.072	0.962	0.963	0.999	0.976
200	0.030	0.669	0.602	0.603	0.625	0.595
205	0.044	0.392	0.360	0.357	0.370	0.326
210	0.058	0.358	0.331	0.329	0.339	0.281
215	0.070	0.371	0.342	0.340	0.351	0.281
220	0.082	0.400	0.369	0.368	0.379	0.297
225	0.091	0.382	0.352	0.352	0.362	0.271
230	0.099	0.285	0.267	0.266	0.273	0.174
240	0.112	0.174	0.164	0.163	0.167	0.055
250	0.122	0.174	0.166	0.165	0.168	0.046
255	0.126	0.182	0.174	0.175	0.177	0.051
260	0.130	0.193	0.186	0.186	0.188	0.058
270	0.138	0.215	0.207	0.208	0.210	0.072
280	0.144	0.213	0.206	0.208	0.209	0.065
290	0.149	0.186	0.183	0.184	0.184	0.035
300	0.151	0.182	0.179	0.180	0.180	0.029
310	0.152	0.178	0.175	0.175	0.176	0.024
320	0.152	0.175	0.173	0.172	0.173	0.021
330	0.178	0.204	0.202	0.202	0.203	0.025
340	0.179	0.200	0.200	0.198	0.199	0.020
350	0.179	0.197	0.196	0.195	0.196	0.017
360	0.179	0.194	0.192	0.192	0.193	0.014
370	0.179	0.190	0.190	0.189	0.190	0.011
380	0.179	0.188	0.188	0.187	0.188	0.009
390	0.179	0.187	0.187	0.186	0.187	0.008
400	0.182	0.188	0.188	0.188	0.188	0.006
410	0.184	0.188	0.188	0.188	0.188	0.004
420	0.185	0.188	0.188	0.188	0.188	0.003
430	0.186	0.189	0.189	0.189	0.189	0.003
440	0.188	0.190	0.190	0.190	0.190	0.002
450	0.189	0.191	0.191	0.191	0.191	0.002
460	0.190	0.192	0.192	0.192	0.192	0.002
470	0.191	0.193	0.193	0.193	0.193	0.002

Table F-8. UV/Visible spectrometer results for the enzyme stabilizer (1:10,000) from wavelengths 190-750 nm (continued)

λ	*Millipore*	*Enzyme 1:10,000*			*Average*	*Absorbance*
480	0.192	0.193	0.193	0.193	0.193	0.001
490	0.194	0.195	0.195	0.195	0.195	0.001
500	0.196	0.196	0.196	0.196	0.196	0.000
510	0.197	0.197	0.197	0.197	0.197	0.000
520	0.198	0.199	0.199	0.199	0.199	0.001
530	0.199	0.199	0.199	0.199	0.199	0.000
540	0.201	0.200	0.200	0.200	0.200	-0.001
550	0.202	0.201	0.201	0.201	0.201	-0.001
560	0.203	0.202	0.202	0.202	0.202	-0.001
570	0.204	0.202	0.202	0.202	0.202	-0.002
580	0.204	0.203	0.203	0.203	0.203	-0.001
590	0.205	0.204	0.204	0.204	0.204	-0.001
600	0.205	0.205	0.205	0.205	0.205	0.000
610	0.206	0.205	0.206	0.206	0.206	0.000
620	0.207	0.206	0.206	0.206	0.206	-0.001
630	0.207	0.206	0.206	0.206	0.206	-0.001
640	0.208	0.206	0.206	0.206	0.206	-0.002
650	0.208	0.206	0.206	0.206	0.206	-0.002
660	0.208	0.206	0.206	0.206	0.206	-0.002
670	0.208	0.206	0.206	0.206	0.206	-0.002
680	0.208	0.207	0.207	0.207	0.207	-0.001
690	0.208	0.208	0.208	0.208	0.208	0.000
700	0.209	0.208	0.208	0.208	0.208	-0.001
710	0.209	0.208	0.208	0.208	0.208	-0.001
720	0.210	0.208	0.208	0.209	0.208	-0.002
730	0.210	0.209	0.209	0.209	0.209	-0.001
740	0.210	0.209	0.209	0.209	0.209	-0.001
750	0.210	0.209	0.209	0.209	0.209	-0.001

Table F-9. UV/Visible spectrometer results for the enzyme stabilizer (1:5,000) from wavelengths 190-600 nm

λ	*Millipore*	*Enzyme 1:500*				*Average*	*Absorbance*
190	0.000	1.707	1.693	1.678	1.674	1.6880	1.688
192	0.060	1.814	1.818	1.807	1.804	1.8108	1.751
195	0.012	1.773	1.783	1.773	1.771	1.7750	1.763
200	0.028	1.012	1.003	0.983	0.986	0.9960	0.968
205	0.039	0.602	0.563	0.555	0.556	0.5690	0.530
210	0.047	0.529	0.494	0.488	0.489	0.5000	0.453
215	0.052	0.519	0.495	0.490	0.491	0.4988	0.447
217	0.054	0.527	0.507	0.504	0.504	0.5105	0.457
220	0.056	0.540	0.527	0.525	0.525	0.5293	0.473
222	0.058	0.538	0.529	0.526	0.527	0.5300	0.472
225	0.060	0.498	0.495	0.492	0.493	0.4945	0.435
230	0.064	0.355	0.352	0.348	0.348	0.3508	0.287
235	0.068	0.223	0.215	0.213	0.213	0.2160	0.148
240	0.069	0.184	0.177	0.176	0.176	0.1783	0.109
245	0.070	0.171	0.166	0.165	0.165	0.1668	0.097
250	0.072	0.167	0.163	0.162	0.162	0.1635	0.092
255	0.073	0.170	0.166	0.166	0.166	0.1670	0.094
260	0.074	0.177	0.174	0.174	0.174	0.1748	0.101
265	0.075	0.187	0.185	0.185	0.185	0.1855	0.111
270	0.076	0.197	0.195	0.195	0.195	0.1955	0.120
275	0.076	0.198	0.197	0.196	0.197	0.1970	0.121
280	0.077	0.187	0.186	0.186	0.185	0.1860	0.109
285	0.076	0.163	0.161	0.160	0.161	0.1613	0.085
290	0.076	0.146	0.143	0.142	0.143	0.1435	0.068
295	0.075	0.140	0.137	0.137	0.137	0.1378	0.063
300	0.074	0.136	0.133	0.132	0.132	0.1333	0.059
310	0.072	0.127	0.124	0.124	0.123	0.1245	0.053
320	0.070	0.119	0.117	0.116	0.116	0.1170	0.047
325	0.069	0.116	0.113	0.112	0.112	0.1133	0.044
330	0.087	0.129	0.128	0.128	0.127	0.1280	0.041
340	0.085	0.122	0.120	0.120	0.119	0.1203	0.035
350	0.083	0.115	0.113	0.113	0.112	0.1133	0.030
360	0.081	0.109	0.107	0.107	0.106	0.1073	0.026
370	0.078	0.102	0.100	0.101	0.100	0.1008	0.023
380	0.078	0.098	0.096	0.096	0.096	0.0965	0.019
390	0.077	0.093	0.092	0.092	0.092	0.0923	0.015
400	0.077	0.092	0.091	0.090	0.090	0.0908	0.014
410	0.077	0.090	0.089	0.088	0.088	0.0888	0.012

Table F-9. UV/Visible spectrometer results for the enzyme stabilizer (1:5,000) from wavelengths 190-600 nm (continued)

λ	*Millipore*	*Enzyme 1:500*				*Average*	*Absorbance*
420	0.076	0.088	0.087	0.086	0.086	0.0868	0.011
430	0.076	0.087	0.086	0.085	0.084	0.0855	0.010
440	0.076	0.086	0.083	0.084	0.084	0.0843	0.008
450	0.076	0.085	0.083	0.083	0.083	0.0835	0.008
460	0.076	0.085	0.083	0.083	0.082	0.0833	0.007
470	0.076	0.084	0.083	0.082	0.082	0.0828	0.007
480	0.076	0.083	0.082	0.082	0.082	0.0823	0.006
490	0.076	0.082	0.081	0.081	0.081	0.0813	0.005
500	0.076	0.082	0.081	0.081	0.081	0.0813	0.005
510	0.077	0.082	0.081	0.081	0.081	0.0813	0.004
520	0.077	0.082	0.081	0.081	0.081	0.0813	0.004
530	0.078	0.082	0.081	0.081	0.081	0.0813	0.003
540	0.078	0.082	0.081	0.081	0.081	0.0813	0.003
550	0.078	0.083	0.081	0.081	0.081	0.0815	0.004
560	0.079	0.083	0.082	0.081	0.081	0.0818	0.003
570	0.079	0.083	0.082	0.081	0.081	0.0818	0.003
580	0.079	0.083	0.082	0.081	0.081	0.0818	0.003
590	0.079	0.083	0.082	0.081	0.081	0.0818	0.003
600	0.079	0.083	0.082	0.081	0.081	0.0818	0.003

Table F-10. UV/Visible spectrometer results for the enzyme stabilizer (1:1,000) from wavelengths 190-305 nm

λ	*Millipore*	*Enzyme 1:1000*				*Average*	*Absorbance*
190	0.000	3.020	3.080	3.076	3.076	3.0630	3.0630
195	0.011	3.325	3.385	3.369	3.385	3.3660	3.3550
200	0.003	3.385	3.474	3.474	3.474	3.4518	3.4488
205	0.003	2.290	2.576	2.550	2.591	2.5018	2.4988
210	0.008	1.904	2.149	2.129	2.163	2.0863	2.0783
215	0.013	1.907	2.143	2.126	2.157	2.0833	2.0703
217	0.015	1.982	2.217	2.200	2.230	2.1573	2.1423
220	0.017	2.124	2.365	2.347	2.382	2.3045	2.2875
222	0.018	2.189	2.429	2.412	2.449	2.3698	2.3518
225	0.019	2.117	2.357	2.341	2.379	2.2985	2.2795
230	0.019	1.507	1.695	1.683	1.714	1.6498	1.6308
235	0.019	0.641	0.721	0.718	0.730	0.7025	0.6835
240	0.019	0.443	0.496	0.494	0.502	0.4838	0.4648
245	0.018	0.399	0.446	0.445	0.452	0.4355	0.4175
250	0.017	0.390	0.436	0.434	0.441	0.4253	0.4083
255	0.018	0.406	0.454	0.452	0.460	0.4430	0.4250
260	0.018	0.445	0.498	0.495	0.504	0.4855	0.4675
265	0.019	0.501	0.561	0.557	0.567	0.5465	0.5275
267	0.019	0.526	0.589	0.585	0.595	0.5738	0.5548
270	0.020	0.555	0.621	0.617	0.627	0.6050	0.5850
275	0.020	0.586	0.657	0.652	0.664	0.6398	0.6198
280	0.020	0.521	0.583	0.579	0.590	0.5683	0.5483
285	0.019	0.415	0.465	0.463	0.471	0.4535	0.4345
290	0.019	0.300	0.335	0.334	0.339	0.3270	0.3080
295	0.018	0.274	0.306	0.305	0.309	0.2985	0.2805
300	0.016	0.258	0.288	0.287	0.291	0.2810	0.2650
305	0.015	0.242	0.270	0.269	0.274	0.2638	0.2488

Table F-11. Raw data from titration of the ionic stabilizer

Titration: Ionic Stabilizer (1:500 filtered dilution)
Titrants: 1.0 N NaOH
Initial Volume: 50 mL (each replicate)

Base Added	*Replicate #1* *pH*	*Replicate #2* *pH*	*Replicate #3* *pH*
0.00	1.83	1.83	1.83
0.50	1.91	1.90	1.90
1.00	2.02	2.01	2.01
1.50	2.16	2.15	2.15
2.00	2.36	2.35	2.35
2.20	2.46	2.45	2.44
2.30	2.52	2.51	2.50
2.40	2.59	2.58	2.57
2.50	2.67	2.65	2.65
2.53	2.70	2.68	2.67
2.56	2.73	2.71	2.70
2.59	2.76	2.74	2.73
2.62	2.79	2.77	2.76
2.65	2.83	2.80	2.80
2.68	2.86	2.84	2.83
2.71	2.91	2.88	2.87
2.74	2.95	2.92	2.92
2.77	3.01	2.97	2.97
2.80	3.07	3.02	3.02
2.83	3.14	3.09	3.09
2.86	3.22	3.16	3.16
2.89	3.32	3.25	3.26
2.92	3.46	3.36	3.37
2.95	3.67	3.52	3.53
2.98	4.09	3.76	3.80
3.01	10.05	4.46	4.88
3.04	10.71	10.36	10.45
3.07	10.97	10.80	10.84
3.10	11.14	11.04	11.04

Note: 1 mL of ionic stabilizer in 500 ml volumetric flask filled with CO_2 free water. Filtered using 0.7 μm filter. Stirred in closed container in glovebox overnight.

Table F-12. Raw data from titration of sulfuric acid

Titration: Sulfuric Acid (1:500 dilution)
Titrants: 1.0 N NaOH
Initial volume: 50 mL

Base Added	*Replicate #1 pH*	*Replicate #2 pH*	*Replicate #3 pH*
0.00	1.88	1.90	1.92
0.50	1.96	1.98	1.99
1.00	2.06	2.07	2.09
1.50	2.18	2.19	2.21
2.00	2.33	2.34	2.37
2.20	2.41	2.41	2.44
2.30	2.45	2.45	2.48
2.40	2.50	2.50	2.53
2.50	2.55	2.55	2.58
2.53	2.57	2.57	2.60
2.56	2.59	2.59	2.62
2.59	2.61	2.60	2.63
2.62	2.63	2.62	2.65
2.65	2.65	2.64	2.67
2.68	2.67	2.66	2.69
2.71	2.68	2.68	2.71
2.74	2.70	2.70	2.74
2.77	2.73	2.72	2.76
2.80	2.76	2.74	2.78
2.83	2.78	2.78	2.81
2.86	2.80	2.80	2.83
2.89	2.83	2.82	2.86
2.92	2.87	2.85	2.89
2.95	2.90	2.88	2.93
2.98	2.93	2.91	2.96
3.01	2.97	2.95	3.00
3.04	3.01	2.98	3.04
3.07	3.06	3.03	3.08
3.10	3.11	3.08	3.13
3.13	3.16	3.12	3.19
3.16	3.23	3.18	3.26
3.19	3.30	3.24	3.33
3.22	3.39	3.32	3.43
3.25	3.51	3.42	3.55
3.28	3.69	3.55	3.72
3.31	3.97	3.72	4.02
3.34	6.27	4.02	7.20
3.37	10.54	9.84	10.6
3.40	10.86	10.65	10.91
3.43		10.96	11.08

Table F-13. Raw data from titration of sulfonated limonene

Titration: Sulfonated Limonene (1:500 dilution of stock solution--3% limonene; 97% sulfuric acid)
Titrants: 1.0 N NaOH
Initial Volume: 50 mL

Base Added	*Replicate #1* *pH*	*Replicate #2* *pH*	*Replicate #3* *pH*
0.00	1.79	1.79	1.79
0.50	1.86	1.87	1.87
1.00	1.97	1.98	1.98
1.50	2.11	2.11	2.11
2.00	2.29	2.29	2.30
2.20	2.38	2.39	2.39
2.30	2.44	2.44	2.45
2.40	2.50	2.50	2.51
2.50	2.57	2.57	2.58
2.53	2.59	2.59	2.60
2.56	2.62	2.62	2.63
2.59	2.64	2.64	2.65
2.62	2.67	2.67	2.68
2.65	2.69	2.70	2.71
2.68	2.72	2.73	2.74
2.71	2.76	2.76	2.77
2.74	2.79	2.79	2.81
2.77	2.83	2.83	2.85
2.80	2.87	2.87	2.89
2.83	2.91	2.92	2.94
2.86	2.96	2.97	2.99
2.89	3.02	3.02	3.05
2.92	3.09	3.09	3.12
2.95	3.16	3.16	3.20
2.98	3.25	3.26	3.30
3.01	3.37	3.37	3.42
3.04	3.54	3.54	3.61
3.07	3.81	3.82	4.00
3.10	5.07	5.05	9.85
3.13	10.49	10.48	10.67
3.16	10.86	10.86	10.95
3.19	11.06	11.06	11.12

Table F-14. Raw data from titration of the polymer stabilizer

Titration: Polymer (1:10 dilution)
Titrants: 1.0 N HCl
1.0 N NaOH
Initial vol. 50 mL (each replicate)

REPLICATE #1					
Acid Addition: pH = 11.23 to pH = 1.90			Base Addition: pH = 1.90 to pH = 11.10		
Addition #	*pH*	*Acid added (mL)*	*Addition #*	*pH*	*Base added (mL)*
0	11.23	0.00	0	1.90	0.00
1	11.17	0.30	1	1.94	0.10
2	11.13	0.60	2	1.98	0.20
3	11.07	0.90	3	2.03	0.30
4	11.04	1.20	4	2.08	0.40
5	10.99	1.50	5	2.11	0.50
6	10.95	1.80	6	2.24	0.60
7	10.92	2.10	7	2.33	0.70
8	10.87	2.40	8	2.45	0.80
9	10.86	2.60	9	2.62	0.90
10	10.83	2.80	10	2.90	1.00
11	10.80	3.00	11	3.58	1.10
12	10.77	3.20	12	5.60	1.20
13	10.75	3.40	13	6.51	1.30
14	10.73	3.60	14	6.98	1.40
15	10.71	3.80	15	7.33	1.50
16	10.70	4.00	16	7.64	1.60
17	10.66	4.20	17	7.81	1.70
18	10.64	4.40	18	8.01	1.80
19	10.61	4.60	19	8.16	1.90
20	10.57	4.80	20	8.35	2.00
21	10.54	5.00	21	8.50	2.10
22	10.50	5.20	22	8.63	2.20
23	10.47	5.40	23	8.76	2.30
24	10.43	5.60	24	8.89	2.40
25	10.40	5.80	25	9.01	2.50
26	10.37	6.00	26	9.12	2.60
27	10.32	6.20	27	9.22	2.70
28	10.28	6.40	28	9.33	2.80
29	10.24	6.60	29	9.44	2.90
30	10.19	6.80	30	9.53	3.00
31	10.13	7.00	31	9.62	3.10
32	10.08	7.20	32	9.72	3.20
33	10.01	7.40	33	9.80	3.30

Table F-14. Raw data from titration of the polymer stabilizer (continued)

Addition #	*pH*	*Acid added (mL)*	*Addition #*	*pH*	*Base added (mL)*
34	9.95	7.60	34	9.87	3.40
35	9.88	7.80	35	9.94	3.50
36	9.79	8.00	36	10.00	3.60
37	9.85	8.10	37	10.06	3.70
38	9.83	8.20	38	10.12	3.80
39	9.79	8.30	39	10.18	3.90
40	9.74	8.40	40	10.24	4.00
41	9.69	8.50	41	10.30	4.10
42	9.64	8.60	42	10.33	4.20
43	9.59	8.70	43	10.38	4.30
44	9.53	8.80	44	10.43	4.40
45	9.47	8.90	45	10.48	4.50
46	9.41	9.00	46	10.52	4.60
47	9.35	9.10	47	10.55	4.70
48	9.28	9.20	48	10.57	4.80
49	9.21	9.30	49	10.59	4.90
50	9.14	9.40	50	10.64	5.00
51	9.06	9.50	51	10.67	5.10
52	8.98	9.60	52	10.68	5.20
53	8.90	9.70	53	10.71	5.30
54	8.81	9.80	54	10.74	5.40
55	8.72	9.90	55	10.77	5.50
56	8.63	10.00	56	10.80	5.60
57	8.53	10.10	57	10.83	5.70
58	8.42	10.20	58	10.85	5.80
59	8.31	10.30	59	10.87	5.90
60	8.19	10.40	60	10.89	6.00
61	8.06	10.50	61	10.92	6.10
62	7.93	10.60	62	10.93	6.20
63	7.86	10.65	63	10.94	6.30
64	7.79	10.70	64	10.96	6.40
65	7.71	10.75	65	10.97	6.50
66	7.63	10.80	66	10.98	6.60
67	7.55	10.85	67	10.99	6.70
68	7.46	10.90	68	11.01	6.80
69	7.46	10.95	69	11.02	6.90
70	7.46	11.00	70	11.05	7.00
71	7.46	11.05	71	11.06	7.10
72	7.46	11.10	72	11.08	7.20
73	7.46	11.15	73	11.09	7.30
74	7.46	11.20	74	11.09	7.40
75	7.46	11.25	75	11.10	7.50
76	7.46	11.30			

Table F-14. Raw data from titration of the polymer stabilizer (continued)

Addition #	*pH*	*Acid added (mL)*	*Addition #*	*pH*	*Base added (mL)*
77	7.46	11.35			
78	7.46	11.40			
79	7.46	11.45			
80	6.13	11.50			
81	5.28	11.55			
82	4.17	11.60			
83	3.91	11.65			
84	3.81	11.70			
85	2.91	11.75			
86	2.71	11.80			
87	2.61	11.85			
88	2.54	11.90			
89	2.46	11.95			
90	2.41	12.00			
91	2.36	12.05			
92	2.32	12.10			
93	2.29	12.15			
94	2.26	12.20			
95	2.22	12.25			
96	2.19	12.30			
97	2.16	12.35			
98	2.13	12.40			
99	2.09	12.45			
100	2.06	12.50			
101	2.04	12.55			
102	2.02	12.60			
103	2.00	12.65			
104	1.97	12.70			
105	1.95	12.75			
106	1.94	12.80			
107	1.93	12.85			
108	1.91	12.90			
109	1.90	12.95			
REPLICATE #2					
Acid Addition: pH = 11.05 to pH = 1.82			Base Addition: pH = 1.79 to pH = 10.99		
Addition #	*pH*	*Acid added (mL)*	*Addition #*	*pH*	*Base added (mL)*
0	11.05	0.00	0	1.79	0.00
1	10.96	0.60	1	1.81	0.10
2	10.87	1.20	2	1.84	0.20
3	10.78	1.80	3	1.89	0.30
4	10.70	2.40	4	1.94	0.40

Table F-14. Raw data from titration of the polymer stabilizer (continued)

Addition #	*pH*	*Acid added (mL)*	*Addition #*	*pH*	*Base added (mL)*
5	10.67	2.80	5	1.99	0.50
6	10.63	3.20	6	2.06	0.60
7	10.58	3.60	7	2.13	0.70
8	10.54	4.00	8	2.21	0.80
9	10.50	4.40	9	2.32	0.90
10	10.46	4.80	10	2.46	1.00
11	10.40	5.20	11	2.66	1.10
12	10.36	5.60	12	2.99	1.20
13	10.32	6.00	13	3.53	1.30
14	10.25	6.40	14	5.52	1.40
15	10.18	6.80	15	6.01	1.50
16	10.07	7.20	16	6.72	1.60
17	9.96	7.60	17	6.84	1.70
18	9.80	8.00	18	6.90	1.80
19	9.72	8.20	19	7.12	1.90
20	9.63	8.40	20	7.28	2.00
21	9.52	8.60	21	7.36	2.10
22	9.41	8.80	22	7.39	2.20
23	9.28	9.00	23	7.44	2.30
24	9.13	9.20	24	8.57	2.40
25	8.97	9.40	25	8.71	2.50
26	8.81	9.60	26	8.85	2.60
27	8.63	9.80	27	8.99	2.70
28	8.42	10.00	28	9.12	2.80
29	8.20	10.20	29	9.24	2.90
30	7.94	10.40	30	9.35	3.00
31	7.65	10.60	31	9.46	3.10
32	7.50	10.70	32	9.56	3.20
33	7.33	10.80	33	9.66	3.30
34	7.14	10.90	34	9.75	3.40
35	7.00	11.00	35	9.82	3.50
36	7.00	11.10	36	9.90	3.60
37	6.99	11.20	37	9.97	3.70
38	6.99	11.30	38	10.02	3.80
39	7.00	11.40	39	10.09	3.90
40	6.44	11.50	40	10.14	4.00
41	5.60	11.60	41	10.19	4.10
42	5.50	11.70	42	10.25	4.20
43	5.35	11.80	43	10.29	4.30
44	5.02	11.90	44	10.34	4.40
45	4.76	12.00	45	10.39	4.50
46	4.64	12.10	46	10.44	4.60
47	4.47	12.20	47	10.46	4.70

Table F-14. Raw data from titration of the polymer stabilizer (continued)

Addition #	*pH*	*Acid added (mL)*	*Addition #*	*pH*	*Base added (mL)*
48	2.19	12.30	48	10.50	4.80
49	2.05	12.40	49	10.52	4.90
50	2.00	12.50	50	10.54	5.00
51	1.97	12.60	51	10.58	5.10
52	1.92	12.70	52	10.61	5.20
53	1.88	12.80	53	10.62	5.30
54	1.85	12.90	54	10.59	5.40
55	1.82	13.00	55	10.63	5.50
			56	10.66	5.60
			57	10.69	5.70
			58	10.72	5.80
			59	10.75	5.90
			60	10.78	6.00
			61	10.80	6.10
			62	10.83	6.20
			63	10.85	6.30
			64	10.82	6.40
			65	10.82	6.50
			66	10.85	6.60
			67	10.85	6.70
			68	10.86	6.80
			69	10.86	6.90
			70	10.88	7.00
			71	10.88	7.10
			72	10.88	7.20
			73	10.89	7.30
			74	10.90	7.40
			75	10.91	7.50
			76	10.93	7.60
			77	10.94	7.70
			78	10.95	7.80
			79	10.95	7.90
			80	10.96	8.00
			81	10.97	8.10
			82	10.97	8.20
			83	10.97	8.30
			84	10.98	8.40
			85	10.99	8.50

Table F-15. Raw data from titration of the enzyme stabilizer

Titrants: 1.0 N HCl
1.0 N NaOH
Initial vol. 50 mL

REPLICATE #1					
Acid Addition: pH = 4.00 to pH = 1.98			Base Addition: pH = 1.97 to pH = 11.04		
Addition #	*pH*	*Acid added (mL)*	*Addition #*	*pH*	*Base added (mL)*
0	4.00	0.00	0	1.97	0.00
1	2.65	0.20	1	2.18	0.20
2	2.39	0.30	2	2.34	0.30
3	2.22	0.40	3	2.59	0.40
4	2.16	0.45	4	2.79	0.45
5	2.11	0.50	5	3.08	0.50
6	2.06	0.55	6	3.51	0.55
7	2.01	0.60	7	4.00	0.60
8	1.98	0.65	8	4.56	0.65
			9	5.49	0.70
			10	9.49	0.75
			11	10.67	0.80
			12	11.04	0.85
REPLICATE #2					
Acid Addition: pH = 4.01 to pH = 1.96			Base Addition: pH = 1.96 to pH = 11.03		
Addition #	*pH*	*Acid added (mL)*	*Addition #*	*pH*	*Base added (mL)*
0	4.01	0.00	0	1.96	0.000
1	3.54	0.05	1	2.00	0.050
2	3.15	0.10	2	2.05	0.100
3	2.85	0.15	3	2.11	0.150
4	2.64	0.20	4	2.17	0.200
5	2.49	0.25	5	2.25	0.250
6	2.38	0.30	6	2.34	0.300
7	2.29	0.35	7	2.44	0.350
8	2.22	0.40	8	2.59	0.400
9	2.15	0.45	9	2.79	0.450
10	2.10	0.50	10	3.08	0.500
11	2.05	0.55	11	3.49	0.550
12	2.00	0.60	12	3.97	0.600
13	1.96	0.65	13	4.53	0.650
			14	4.89	0.675
			15	5.42	0.700

Table F-15. Raw data from titration of the enzyme stabilizer (continued)

Acid Addition: pH = 4.01 to pH = 1.96			Base Addition: pH = 1.96 to pH = 11.03		
Addition #	*pH*	*Acid added (mL)*	*Addition #*	*pH*	*Base added (mL)*
			16	7.17	0.725
			17	9.41	0.750
			18	10.26	0.775
			19	10.66	0.800
			20	10.88	0.825
			21	11.03	0.850
REPLICATE #3					
Acid Addition: pH = 4.00 to pH = 1.95			Base Addition: pH = 1.95 to pH = 11.05		
Addition #	*pH*	*Acid added (mL)*	*Addition #*	*pH*	*Base added (mL)*
0	4.00	0.00	0	1.95	0.000
1	3.53	0.05	1	1.99	0.050
2	3.12	0.10	2	2.04	0.100
3	2.82	0.15	3	2.10	0.150
4	2.62	0.20	4	2.16	0.200
5	2.48	0.25	5	2.24	0.250
6	2.36	0.30	6	2.33	0.300
7	2.27	0.35	7	2.44	0.350
8	2.20	0.40	8	2.59	0.400
9	2.14	0.45	9	2.79	0.450
10	2.08	0.50	10	3.09	0.500
11	2.03	0.55	11	3.53	0.550
12	1.99	0.60	12	4.02	0.600
13	1.95	0.65	13	4.61	0.650
			14	5.01	0.675
			15	5.66	0.700
			16	8.09	0.725
			17	9.57	0.750
			18	10.34	0.775
			19	10.69	0.800
			20	10.90	0.825
			21	11.05	0.850

Table F-15. Raw data from titration of the enzyme stabilizer (continued)

REPLICATE #4					
Acid Addition: pH = 4.01 to pH = 1.96			Base Addition: pH = 1.96 to pH = 11.05		
Addition #	*pH*	*Acid added (mL)*	*Addition #*	*pH*	*Base added (mL)*
0	4.01	0.00	0	1.96	0.000
1	3.54	0.05	1	2.00	0.050
2	3.13	0.10	2	2.05	0.100
3	2.83	0.15	3	2.10	0.150
4	2.63	0.20	4	2.16	0.200
5	2.49	0.25	5	2.23	0.250
6	2.37	0.30	6	2.32	0.300
7	2.29	0.35	7	2.43	0.350
8	2.21	0.40	8	2.57	0.400
9	2.14	0.45	9	2.76	0.450
10	2.09	0.50	10	3.05	0.500
11	2.04	0.55	11	3.46	0.550
12	2.00	0.60	12	3.93	0.600
13	1.96	0.65	13	4.50	0.650
			14	4.84	0.675
			15	5.34	0.700
			16	6.86	0.725
			17	9.35	0.750
			18	10.25	0.775
			19	10.66	0.800
			20	10.89	0.825
			21	11.05	0.850

Table F-16. FAB analysis of the enzyme stabilizer

```
LIST:  d19kz478b                              19-DEC-00   REG    :  00:15.3       #9
Samp:  KSHAW/PZ2                                          Start  :  16:49:18      33
Comm:  Mass Spec Facility, Chem. & Biochem, UT Austin
Mode:  FAB +Q3MS HMR UP LR                                Study  :  FAB/NBA
Oper:  rr                                                 Inlet  :
Base:  562.9                  Inten  :  15567028          Masses:  200 > 2000
Norm:  562.9                  RIC    :  384937976         #peaks:  1837
Peak:  1000.00  mmu
Data:  /10>20
```

No.	Mass	Intensity (1320126)	%RA	%RIC	Flags
1	215.	1432564	9.20	0.37	F#
2	215.	1359979	8.74	0.35	FM#
3	259.	1482875	9.53	0.39	F#
4	303.	1320127	8.48	0.34	F#
5	347.	1391297	8.94	0.36	F#
6	415.	2696231	17.32	0.70	FM#
7	431.	3140779	20.18	0.82	FM#
8	457.	1359217	8.73	0.35	F#
9	459.	4573616	29.38	1.19	F#
10	475.	7694969	49.43	2.00	F#
11	476.	1370896	8.81	0.36	F#
12	501.	1548028	9.94	0.40	F#
13	503.	5191024	33.35	1.35	FM#
14	517.	1619479	10.40	0.42	F#
15	519.	12801280	82.23	3.33	F#
16	520.	2361525	15.17	0.61	F#
17	545.	1569341	10.08	0.41	F#
18	547.	4690414	30.13	1.22	F#
19	547.	1331732	8.55	0.35	F#
20	561.	2265827	14.56	0.59	F#
21	563.	15566848	100.00	4.04	F#
22	564.	3120142	20.04	0.81	F#
23	565.	1321637	8.49	0.34	F#
24	567.	1752087	11.26	0.46	F#
25	567.	2909037	18.69	0.76	F#
26	589.	1336818	8.59	0.35	F#
27	591.	3383953	21.74	0.88	F#
28	605.	2368526	15.22	0.62	F#
29	607.	14122752	90.72	3.67	F#
30	608.	3059936	19.66	0.79	F#
31	609.	1343771	8.63	0.35	F#
32	611.	4369153	28.07	1.14	F#
33	611.	2456067	15.78	0.64	FM#
34	635.	3198283	20.55	0.83	F#
35	635.	1641449	10.54	0.43	F#
36	649.	2062091	13.25	0.54	F#
37	651.	11926016	76.61	3.10	F#
38	652.	2585615	16.61	0.67	F#
39	655.	4821463	30.97	1.25	FM#
40	679.	2081806	13.37	0.54	F#
41	679.	1485246	9.54	0.39	F#
42	693.	1946679	12.51	0.51	F#
43	695.	9538816	61.28	2.48	F#
44	696.	2013442	12.93	0.52	F#
45	699.	4865846	31.26	1.26	F#
46	723.	2051967	13.18	0.53	F#
47	737.	1432923	9.20	0.37	F#
48	738.	6966681	44.75	1.81	F#
49	743.	3210773	20.63	0.83	F#
50	767.	1458718	9.37	0.38	FM#
51	781.	1566072	10.06	0.41	F#
52	782.	3235119	20.78	0.84	F#
53	783.	1531332	9.84	0.40	F#
54	787.	2600359	16.70	0.68	F#
55	826.	1561654	10.03	0.41	F#
56	827.	2474511	15.90	0.64	F#
57	831.	1631558	10.48	0.42	F#

```
Date: Wed Dec 20 14:31:09 2000   ICIS: 8.3.0 SP1 for OSF1 (V4.0) build 97-324 from 20-Nov-97
```

APPENDIX G

XRD RESULTS FOR ORIENTED AND GLYCOLATED SAMPLES

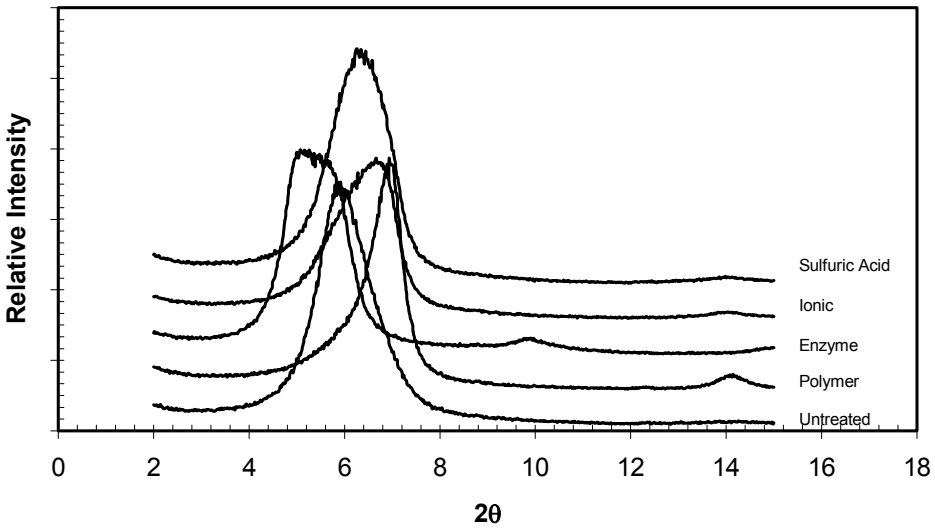

Figure G-1. Oriented results for sodium montmorillonite

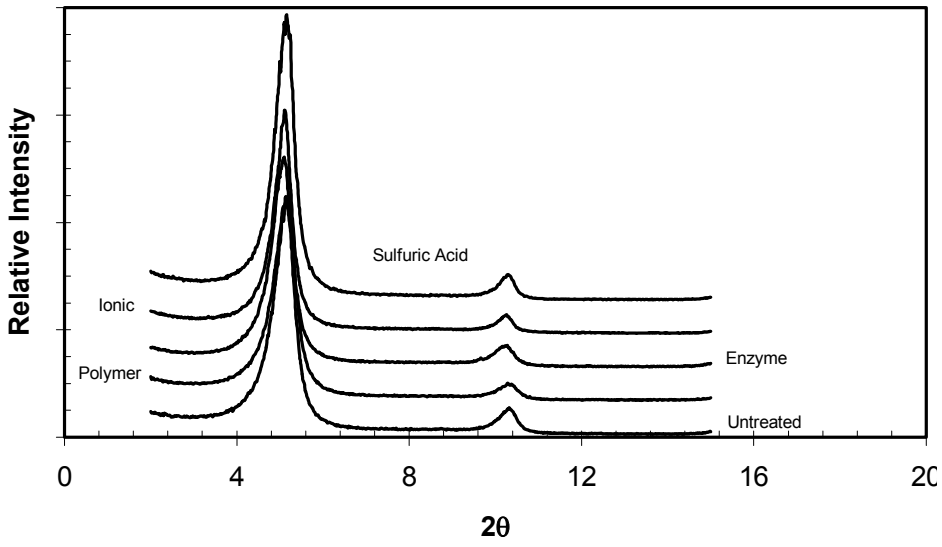

Figure G-2. Glycolated results for sodium montmorillonite

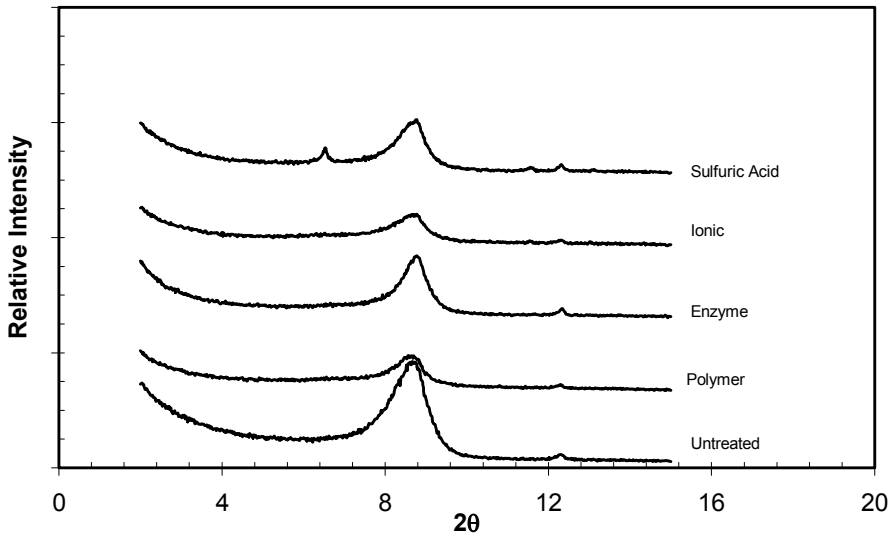

Figure G-3. Oriented results for illite

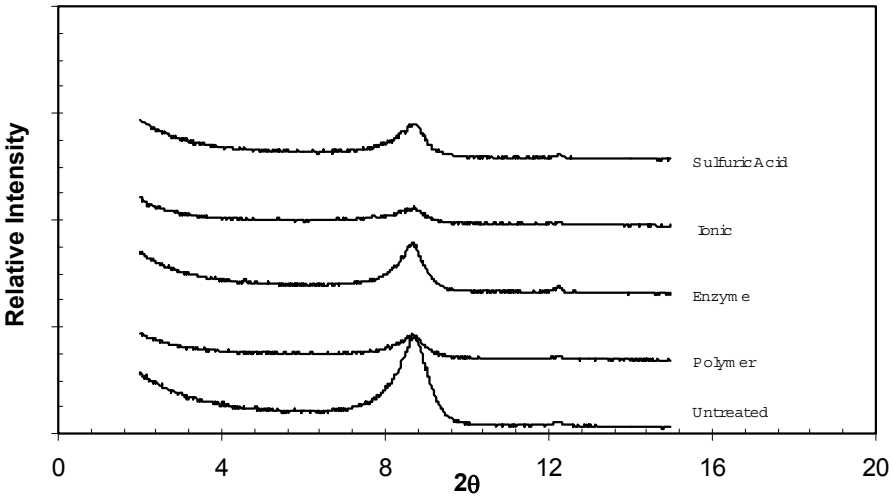

Figure G-4. Glycolated results for illite

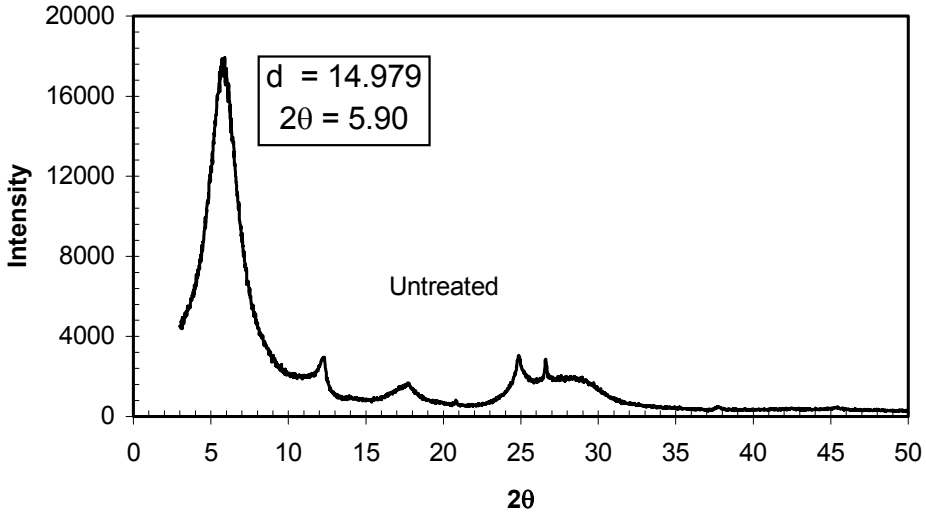

Figure G-5. Oriented results for Bryan soil

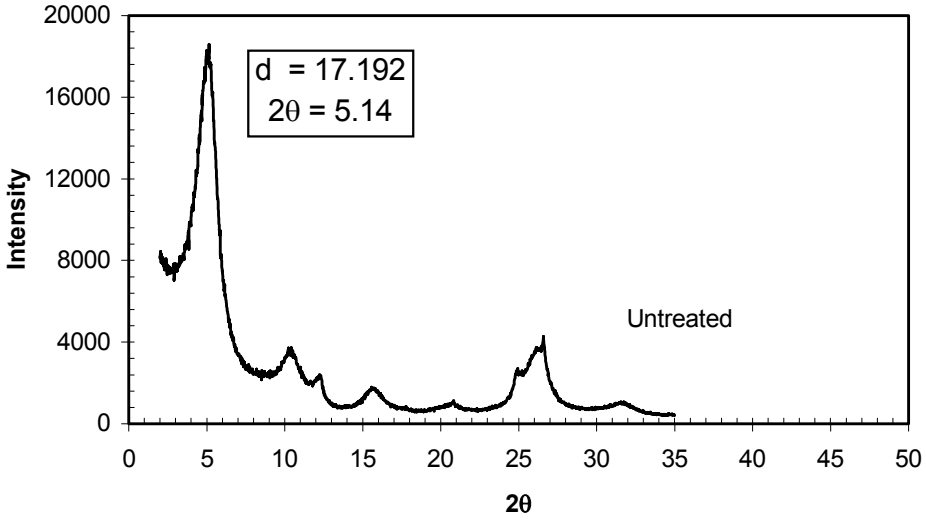

Figure G-6. Glycolated results for Bryan soil

APPENDIX H

RESULTS FROM HYDROMETER ANALYSIS OF GRAIN SIZE DISTRIBUTIONS OF BULK TEST SOILS

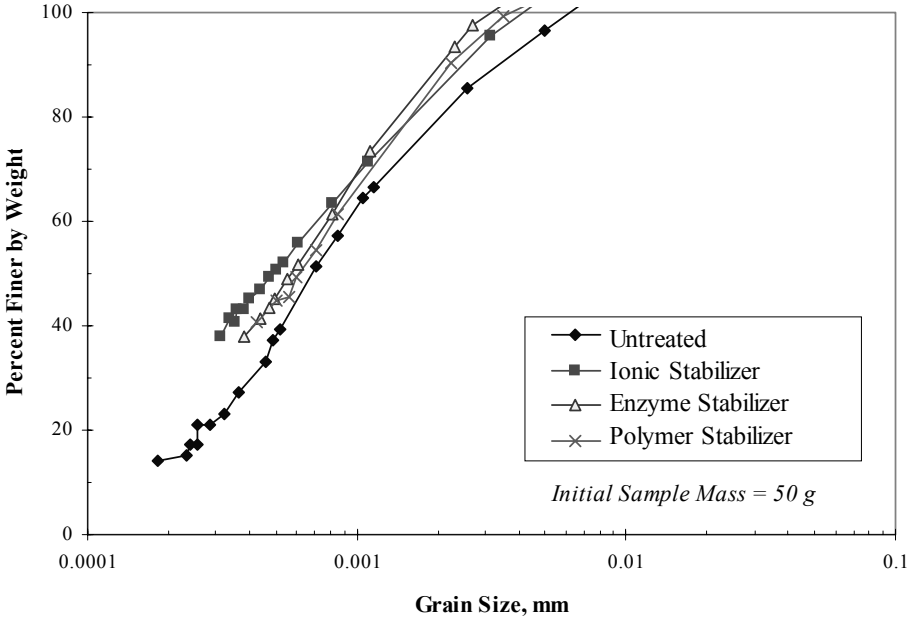

Figure H-1. Grain size distribution of untreated and treated bulk kaolinite

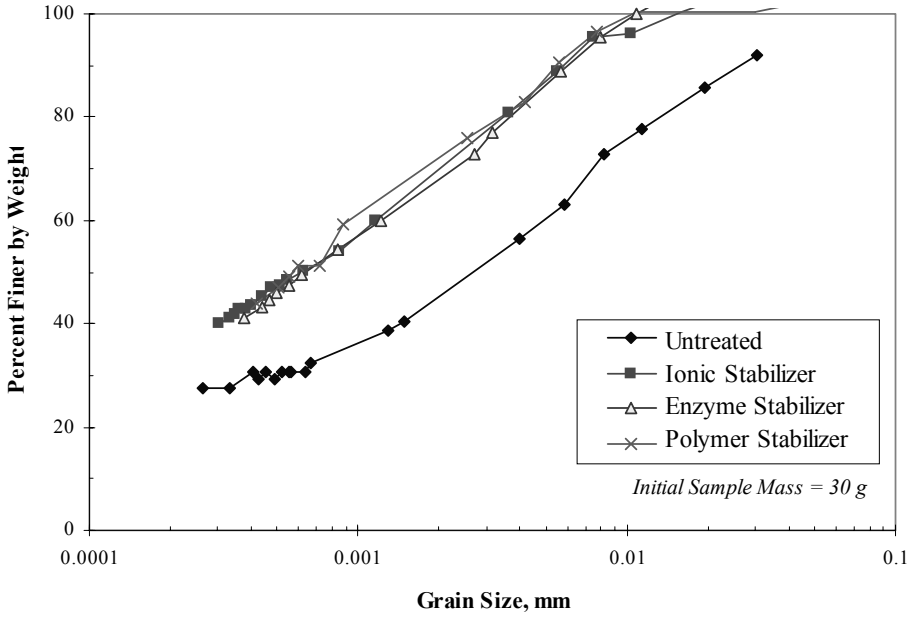

Figure H-2. Grain size distribution of untreated and treated bulk illite

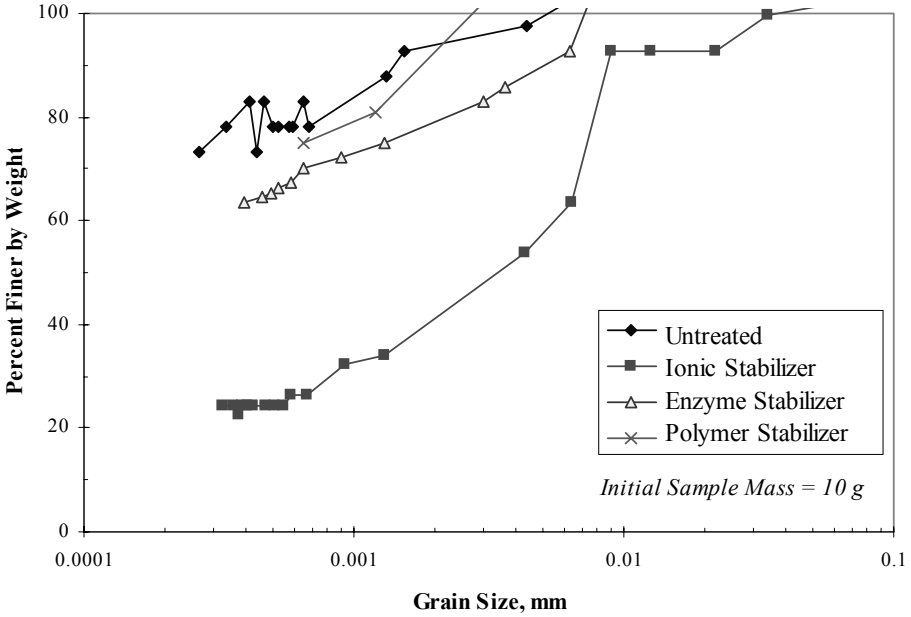

Figure H-3. Grain size distribution of untreated and treated bulk montmorillonite

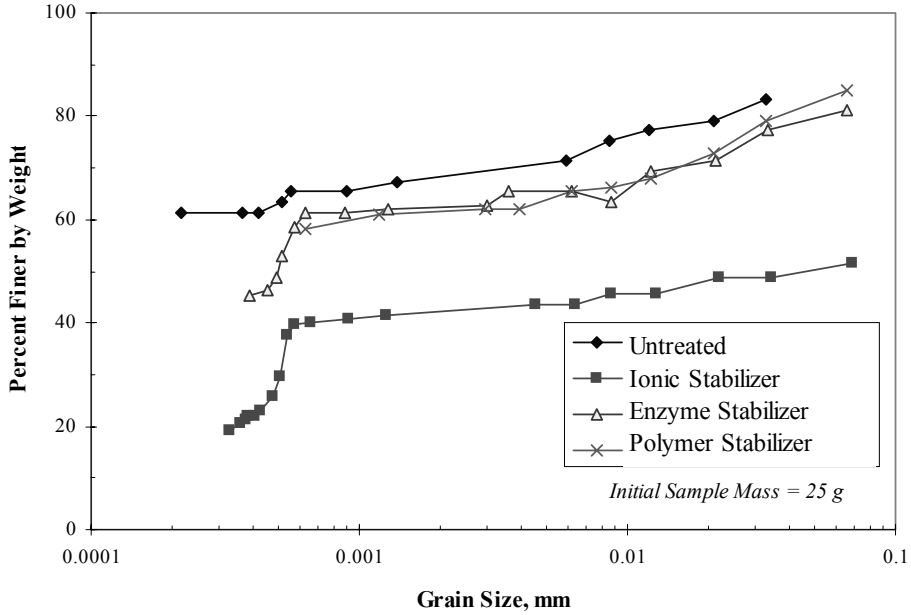

Figure H-4. Grain size distribution of untreated and treated TX Bryan HP

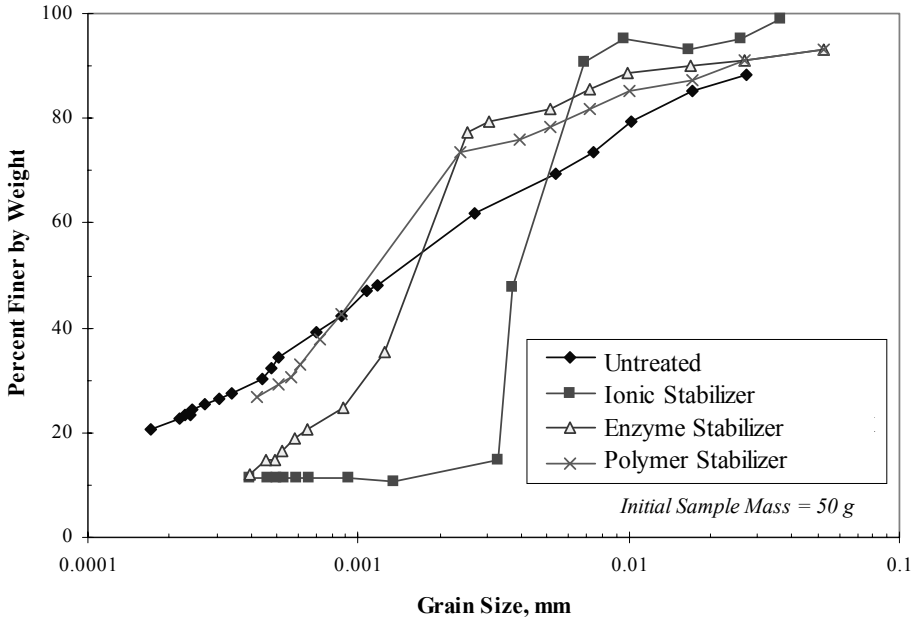

Figure H-5. Grain size distribution of untreated and treated bulk TX Mesquite HS HP

APPENDIX I

MEASURED ATTERBERG LIMITS OF UNTREATED AND TREATED BULK TEST SOILS

Table I-1. Summary of measured Atterberg limits for all untreated and treated bulk soils

Bulk Soil Sample	*Index Property*[1]	*Untreated Soil*	*Soil Treated with Stabilizer Product*		
			Ionic	*Enzyme*	*Polymer*
Kaolinite	PL	32	28	28	27
	LL	51	52	49	47
	PI	19	24	21	20
Illite	PL	24	19	18	18
	LL	44	47	50	44
	PI	20	28	32	26
Montmorillonite	PL	32	36	33	35
	LL	567	485	612	547
	PI	535	449	579	512
TX Bryan HP	PL	20	15	15	14
	LL	68	65	68	62
	PI	48	50	53	48
TX Mesquite HS HP	PL	23	22	19	20
	LL	60	49	53	50
	PI	37	27	34	30

[1] PL = Plastic Limit
LL = Liquid Limit
PI = Plasticity Index (LL – PL)

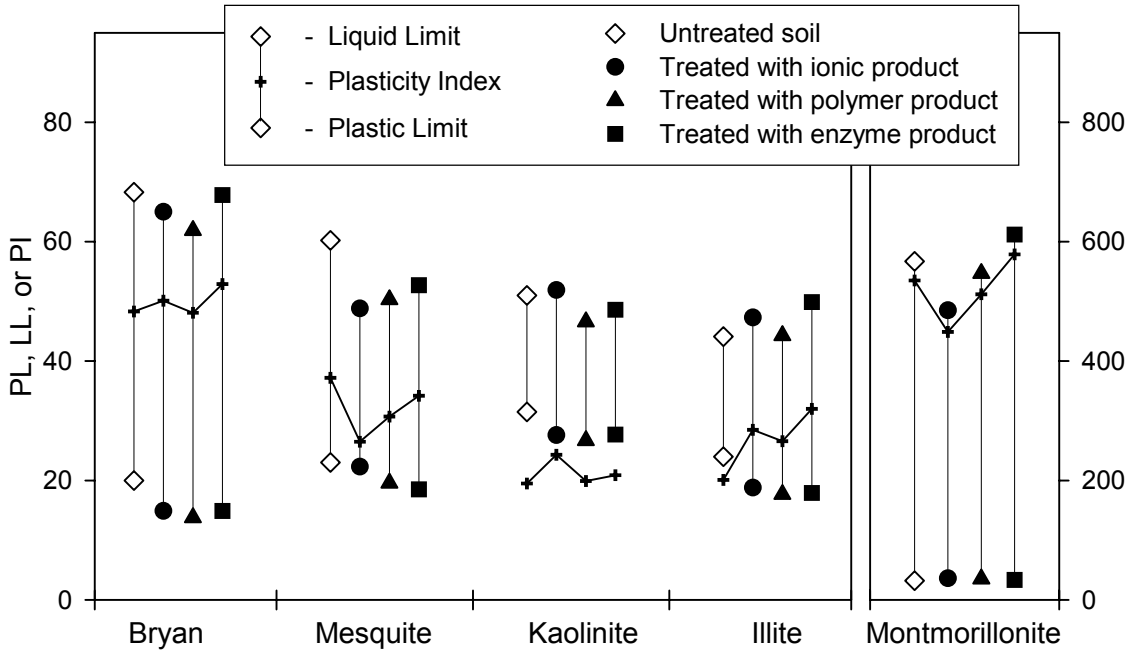

Figure I-1. Summary of measured Atterberg limits for all untreated and treated soils

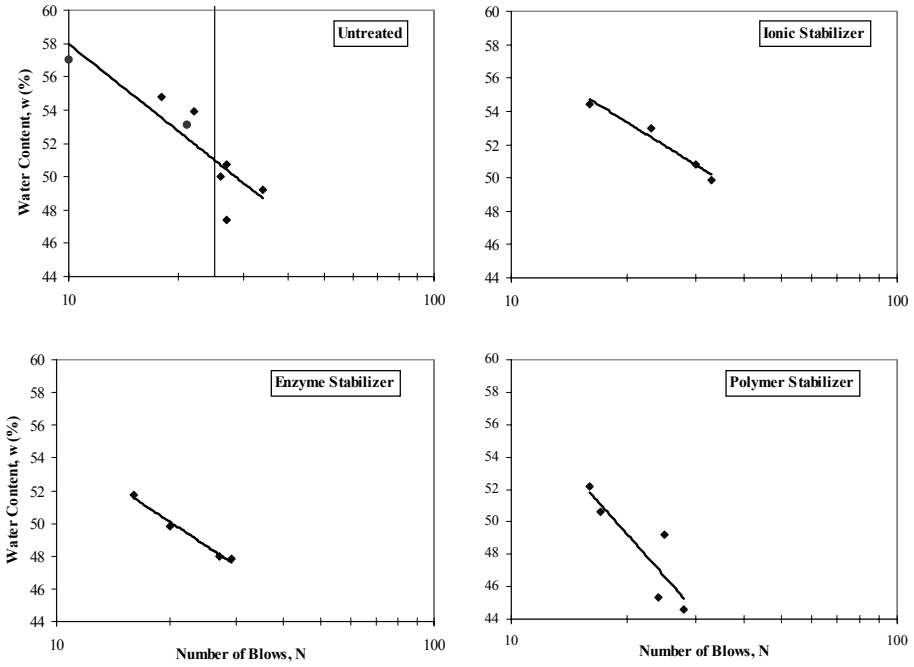

Figure I-2. Liquid limit test results on bulk kaolinite

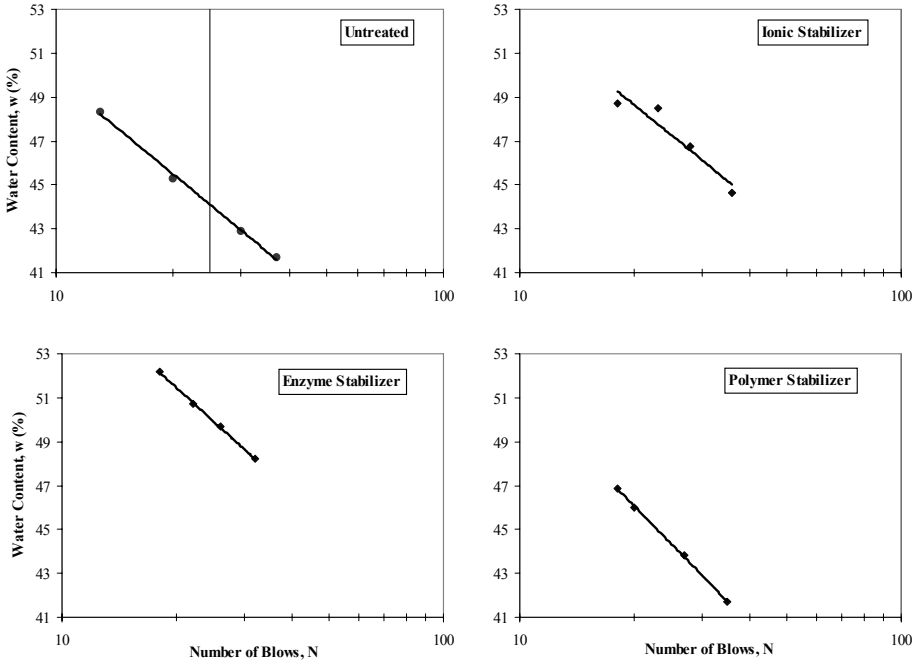

Figure I-3. Liquid Limit test results on bulk illite

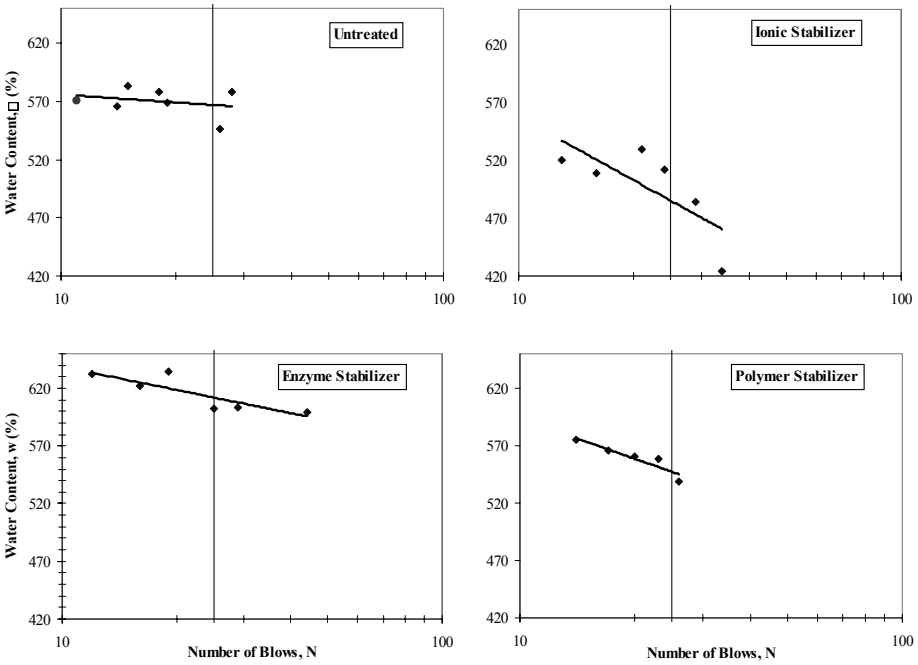

Figure I-4. Liquid limit test results on bulk montmorillonite

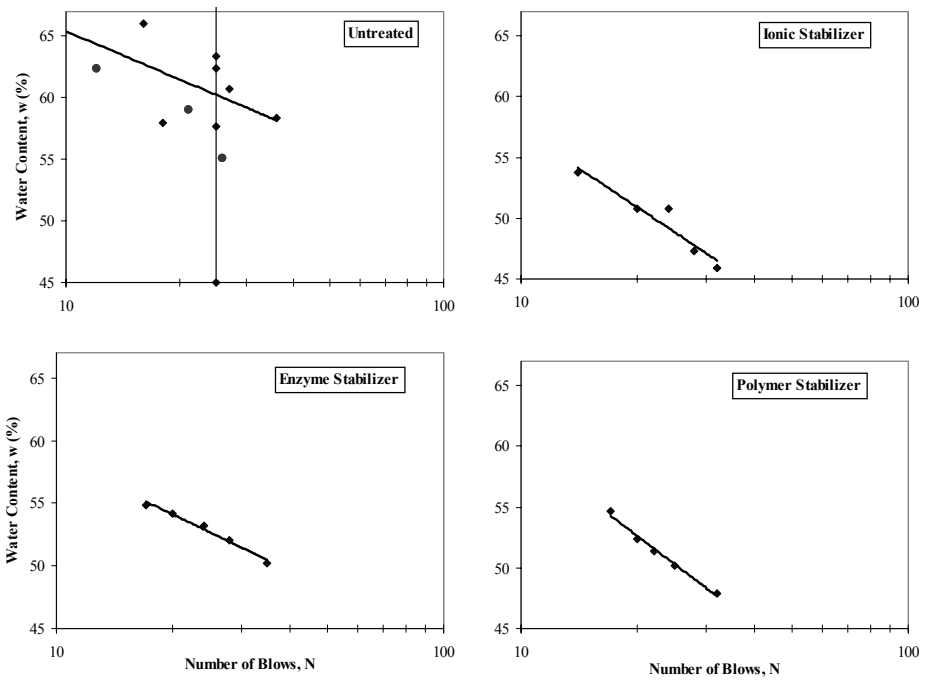

Figure I-5. Liquid limit test results on TX Bryan HP

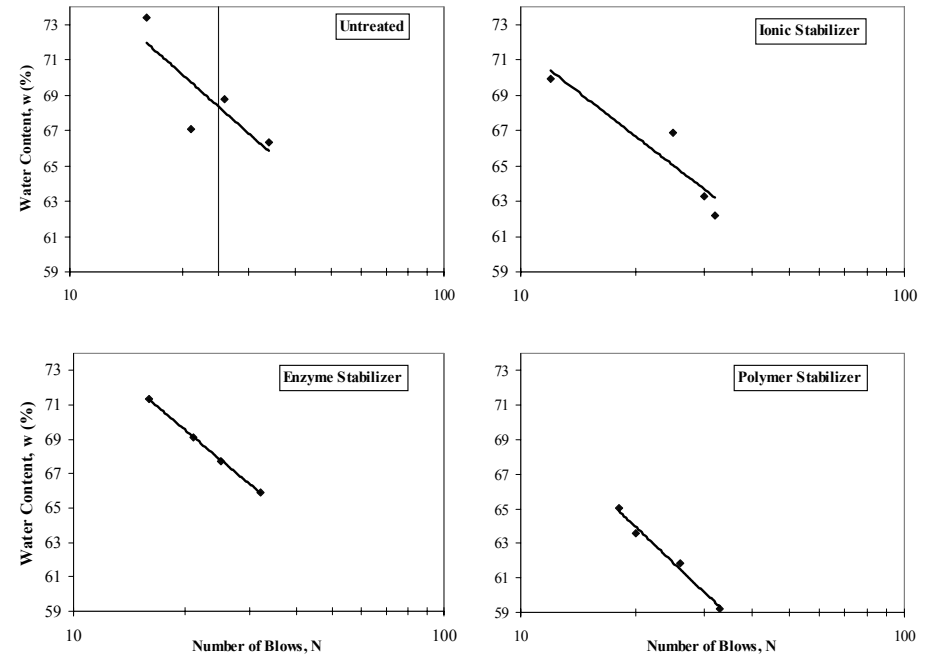

Figure I-6. Liquid limit test results on TX Mesquite HS HP

APPENDIX J

RESULTS FROM COMPACTION TESTS ON UNTREATED BULK TEST SOILS

Table J-1. Summary of compaction test results from tests on untreated bulk soil samples

Bulk Soil Sample	*Maximum Dry Unit Weight (pcf)*	*Optimum Water Content for Compaction (%)*
Kaolinite	98.7	24
Illite	124.5	12
Montmorillonite	96.8	24
TX Bryan HP	115.0	16
TX Mesquite HS HP	112.0	17

Note: All values determined using modified Proctor compaction energy (ASTM D 1557).

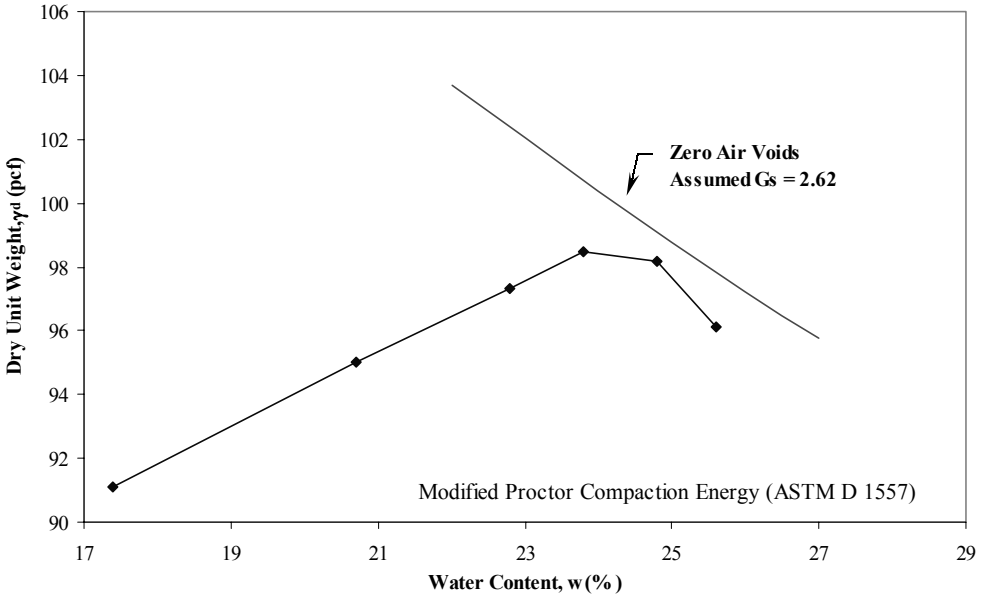

Figure J-1. Compaction test results on bulk kaolinite

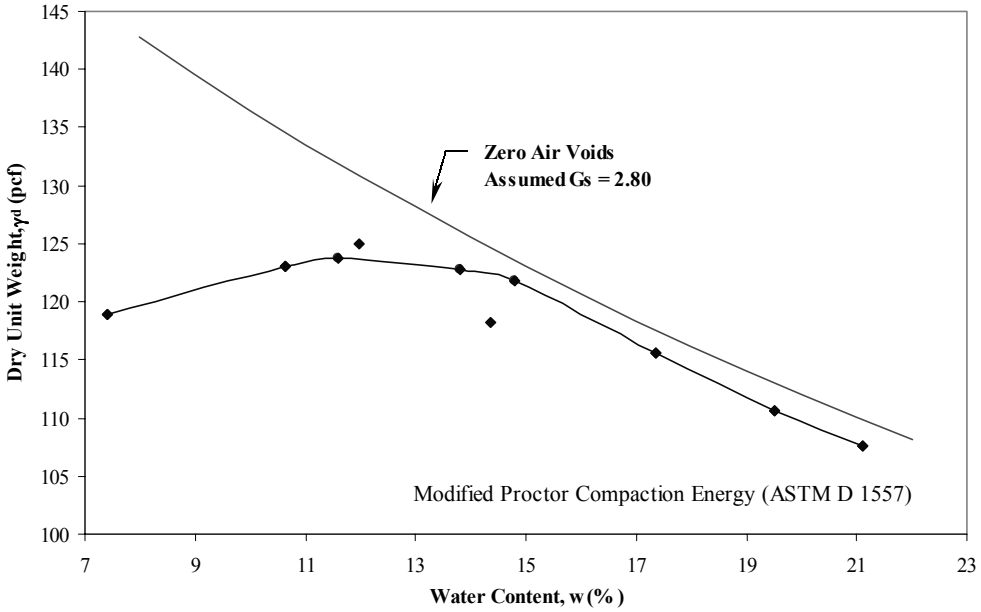

Figure J-2. Compaction test results on bulk illite

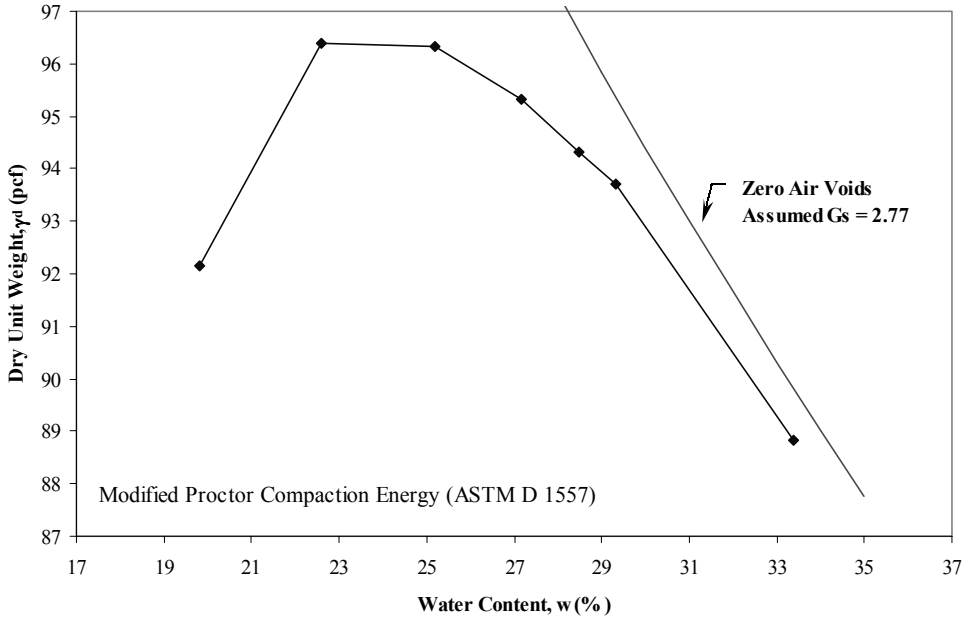

Figure J-3. Compaction test results on bulk montmorillonite

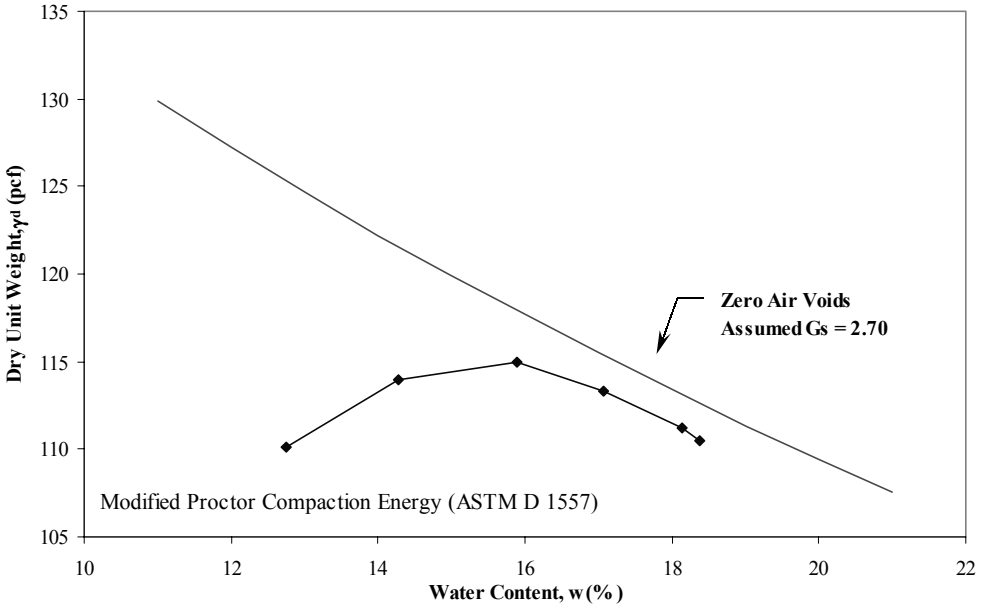

Figure J-4. Compaction test results on TX Bryan HP

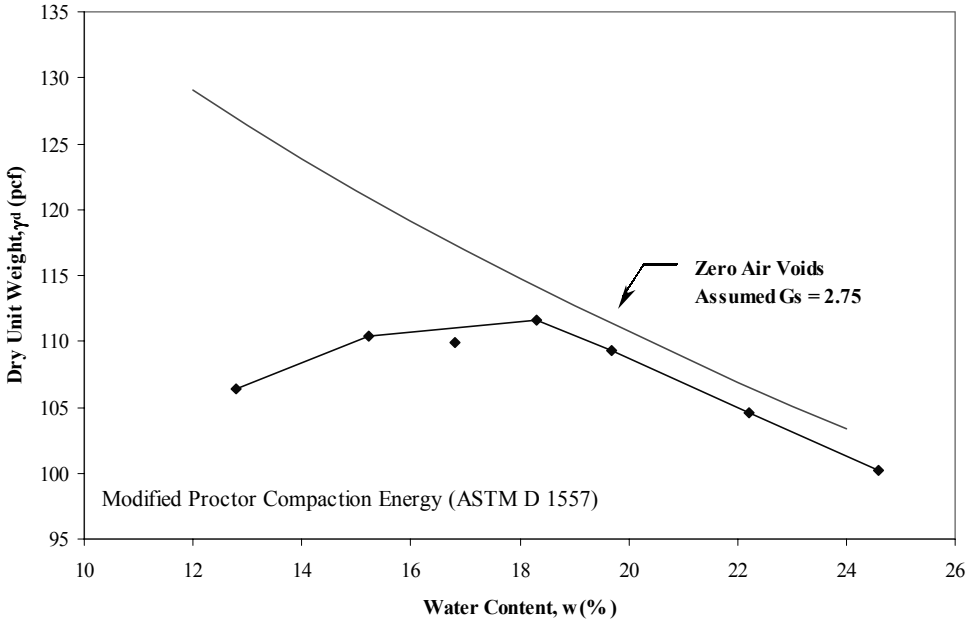

Figure J-5. Compaction test results on TX Mesquite HS HP

APPENDIX K

INDEX PROPERTIES OF TRIAXIAL AND FREE SWELL BULK TEST SPECIMENS

Table K-1. Properties of kaolinite test specimens

Kaolinite $G_s = 2.62$	*Test Specimens*	*Dry Unit Weight (pcf)*	*Water Content (%)*	*Saturation (%)*	*Void Ratio*
Untreated	Compaction Optimum	98.7	24	95.8	0.66
Untreated	UU Triaxial	99.45	23.50	95.65	0.64
	UU Triaxial	99.22	23.27	94.12	0.65
	UU Triaxial	100.49	23.62	98.71	0.63
	UU Triaxial	101.56	23.57	101.28	0.61
	Free Swell	97.97	21.97	85.95	0.67
	Free Swell	99.81	22.03	90.37	0.64
	Free Swell	102.08	22.01	95.72	0.60
Treated with Ionic Product	UU Triaxial	95.20	23.70	86.58	0.72
	UU Triaxial	93.97	23.41	82.93	0.74
	UU Triaxial	94.82	23.38	84.58	0.72
	UU Triaxial	94.87	23.94	86.73	0.72
	Free Swell	92.84	22.67	77.98	0.76
	Free Swell	97.58	22.70	87.96	0.68
	Free Swell	97.28	22.55	86.69	0.68
Treated with Enzyme Product	UU Triaxial	97.22	24.04	92.41	0.68
	UU Triaxial	95.94	24.13	89.78	0.70
	UU Triaxial	96.54	24.11	91.10	0.69
	UU Triaxial	96.75	24.09	91.48	0.69
	Free Swell	97.85	23.50	91.67	0.67
	Free Swell	100.53	23.92	99.98	0.63
	Free Swell	97.37	23.46	90.42	0.68
Treated with Polymer Product	UU Triaxial	95.38	23.18	85.05	0.71
	UU Triaxial	95.20	23.10	84.38	0.72
	UU Triaxial	96.17	23.29	87.17	0.70
	UU Triaxial	95.91	23.18	86.19	0.70
	Free Swell	96.77	22.43	85.13	0.69
	Free Swell	98.62	22.16	88.20	0.66
	Free Swell	96.73	22.32	84.66	0.69

Table K-2. Properties of illite test specimens

Illite $G_s = 2.80$	*Test Specimens*	*Dry Unit Weight (pcf)*	*Water Content (%)*	*Saturation (%)*	*Void Ratio*
Untreated	Compaction Optimum	124.5	12	83.3	0.40
Untreated	UU Triaxial	118.30	10.84	63.64	0.48
	UU Triaxial	120.78	10.19	63.86	0.45
	UU Triaxial	121.70	10.68	68.66	0.44
	Free Swell	125.03	10.19	71.68	0.40
	Free Swell	127.51	10.19	76.98	0.37
	Free Swell	124.24	9.97	68.64	0.41
Treated with Ionic Product	UU Triaxial	122.95	11.05	73.47	0.42
	UU Triaxial	122.87	11.24	74.59	0.42
	UU Triaxial	122.07	11.20	72.69	0.43
	UU Triaxial	123.65	11.50	77.97	0.41
	Free Swell	122.65	11.35	74.74	0.43
	Free Swell	125.44	11.17	79.52	0.39
	Free Swell	126.04	11.12	80.53	0.39
Treated with Enzyme Product	UU Triaxial	123.19	10.33	69.15	0.42
	UU Triaxial	123.99	10.35	70.83	0.41
	UU Triaxial	121.60	10.23	65.55	0.44
	UU Triaxial	123.37	10.27	69.07	0.42
	Free Swell	126.32	10.48	76.45	0.38
	Free Swell	126.24	10.42	75.83	0.38
	Free Swell	124.96	10.04	70.51	0.40
Treated with Polymer Product	UU Triaxial	122.40	13.62	89.20	0.43
	UU Triaxial	121.73	13.60	87.50	0.44
	UU Triaxial	122.18	13.59	88.46	0.43
	UU Triaxial	121.88	13.64	88.10	0.43
	Free Swell	123.83	12.27	83.49	0.41
	Free Swell	124.43	12.25	84.75	0.40
	Free Swell	124.90	12.26	85.92	0.40

Table K-3. Properties of montmorillonite test specimens

Montmorillonite $G_s = 2.77$	*Test Specimens*	*Dry Unit Weight (pcf)*	*Water Content (%)*	*Saturation (%)*	*Void Ratio*
Untreated	Compaction Optimum	96.8	24	84.6	0.79
Untreated	UU Triaxial	88.13	22.68	65.35	0.96
	UU Triaxial	90.36	22.92	69.54	0.91
	UU Triaxial	89.17	22.92	67.65	0.94
	UU Triaxial	88.38	23.03	66.76	0.96
	Free Swell	94.42	22.49	74.94	0.83
	Free Swell	92.26	22.45	71.14	0.87
	Free Swell	90.38	22.15	67.18	0.91
Treated with Ionic Product	UU Triaxial	90.69	23.96	73.27	0.91
	UU Triaxial **	87.59	23.84	67.84	0.97
	UU Triaxial	89.76	24.06	72.00	0.93
	UU Triaxial	90.74	24.01	73.52	0.90
	Free Swell	87.70	22.26	63.46	0.97
	Free Swell	94.30	22.48	74.67	0.83
	Free Swell	90.14	22.07	66.58	0.92
Treated with Enzyme Product	UU Triaxial	88.24	23.64	68.29	0.96
	UU Triaxial	89.68	23.42	69.96	0.93
	UU Triaxial	90.31	23.58	71.46	0.91
	UU Triaxial	88.37	23.44	67.91	0.96
	Free Swell **	89.54	23.17	68.91	0.93
	Free Swell	89.05	23.13	68.01	0.94
	Free Swell	92.23	22.66	71.75	0.87
Treated with Polymer Product	UU Triaxial	97.16	22.51	80.05	0.78
	UU Triaxial	87.30	22.71	64.19	0.98
	UU Triaxial	90.30	22.67	68.71	0.91
	UU Triaxial	80.69	22.76	55.21	1.14
	Free Swell **	97.07	22.40	79.42	0.78
	Free Swell	93.95	22.45	73.98	0.84
	Free Swell	91.28	22.11	68.48	0.89

** *Test results not used in the evaluation of engineering soil properties.*

Table K-4. Properties of TX Bryan HP test specimens

TX Bryan HP $G_s = 2.70$	*Test Specimens*	*Dry Unit Weight (pcf)*	*Water Content (%)*	*Saturation (%)*	*Void Ratio*
Untreated	Compaction Optimum	115.0	16	92.9	0.47
Untreated	UU Triaxial	114.38	14.8	84.59	0.47
	UU Triaxial	113.94	15.1	84.95	0.48
	UU Triaxial **	112.88	14.8	80.92	0.49
	UU Triaxial	114.65	14.8	84.92	0.47
	Free Swell	112.15	15.74	84.52	0.50
	Free Swell	113.75	15.80	88.55	0.48
	Free Swell	114.99	16.09	93.25	0.47
Treated with Ionic Product	UU Triaxial **	109.78	17.4	87.82	0.53
	UU Triaxial	109.69	17.3	87.37	0.54
	UU Triaxial	111.53	17.1	90.36	0.51
	UU Triaxial	109.14	16.9	84.05	0.54
	Free Swell	114.62	14.75	84.63	0.47
	Free Swell	116.83	14.78	90.15	0.44
	Free Swell	114.10	14.87	84.14	0.48
Treated with Enzyme Product	UU Triaxial	109.22	18.2	90.36	0.54
	UU Triaxial	108.42	18.2	88.53	0.55
	UU Triaxial	108.22	18.3	88.69	0.56
	UU Triaxial	109.23	18.3	91.01	0.54
	Free Swell	108.56	19.00	92.81	0.55
	Free Swell	110.54	17.63	90.68	0.52
	Free Swell	109.28	16.83	83.80	0.54
Treated with Polymer Product	UU Triaxial	114.50	15.3	87.86	0.47
	UU Triaxial	114.27	15.4	87.70	0.47
	UU Triaxial	114.21	15.4	87.38	0.48
	UU Triaxial **	114.03	15.2	86.20	0.48
	Free Swell	114.83	15.37	88.68	0.47
	Free Swell	118.31	14.97	95.17	0.42
	Free Swell	114.16	15.42	87.39	0.48

** *Test results not used in the evaluation of engineering soil properties.*

Table K-5. Properties of TX Mesquite HS HP test specimens

TX Mesquite HS HP $G_s = 2.75$	*Test Specimens*	*Dry Unit Weight (pcf)*	*Water Content (%)*	*Saturation (%)*	*Void Ratio*
Untreated	Compaction Optimum	112.0	17	87.9	0.53
Untreated	UU Triaxial	110.41	16.76	83.19	0.55
	UU Triaxial **	109.36	16.89	81.60	0.57
	UU Triaxial	110.66	17.00	84.90	0.55
	UU Triaxial	110.54	16.87	84.00	0.55
	Free Swell	112.97	16.25	86.01	0.52
	Free Swell	115.48	16.28	92.03	0.49
	Free Swell	112.95	16.12	85.27	0.52
Treated with Ionic Product	UU Triaxial **	97.60	17.56	63.69	0.76
	UU Triaxial	96.90	17.71	63.17	0.77
	UU Triaxial	95.72	17.78	61.69	0.79
	UU Triaxial	95.65	17.79	61.62	0.79
	Free Swell	110.67	16.58	82.69	0.55
	Free Swell	113.18	16.63	88.47	0.52
	Free Swell	112.79	15.97	84.14	0.52
Treated with Enzyme Product	UU Triaxial	108.92	14.90	71.21	0.58
	UU Triaxial	109.25	15.15	73.01	0.57
	UU Triaxial	109.16	15.05	72.36	0.57
	UU Triaxial	108.60	15.14	71.78	0.58
	Free Swell	109.42	15.24	73.66	0.57
	Free Swell	113.02	15.40	81.63	0.52
	Free Swell	108.35	15.51	72.95	0.58
Treated with Polymer Product	UU Triaxial	111.49	16.49	84.10	0.54
	UU Triaxial	111.68	16.49	84.49	0.54
	UU Triaxial	111.23	16.49	83.53	0.54
	UU Triaxial	110.12	16.29	80.27	0.56
	Free Swell	110.04	16.81	82.53	0.56
	Free Swell	112.40	16.64	86.77	0.53
	Free Swell	111.88	16.71	86.00	0.53

** *Test results not used in the evaluation of engineering soil properties.*

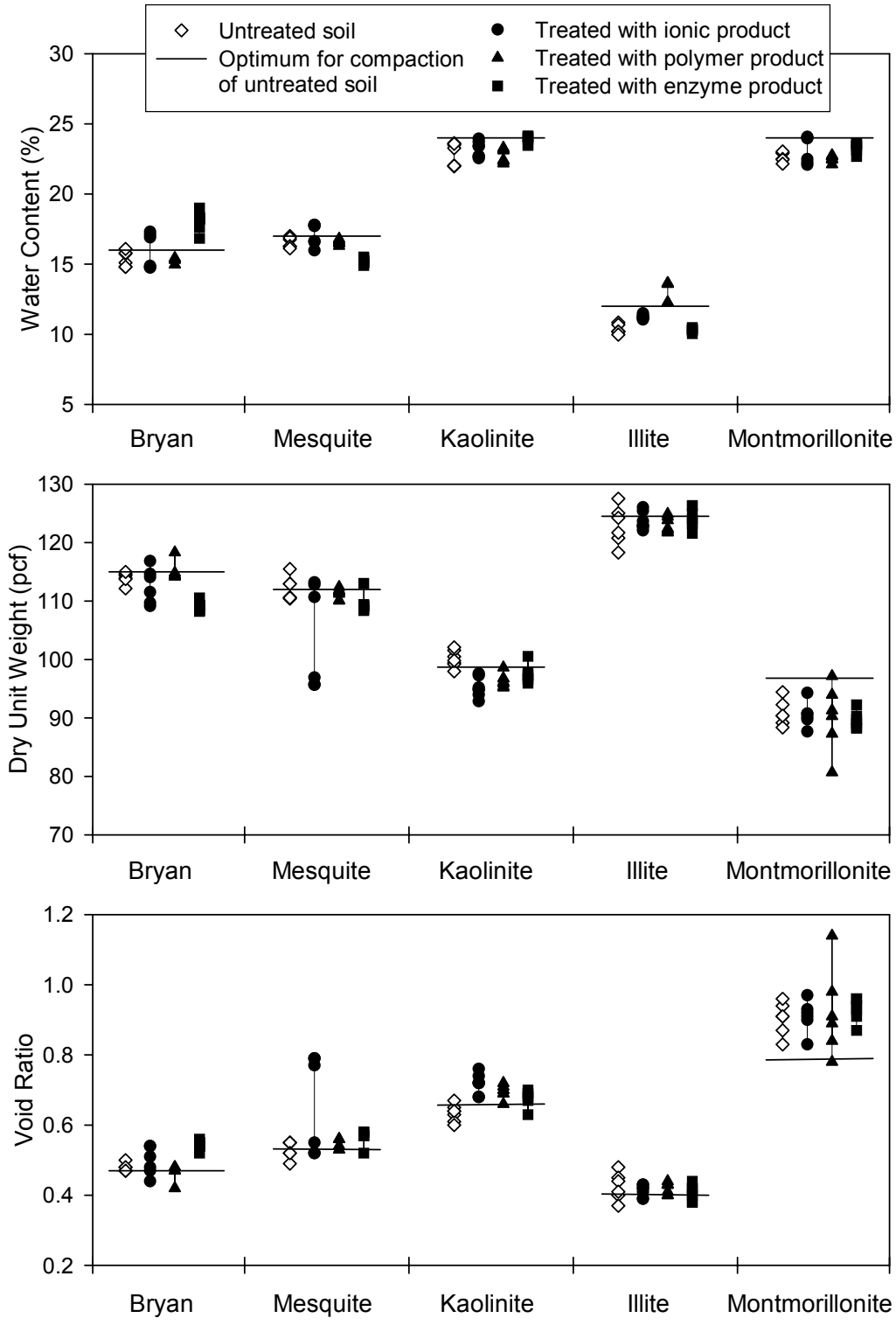

Figure K-1. Summary of the water content, dry unit weight, and void ratio of all triaxial and swell test specimens

APPENDIX L

RESULTS FROM UNCONSOLIDATED-UNDRAINED TRIAXIAL COMPRESSION TESTS ON UNTREATED AND TREATED BULK TEST SOILS

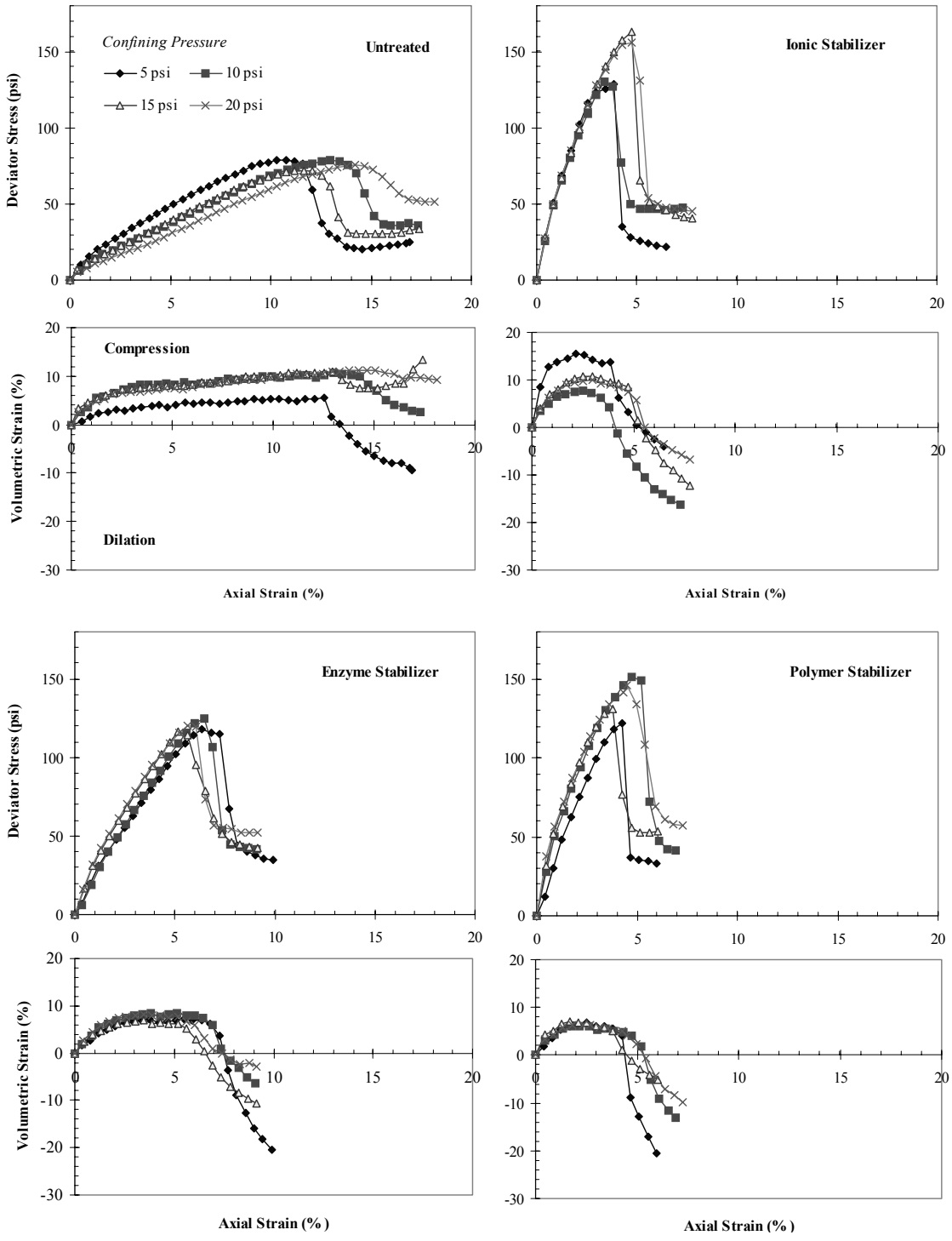

Figure L-1. Results from unconsolidated-undrained triaxial compression tests on untreated and treated bulk kaolinite

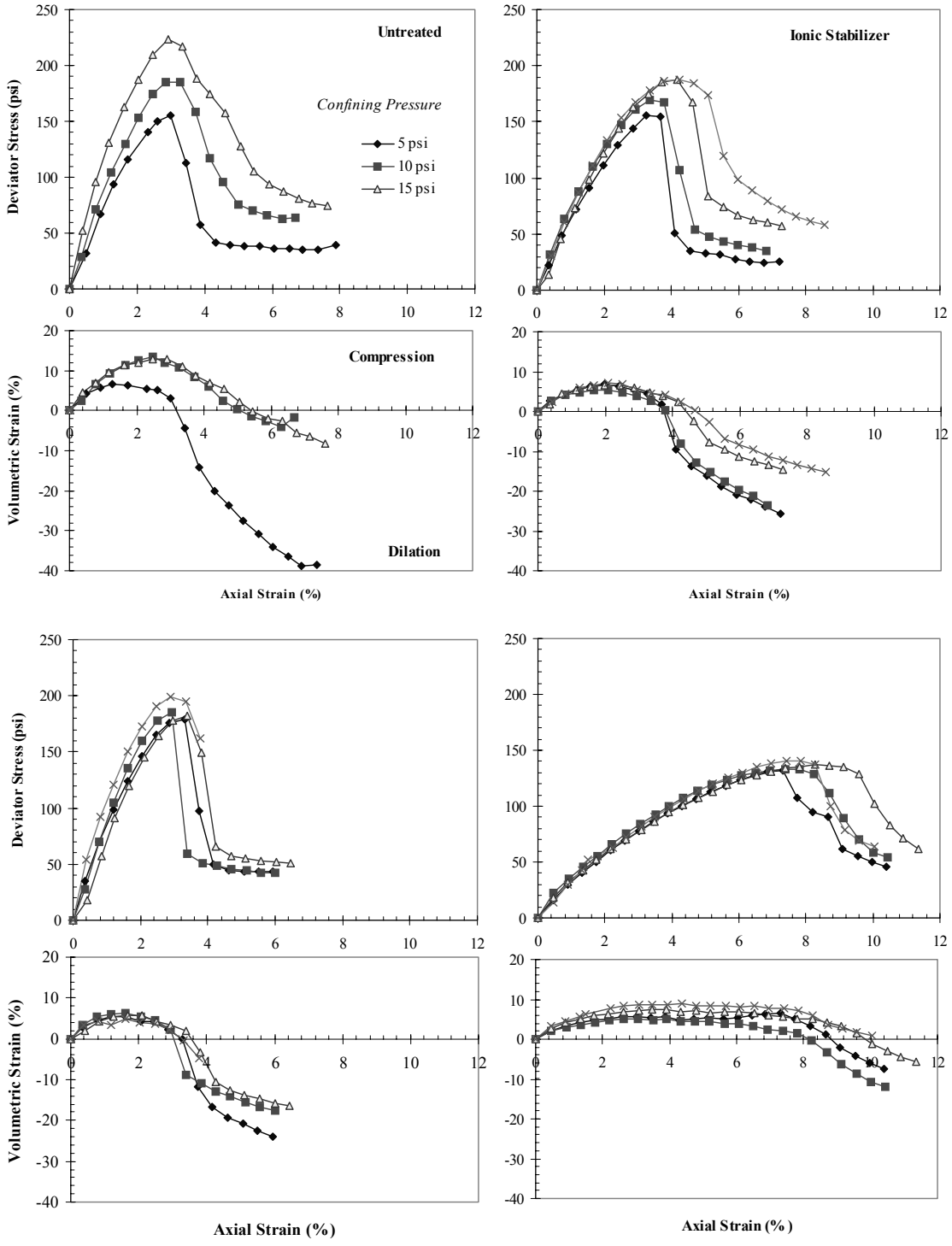

Figure L-2. Results from unconsolidated-undrained triaxial compression tests on untreated and treated bulk illite

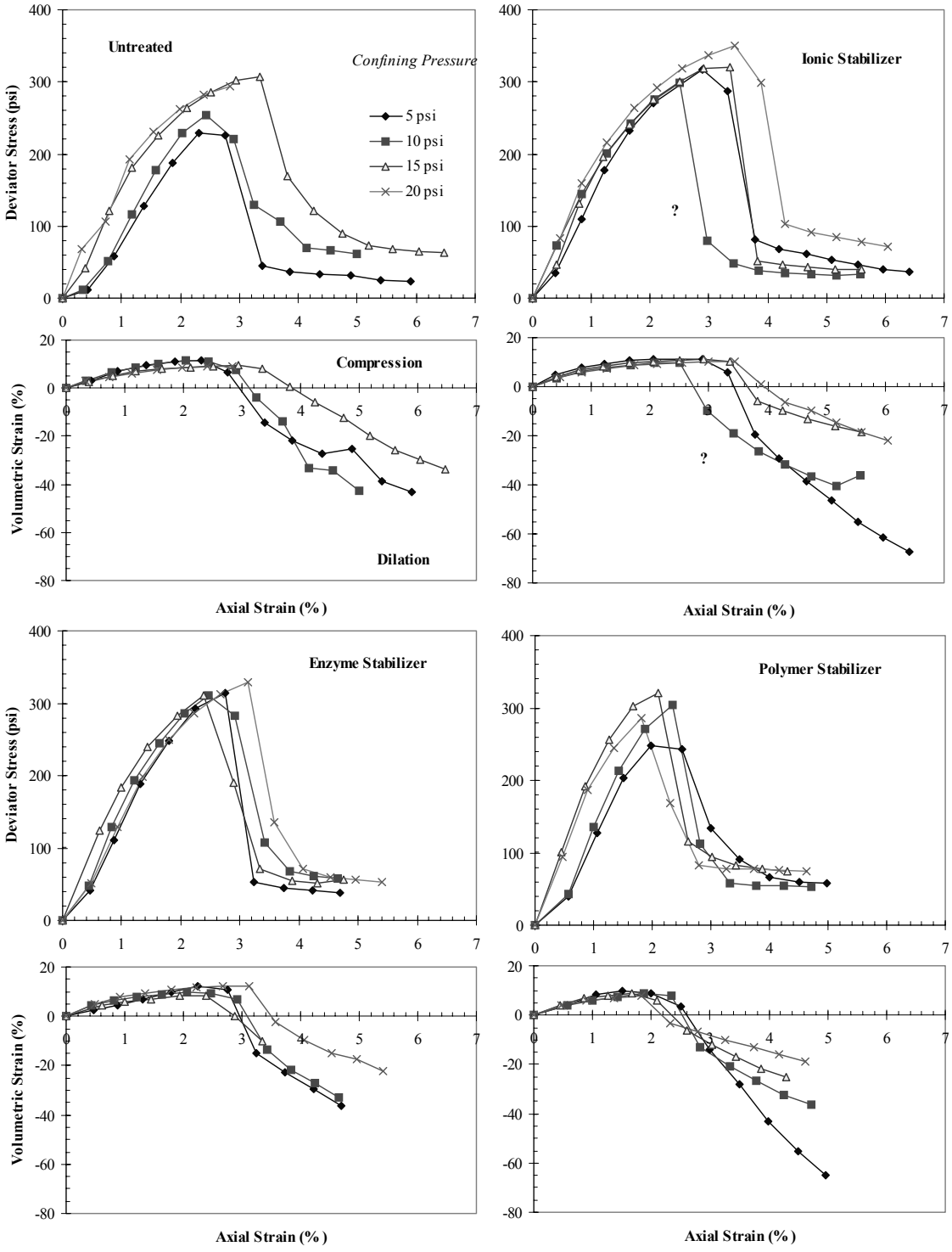

Figure L-3. Results from unconsolidated-undrained triaxial compression tests on untreated and treated bulk montmorillonite

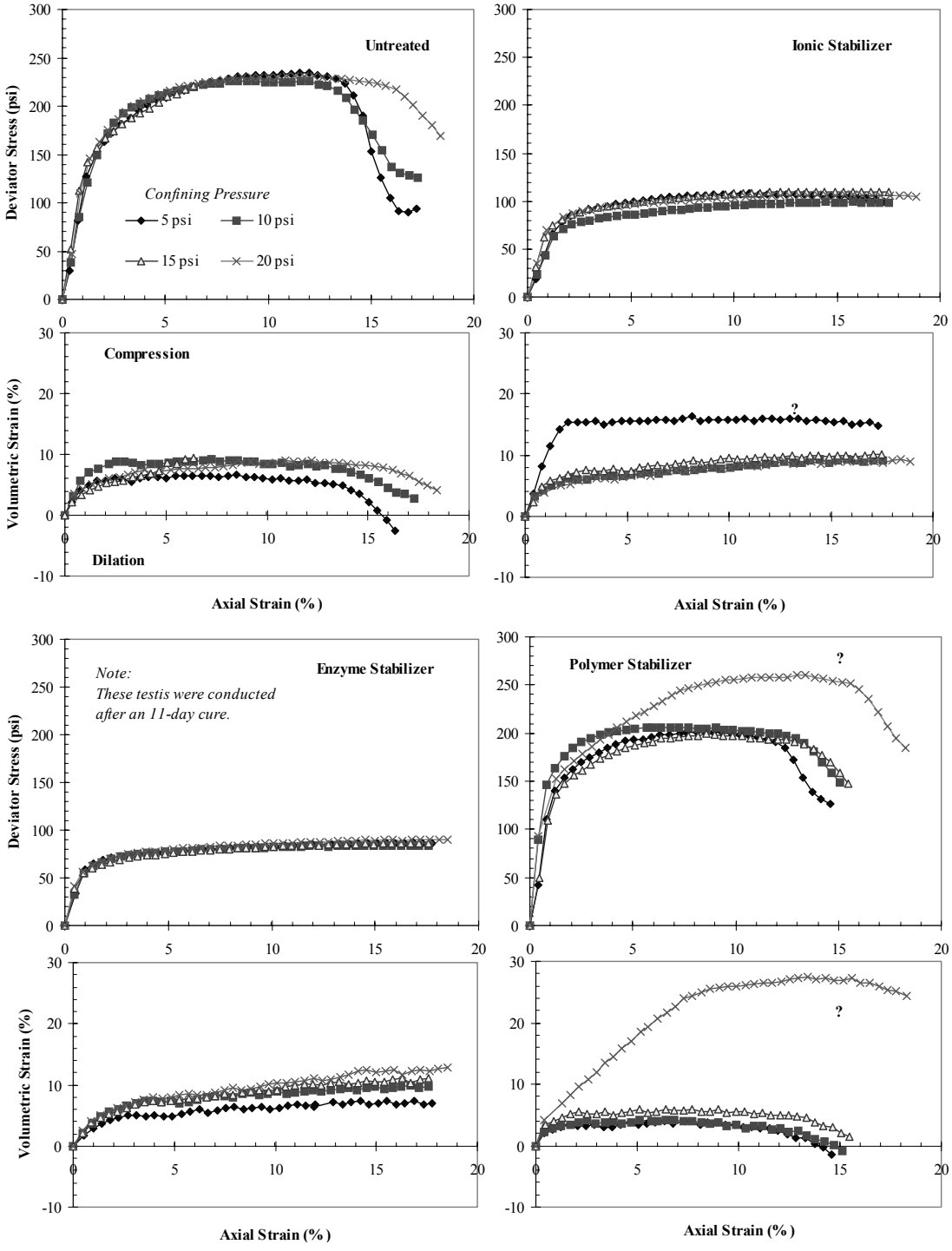

Figure L-4. Results from unconsolidated-undrained triaxial compression tests on untreated and treated TX Bryan HP

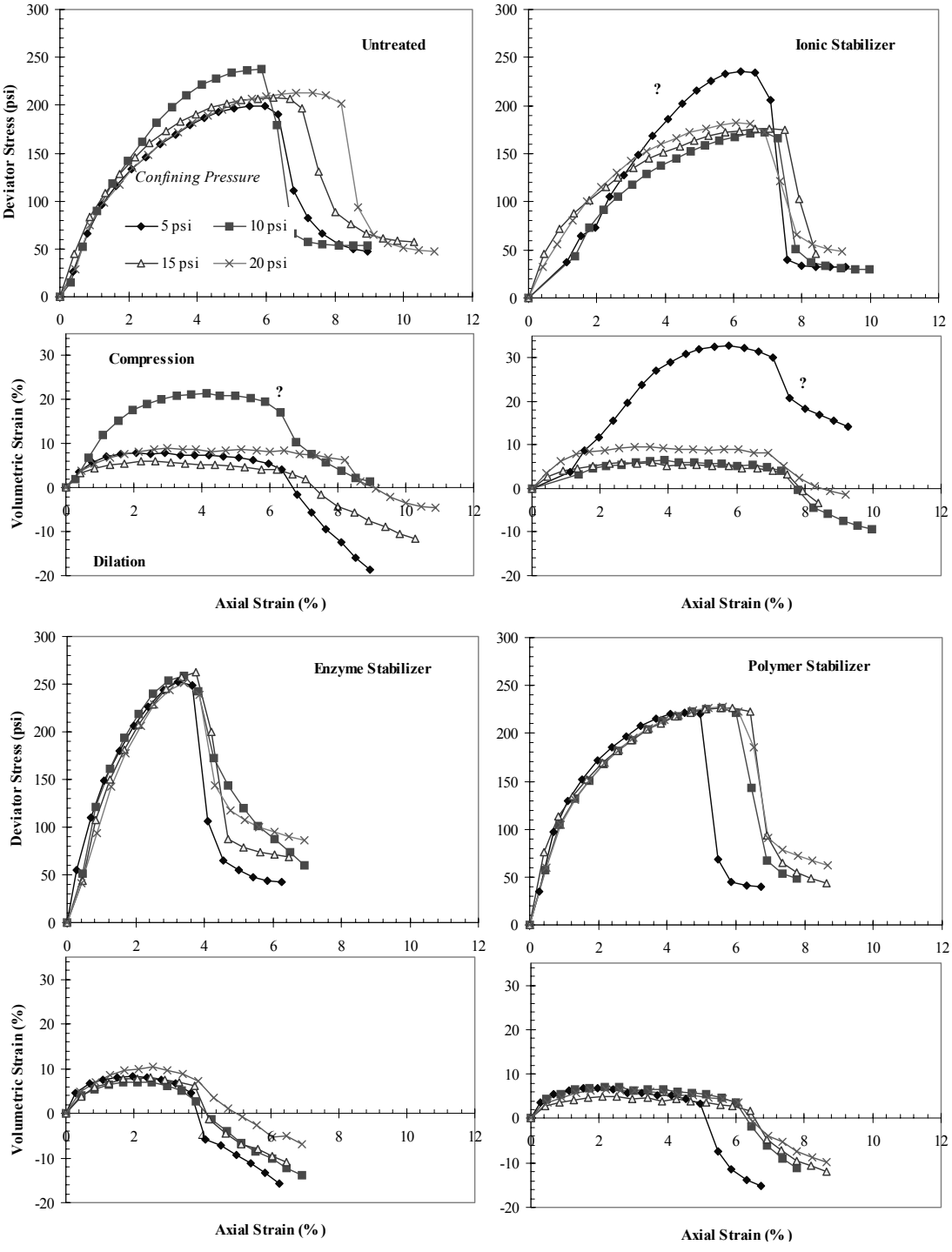

Figure L-5. Results from unconsolidated-undrained triaxial compression tests on untreated and treated TX Mesquite HS HP

APPENDIX M

SHEAR STRENGTH ENVELOPES FIT TO UU TRIAXIAL TEST DATA

Table M-1. Shear strength parameters for envelopes fitted to the UU triaxial data in Figures M-1 through M-5

Test Soil	*Untreated Soil*		*Ionic Product*		*Enzyme Product*		*Polymer Product*	
	c (psi)	ϕ *(deg)*	*c (psi)*	ϕ *(deg)*	*c (psi)*	ϕ *(deg)*	*c (psi)*	ϕ *(deg)*
Kaolinite	38	0	28	35	56	3	31	31
Illite	21	51	39	33	55	24	50	14
Montmorillonite	32	51	79	33	101	22	39	49
TX Bryan HP	113	1	38	13	37	7	96	3
TX Mesquite HS HP	71	18	57	20	111	7	94	9

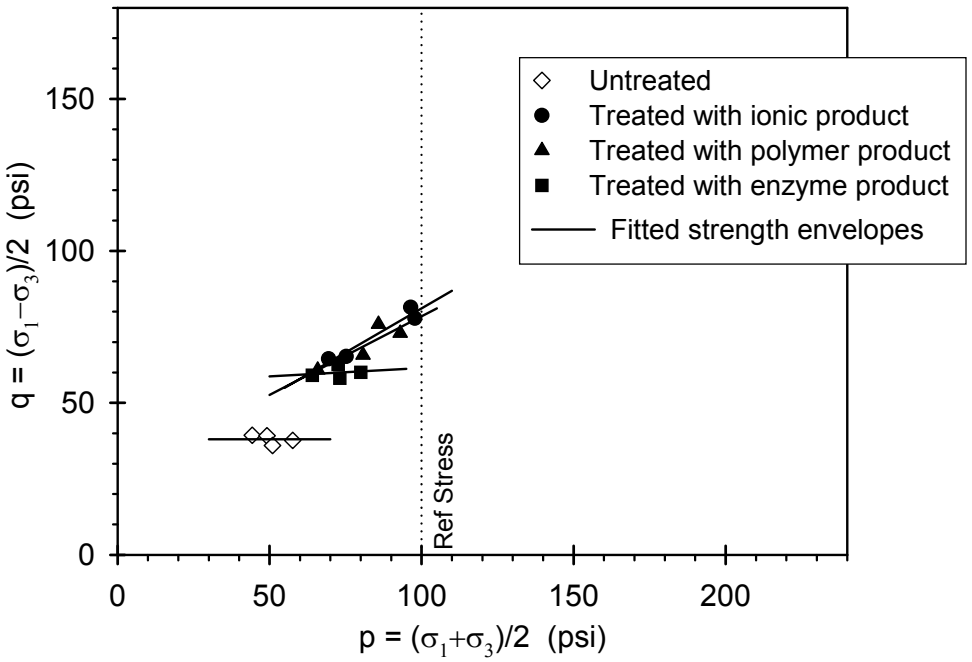

Figure M-1. Fitted shear strength envelopes from UU triaxial tests on untreated and treated bulk kaolinite

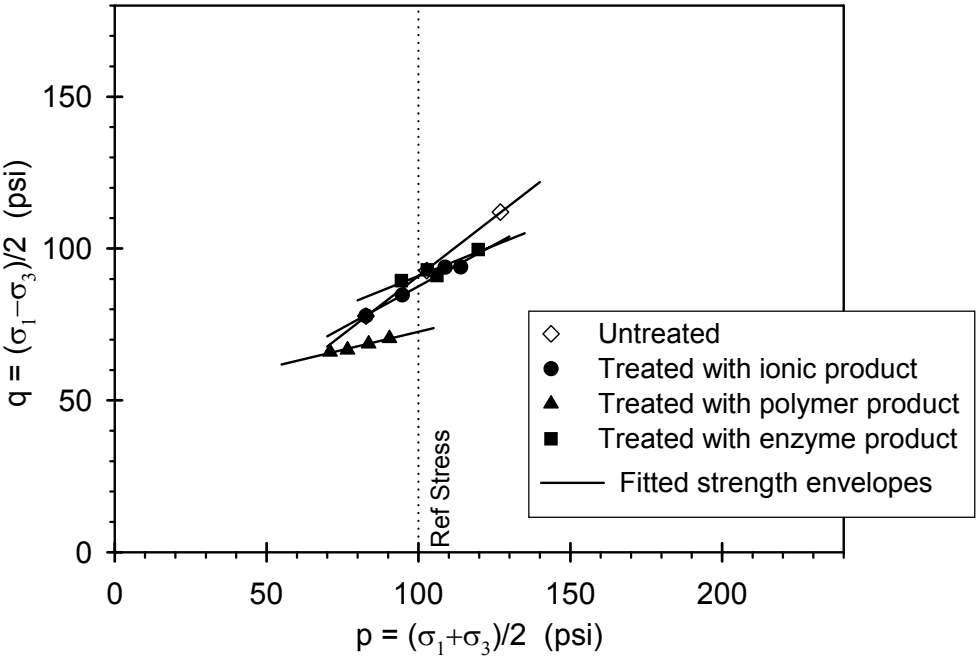

Figure M-2. Fitted shear strength envelopes from UU triaxial tests on untreated and treated bulk illite

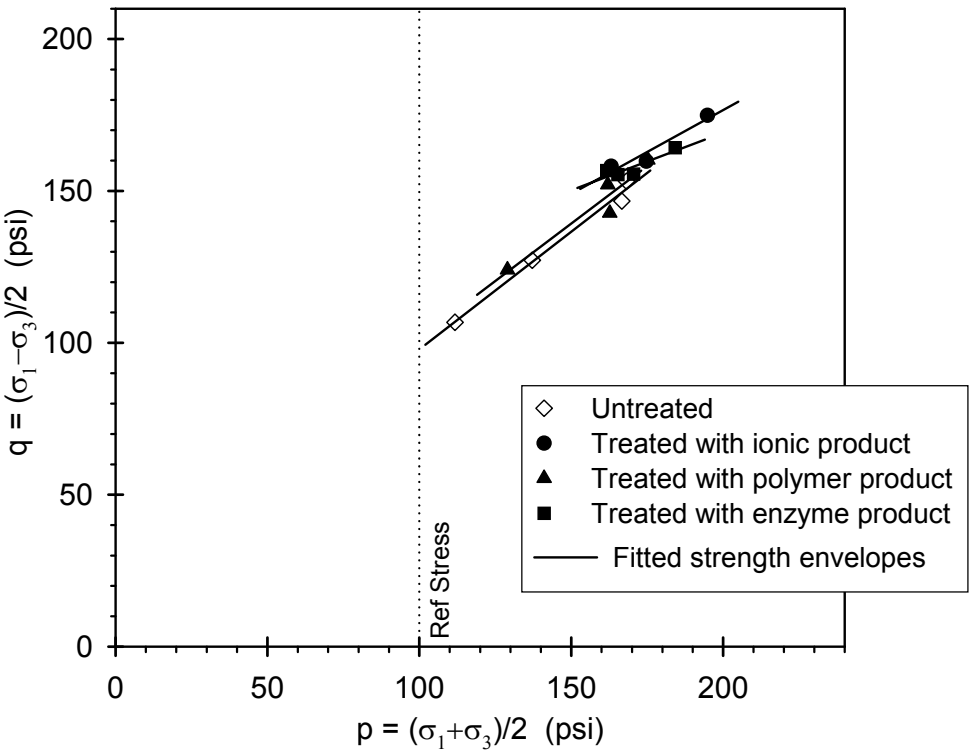

Figure M-3. Fitted shear strength envelopes from UU triaxial tests on untreated and treated bulk montmorillonite

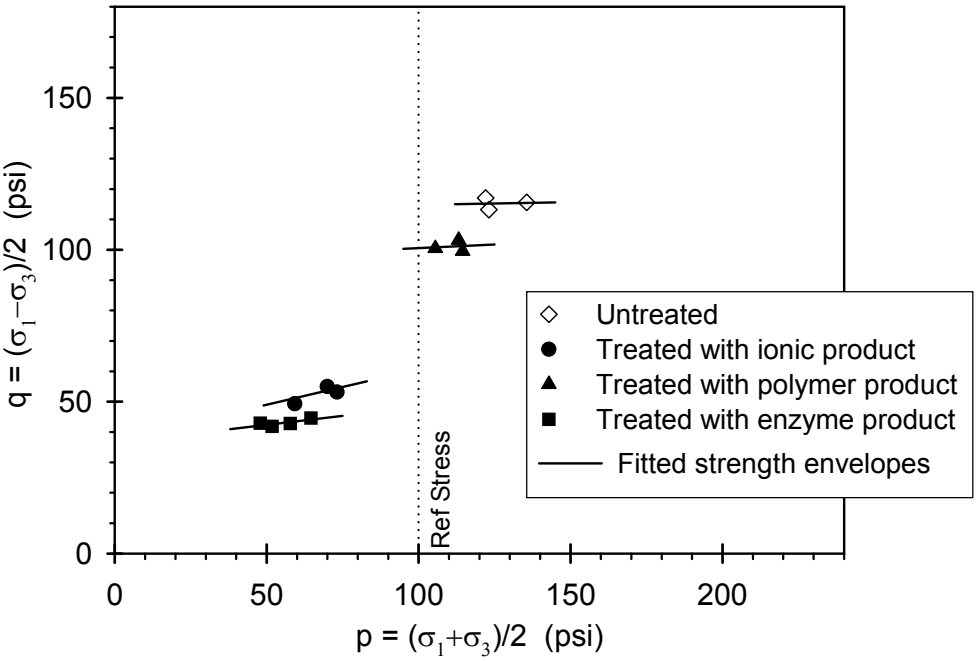

Figure M-4. Fitted shear strength envelopes from UU triaxial tests on untreated and treated TX Bryan HP

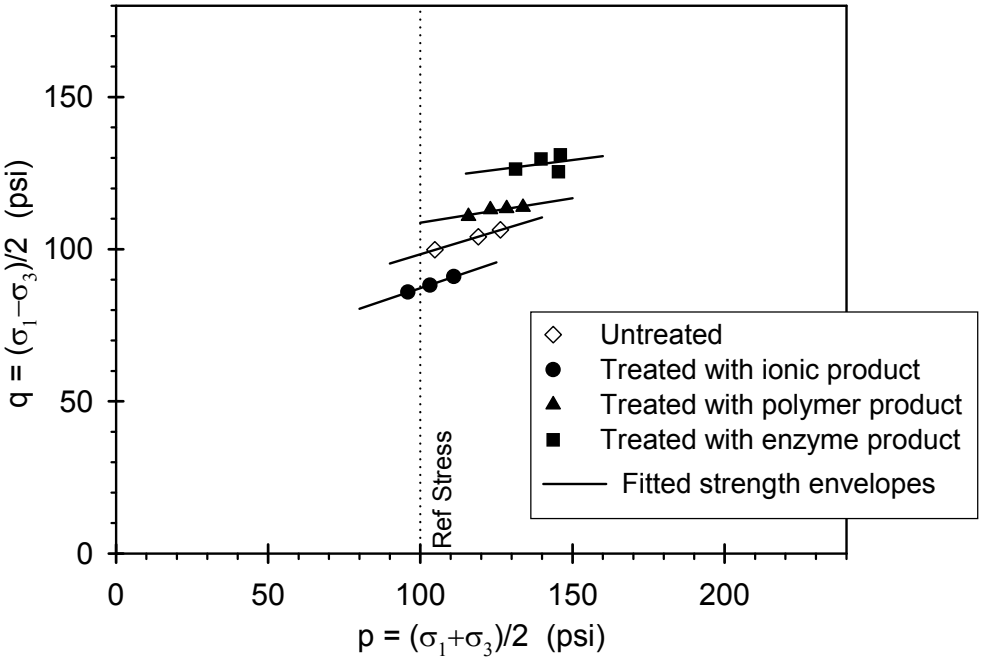

Figure M-5. Fitted shear strength envelopes from UU triaxial tests on untreated and treated TX Mesquite HS HP

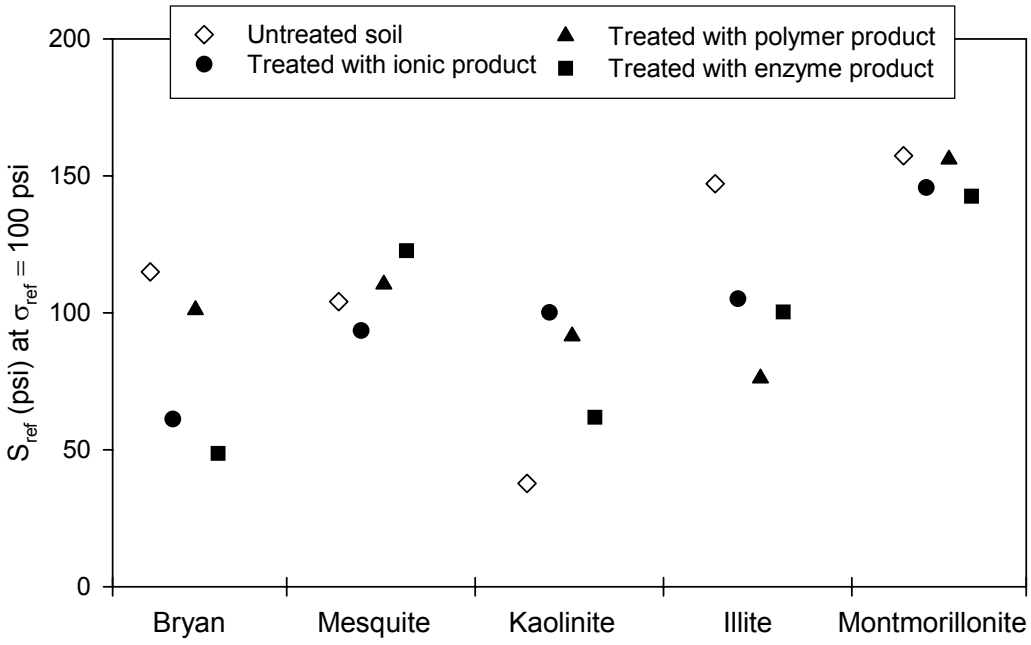

Figure M-6. Comparison of the reference shear strengths measured for all soils and all treatments

APPENDIX N

RESULTS FROM ONE-DIMENSIONAL FREE SWELL TESTS ON UNTREATED AND TREATED BULK TEST SOILS

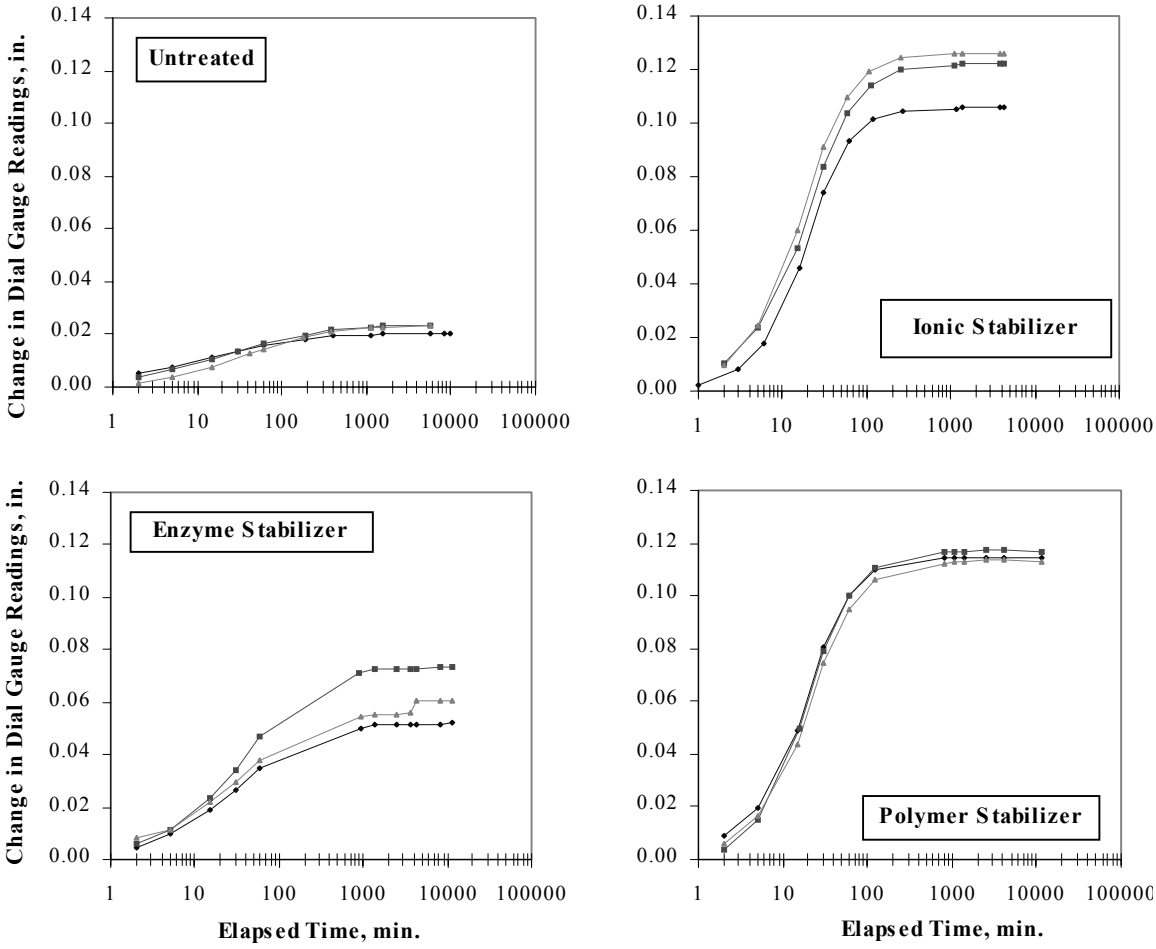

Figure N-1. Results from 1-D free swell tests on untreated and treated bulk kaolinite

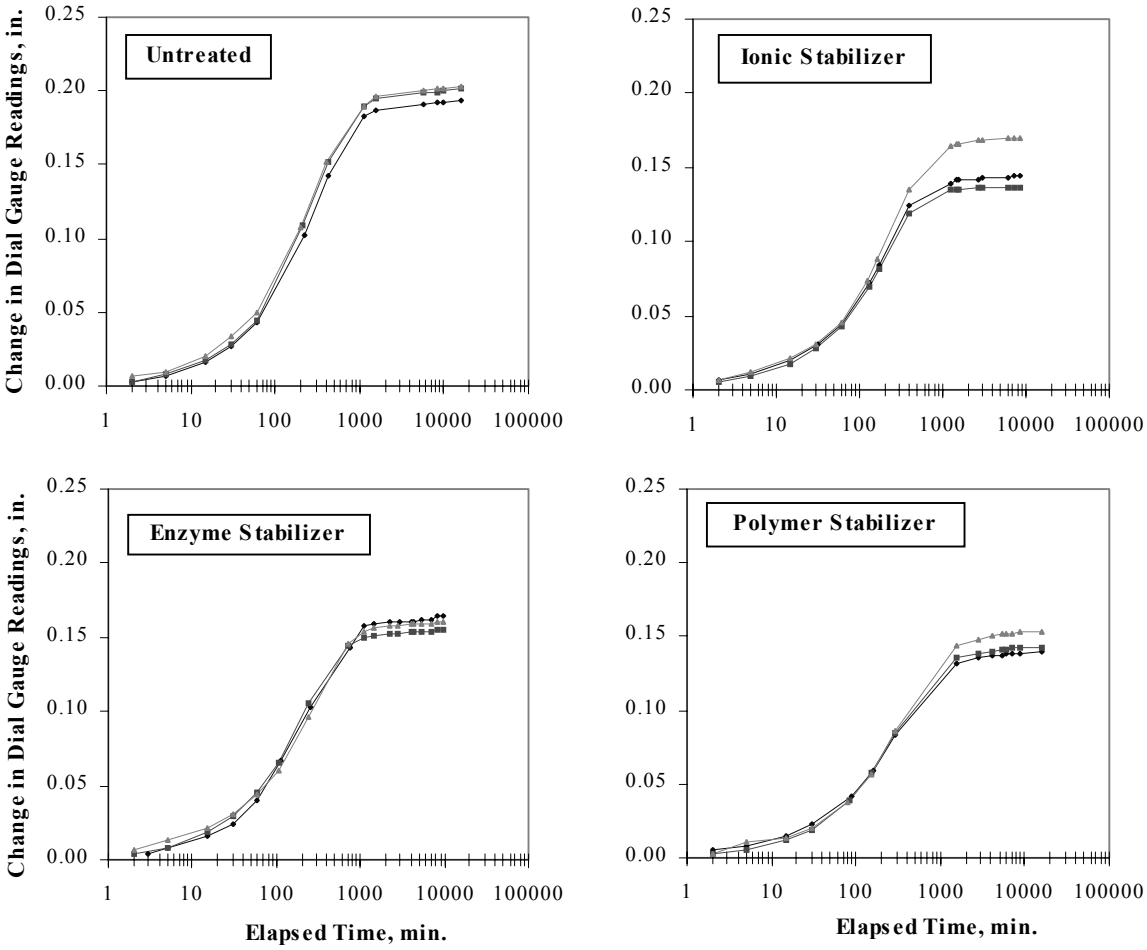

Figure N-2. Results from 1-D free swell tests on untreated and treated bulk illite

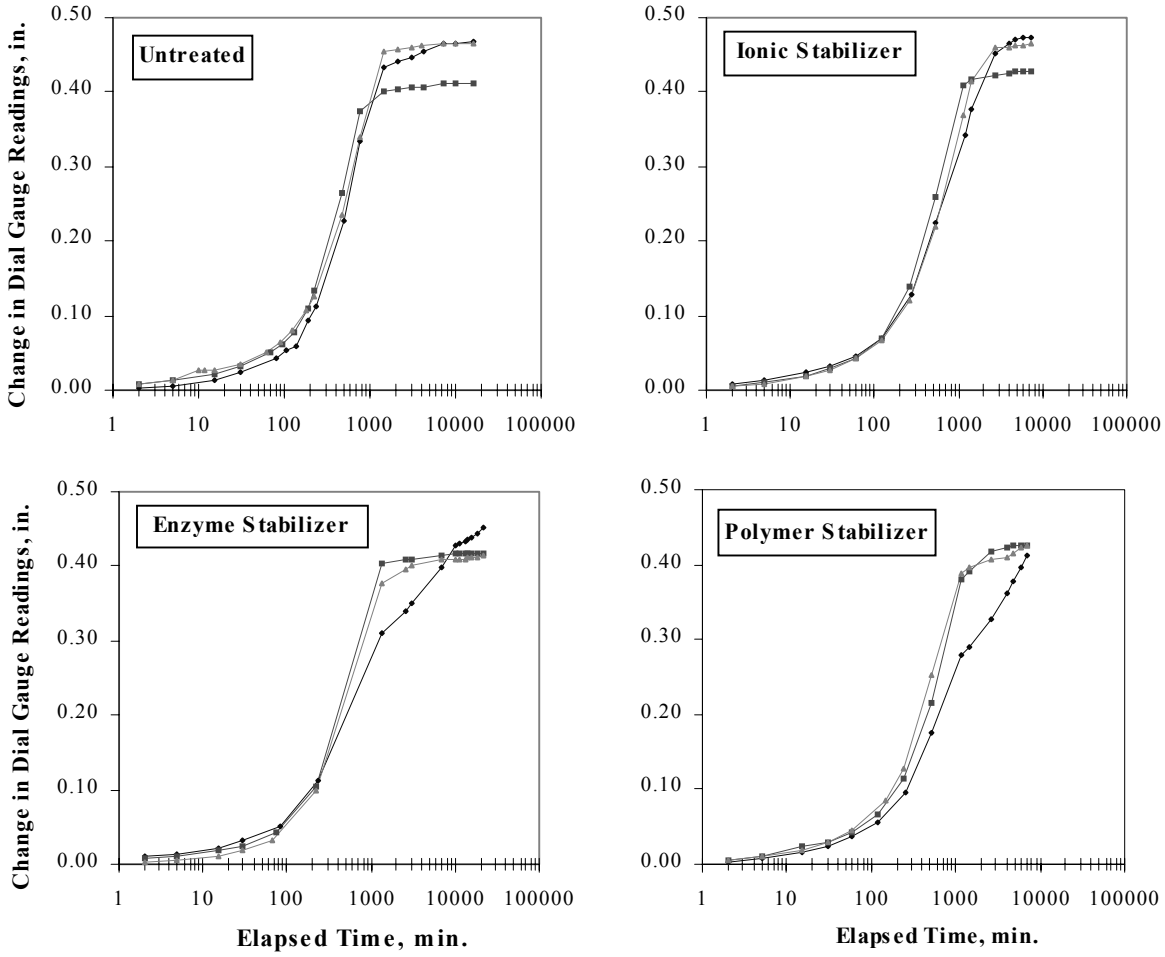

Figure N-3. Results from 1-D free swell tests on untreated and treated bulk montmorillonite (initial sample height = 0.40 inch)

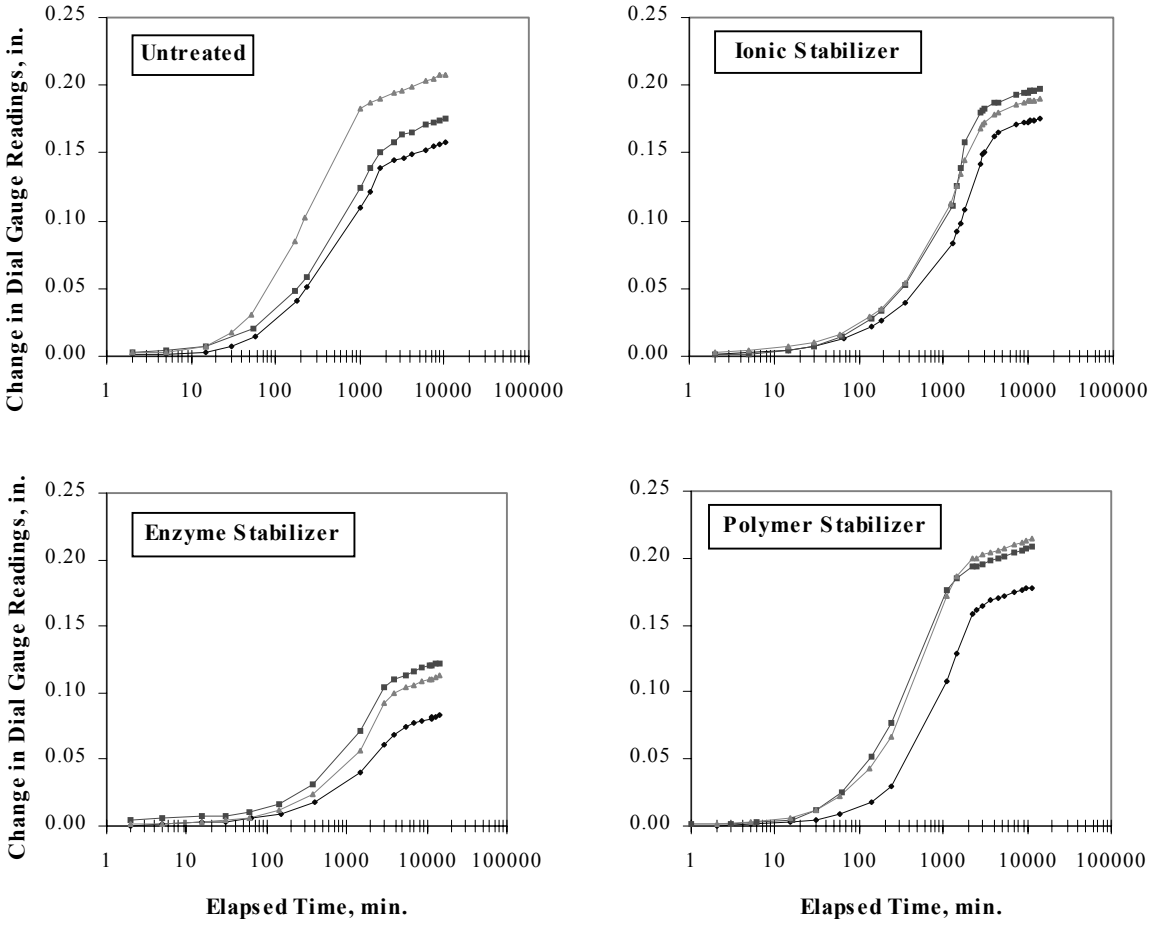

Figure N-4. Results from 1-D free swell tests on untreated and treated TX Bryan HP

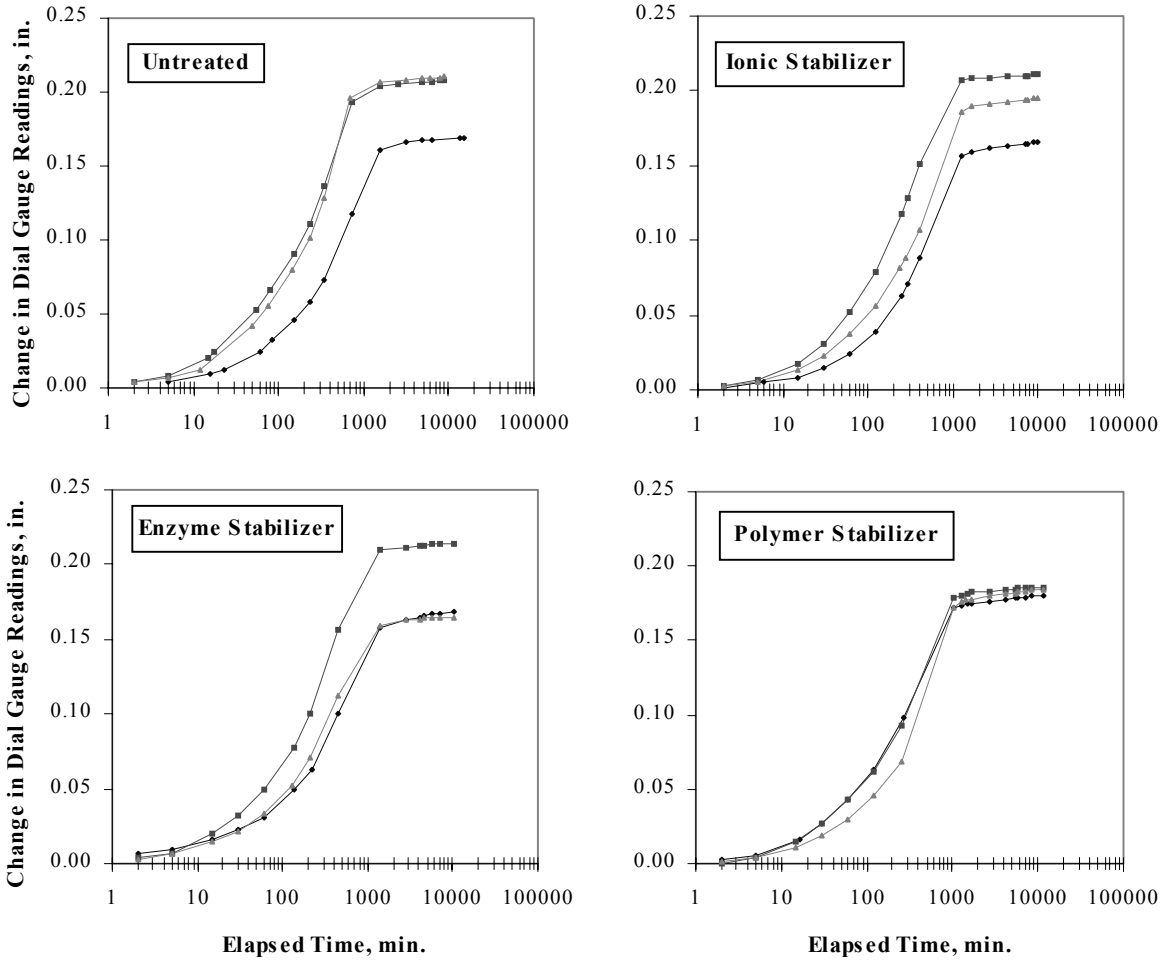

Figure N-5. Results from 1-D free swell tests on untreated and treated TX Mesquite HS HP

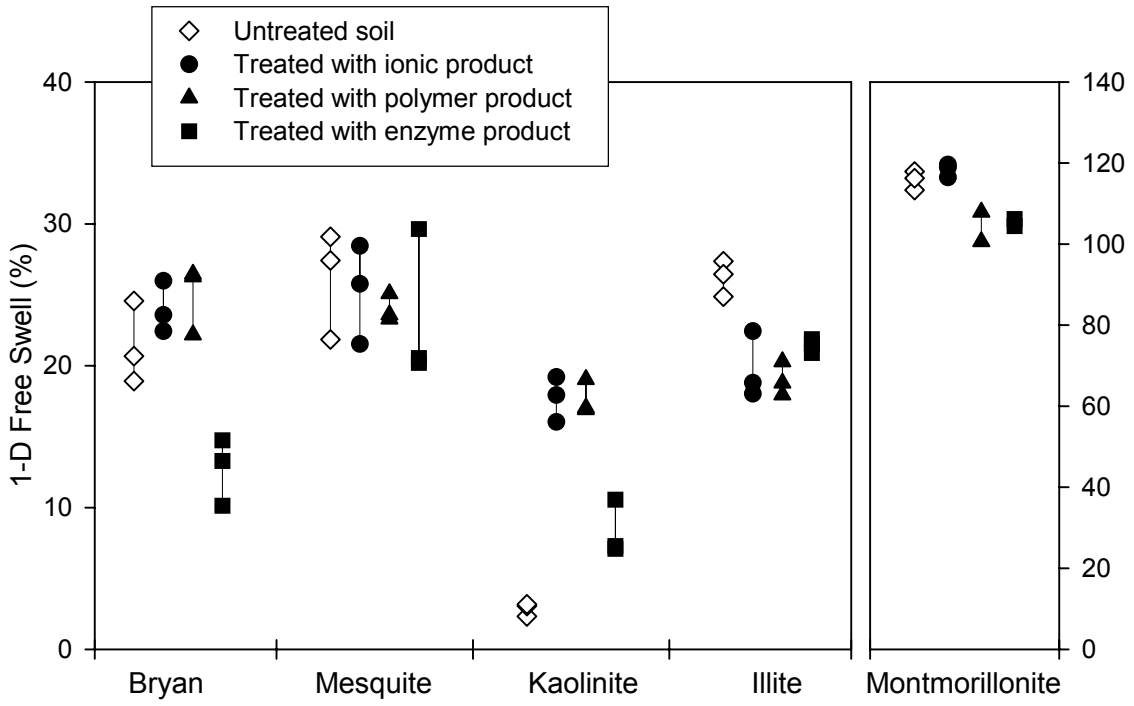

Figure N-6. Comparison of the 1-D free swell measured for all soils and all treatments

APPENDIX O

TEST RESULTS FROM FOLLOW-UP STUDY USING STABILIZERS AT HIGH APPLICATION RATES AND LIME

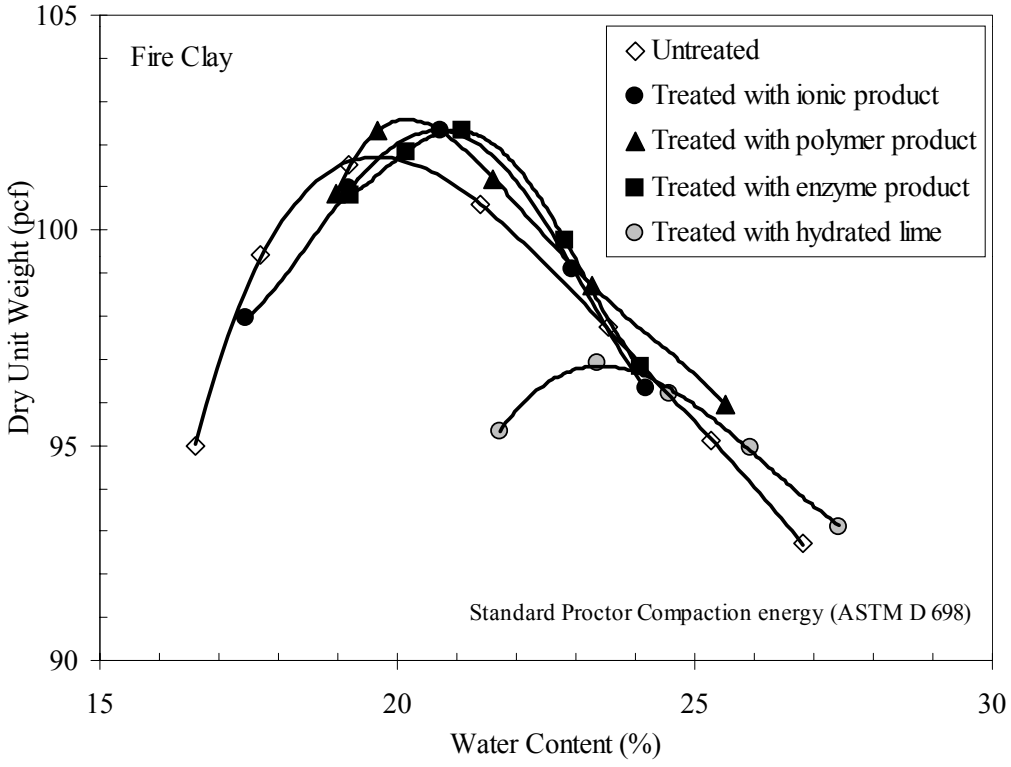

Figure O-1. Compaction test results on Fire clay

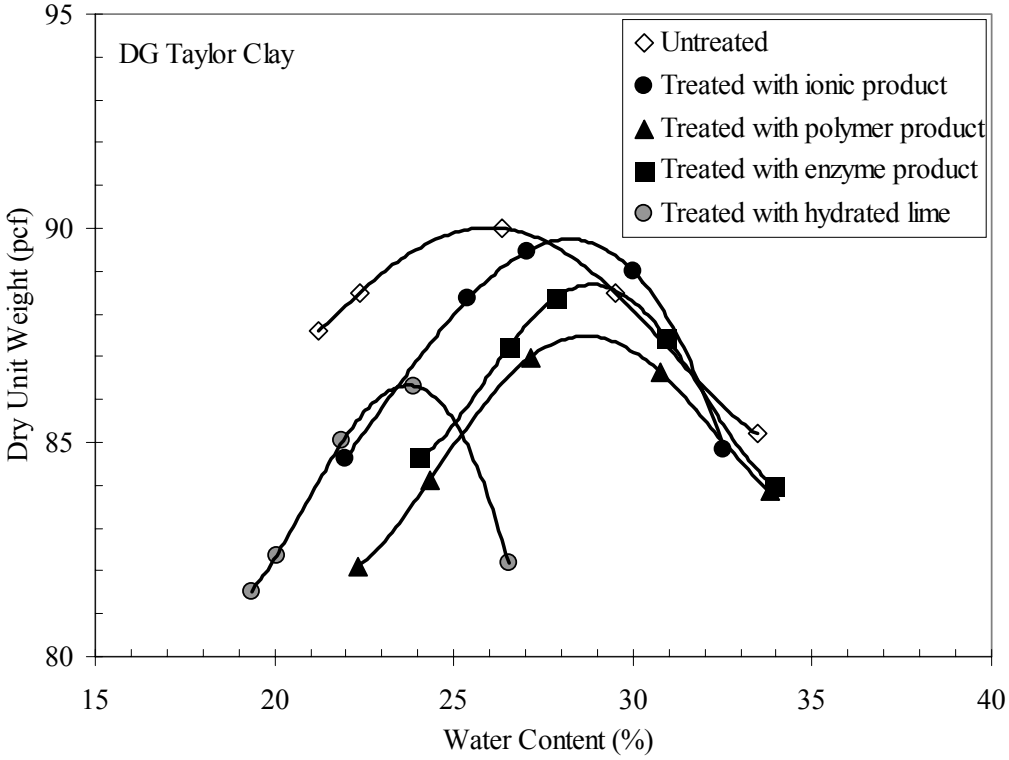

Figure O-2. Compaction test results on DG Taylor clay

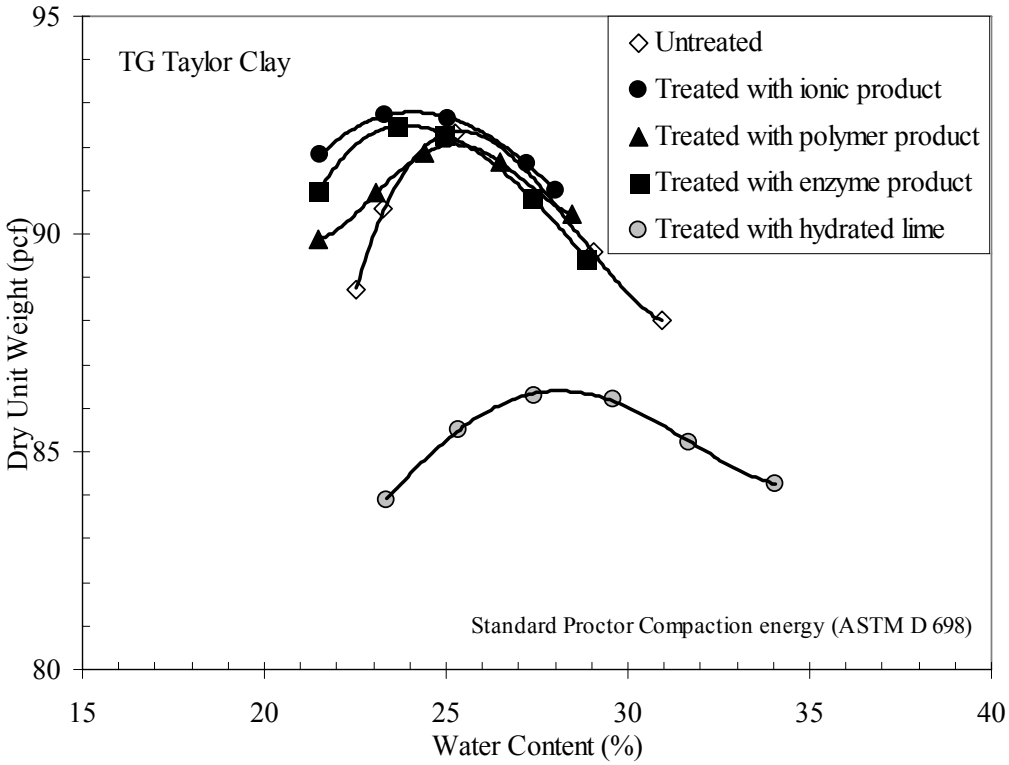

Figure O-3. Compaction test results on TG Taylor clay

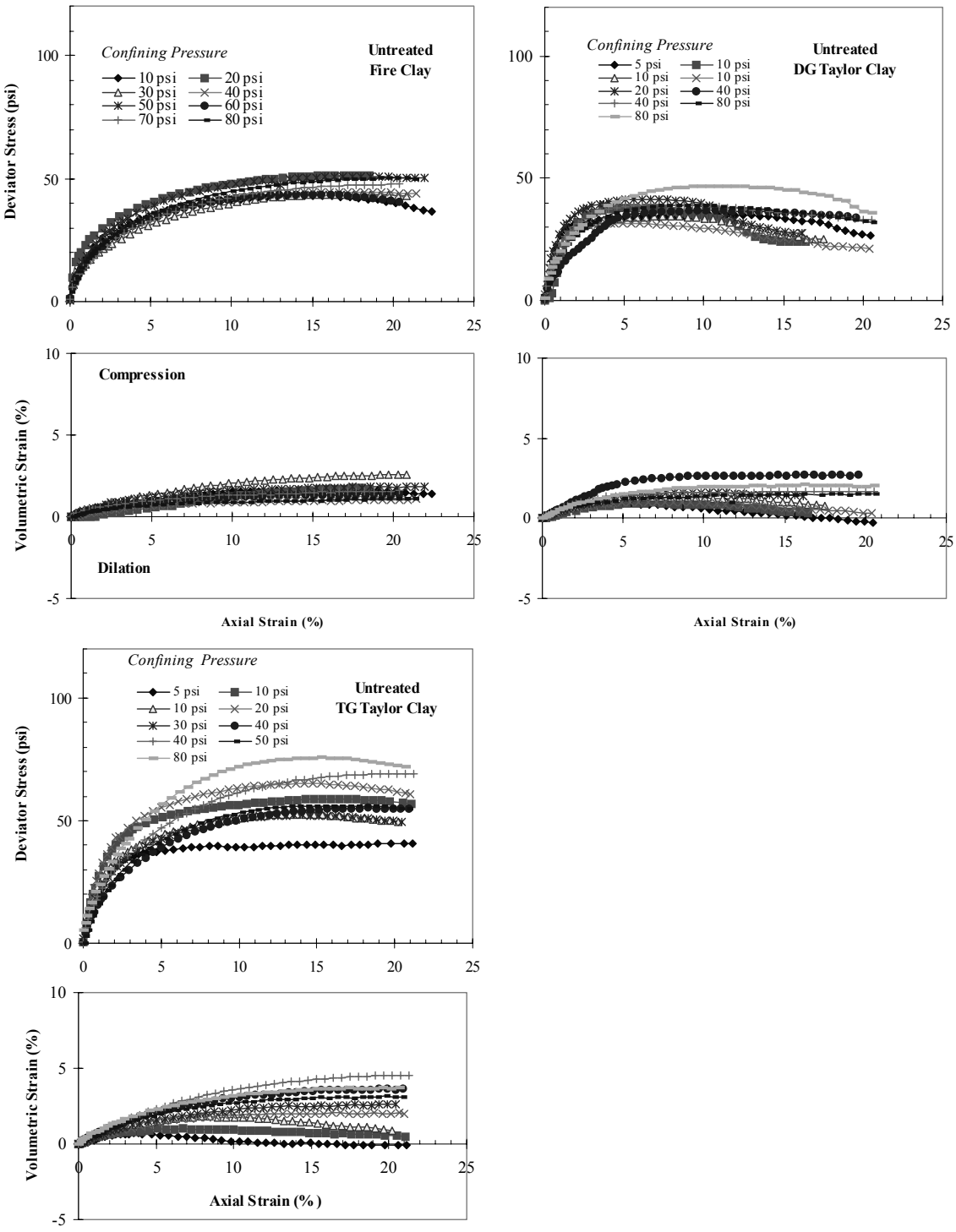

Figure O-4. Results from unconsolidated-undrained triaxial compression tests on the untreated soil in the follow-up study

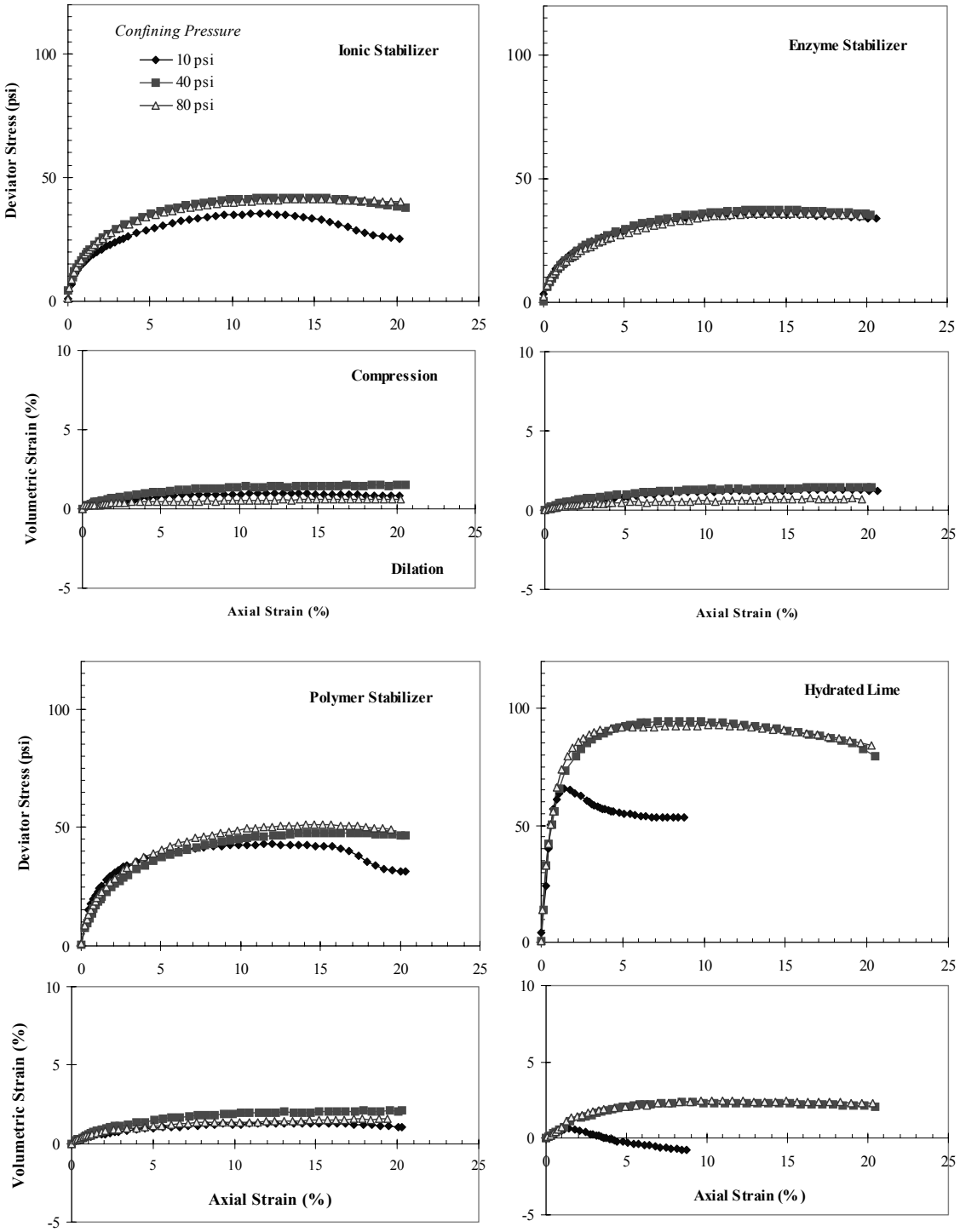

Figure O-5. Results from unconsolidated-undrained triaxial compression tests on treated Fire clay in the follow-up study

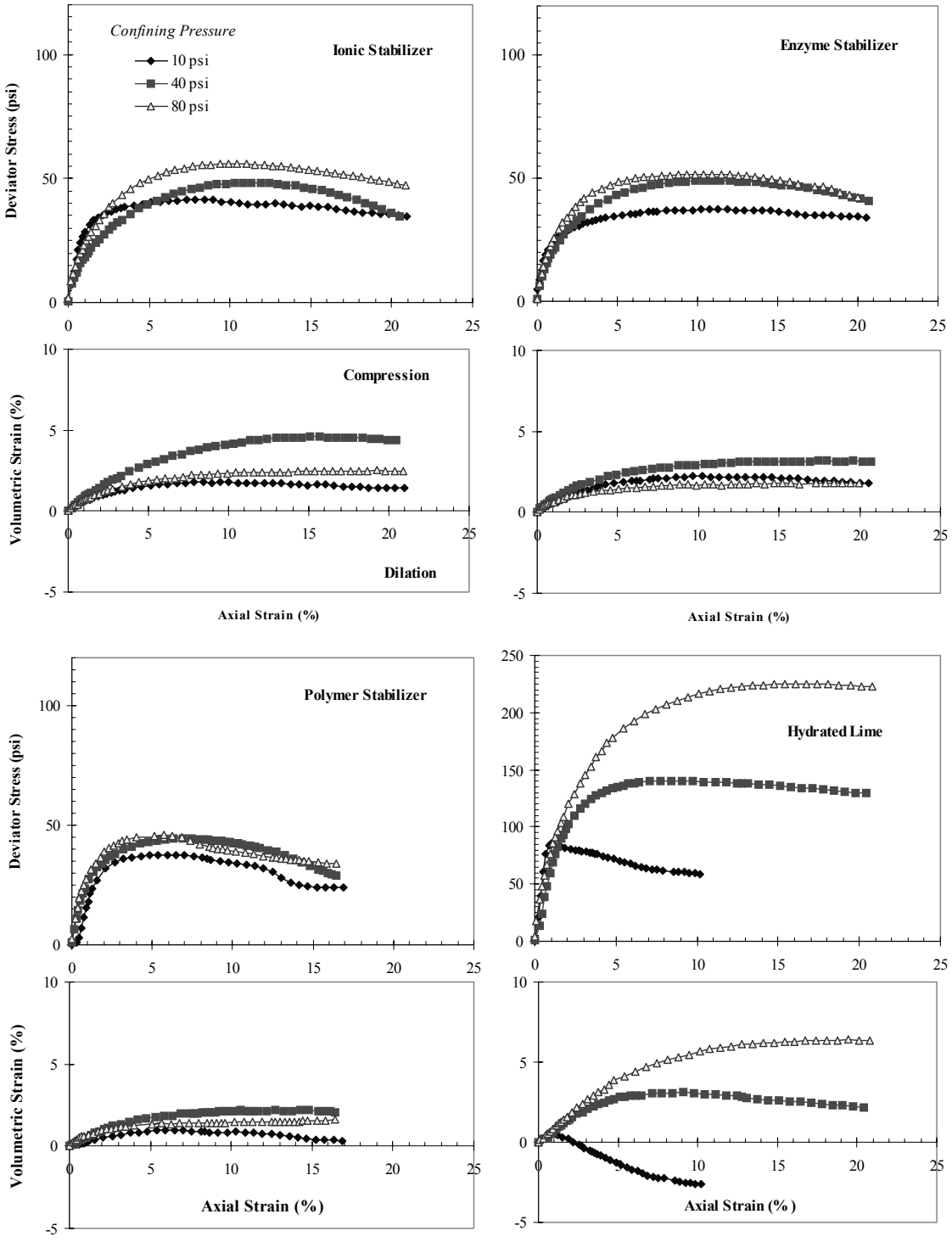

Figure O-6. Results from unconsolidated-undrained triaxial compression tests on treated DG Taylor clay in the follow-up study

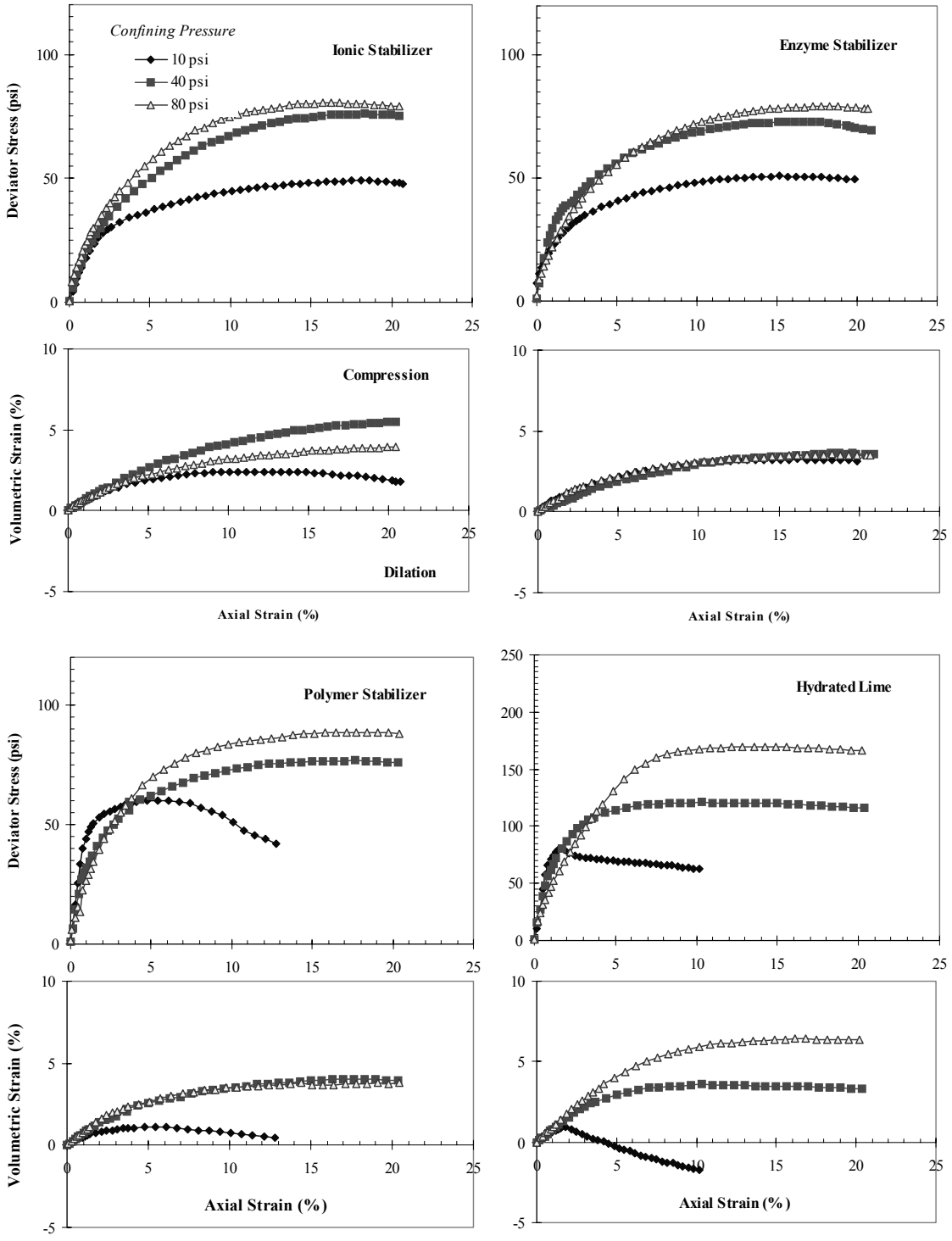

Figure O-7. Results from unconsolidated-undrained triaxial compression tests on treated TG Taylor clay in the follow-up study

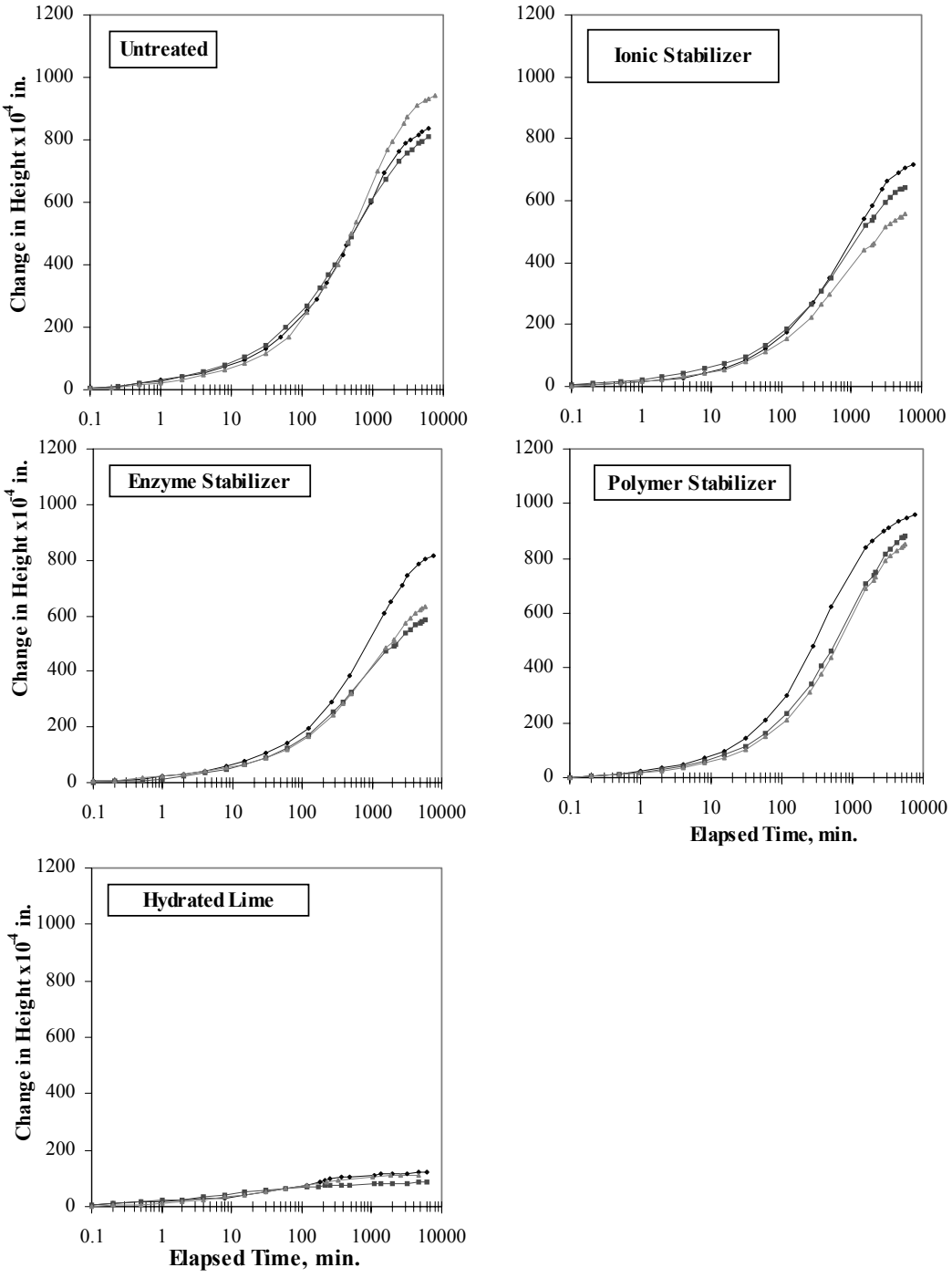

Figure O-8. Results from 1-D free swell tests on Fire clay in the follow-up study

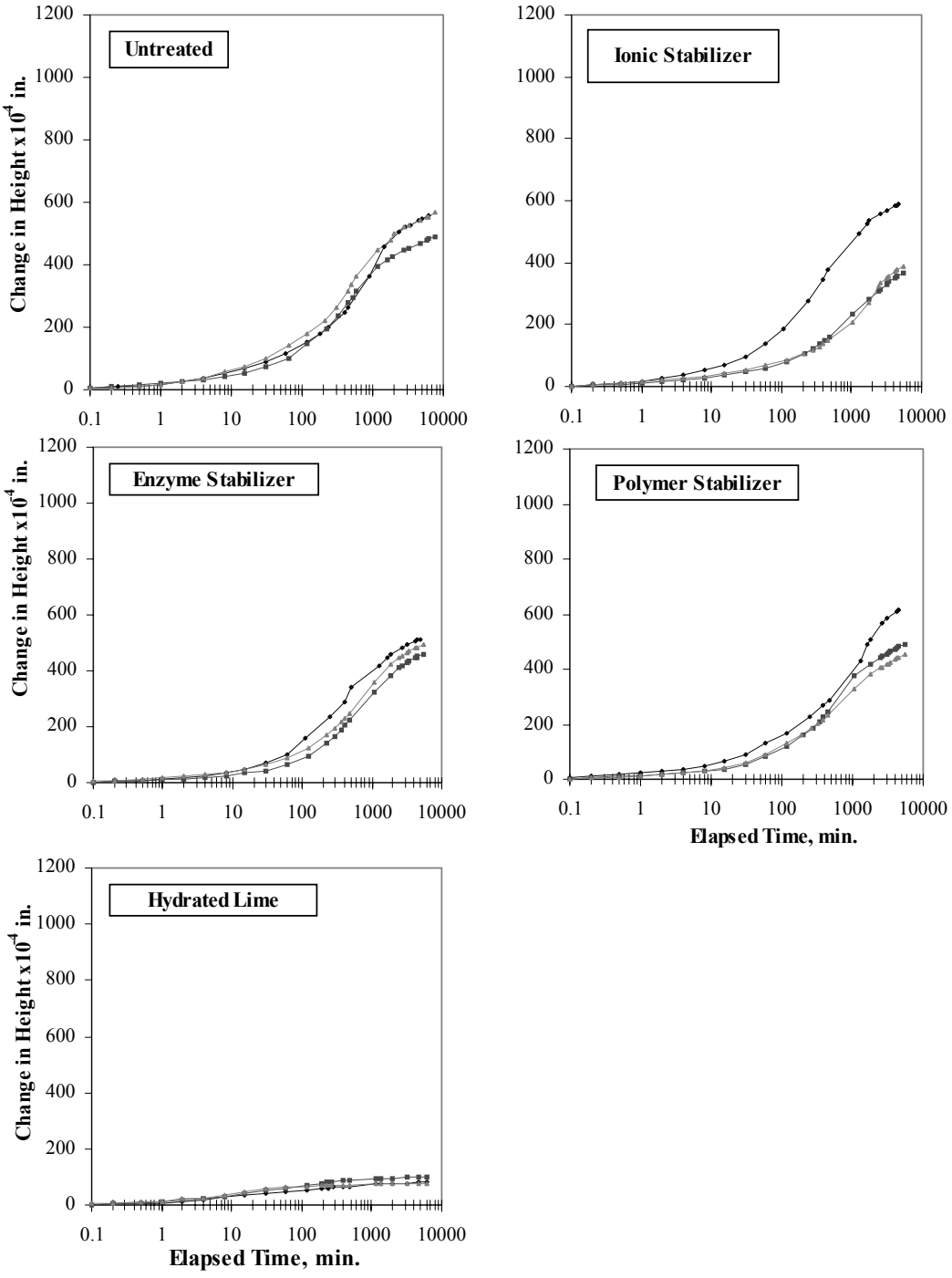

Figure O-9. Results from 1-D free swell tests on DG Taylor clay in the follow-up study

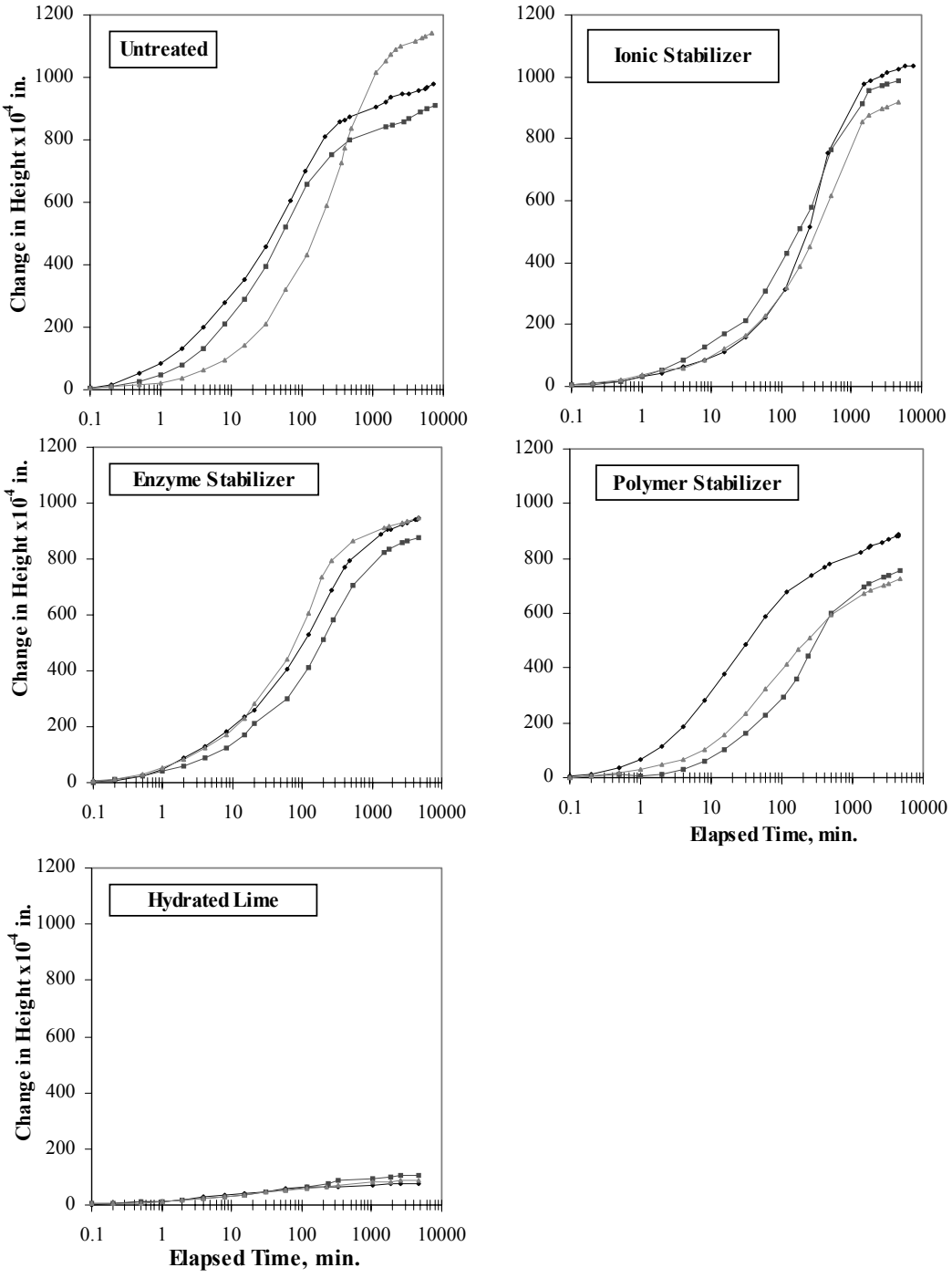

Figure O-10. Results from 1-D free swell tests on TG Taylor clay in the follow-up study

APPENDIX P

PROTOCOL FOR PREPARING LABORATORY TEST SPECIMENS OF SOILS TREATED WITH LIQUID CHEMICAL SOIL STABILIZERS:

Summary of Comments from TxDOT and Industry Representatives

To evaluate the efficacy of a candidate liquid soil stabilizer when treating a given soil, a standardized method is needed for preparing test specimens. Accordingly, the protocol outlined in Chapter 5 was written for preparing treated soil specimens for triaxial and swell testing in this study. In August 2000, this protocol was sent to a number of industry representatives and to the Texas Department of Transportation with a request for comments and criticisms. This appendix documents the comments received (summarized in italics) and provides responses where appropriate. On the basis of the issues raised in this process, a revised specimen preparation protocol (given in Appendix Q) was devised and is recommended for future studies.

DEFINITIONS

Four terms are used to describe the proportions of water and chemical stabilizer in a soil. These terms are defined here:

- IWC = Initial Water Content = mass ratio of water to oven-dry solids in the uncompacted soil prior to the addition of the diluted stabilizer chemical.

- OWC = Optimum Water Content = mass ratio of water to oven-dry soil that yields the maximum dry density of an untreated soil when compacted with a specified compaction effort.

- DMR = Dilution Mass Ratio = mass ratio of concentrated chemical product to water, used to express the dilution recommended for construction operations. This ratio applies only to the diluted product prior to mixing with the soil and has almost no relevance to the final concentration of product in the treated soil.

- AMR = Application Mass Ratio = mass ratio of concentrated chemical product to oven-dry soil in the treated soil.

INDUSTRY AND TxDOT REPRESENTATIVES PROVIDING COMMENTS

Comments on the sample preparation protocols were sought from a variety of people in TxDOT and the soil stabilization industry. The following people responded with comments, which are summarized below.

(1) Mr. Darren Hazlett
Mr. Harold Albers
TxDOT – Materials and Tests Division

(2) Mr. Joe Thompson
Mr. Paul Shover
unidentified person
TxDOT – Dallas District

(3) Dr. Marshall B. Addison
P.O. Box 173908
Arlington, TX 76003-3908

(4) Mr. Arthur D. Pengelly
Hayward Baker Inc.
2510 Decatur Avenue
Fort Worth, TX 76106

(5) Dr. Fred R. Huege
Director, Research and Development
Chemical Lime Co.
P.O. Box 985004
Fort Worth, TX 76185-5004

(6) Mr. Andres Jackson
Mr. Douglas R. Mandrell
Soils Control International, Inc.
1711 E. Central Texas Expressway
Suite 312
Killeen, TX 76541

(7) Mr. Richard I. Mueller
Dallas Roadway Products
14901 Quorum Drive, Suite 715
Dallas, TX 75240

(8) Mr. Russell J. Scharlin
Environmental Soil Stabilization, LLC
4025 Woodland Park Blvd., #165, LB32
Arlington, TX 76013-4377

(9) Mr. Linn Kempner
Soil Science International
1028 Fox Chase Road
Seber Springs, AR 72543

(10) Mr. Roy Alvarez
Roadbond International
1729 Evergreen Court
Harlingen, TX 78550

(11) Mr. Mike Horn
Pro Chemical Soil Stabilization of Texas
P.O. Box 185125
Fort Worth, TX 76181

(12) Ms. Maxine R. Williams, President
Base-Seal International, Inc.
15822 River Roads Drive
Houston, TX 77079

(13) Mr. Bret Braden
Products Division
The Charbon Group, LLC
14492 Morning Glory Road
Tustin, CA 92780

(14) Mr. Sachinder N. Gupta
E2 CR Inc
9004 Yellow Brick Road, Suite E
Baltimore, MD 21237

(15) Mr. Robert Randolph
Soil Stabilization Products Co.
P.O. Box 2779
Merced, CA 95344

PROTOCOL STEPS WITH COMMENTS

Use of distilled or de-ionized water.

Dissolved solids in the pore water may alter the soil chemistry and, in some circumstances, could affect the observed test results. Ideally, samples of the water to be used at the project construction site would be used to prepare the laboratory test specimens, but this is rarely practical. Ordinary tap water will typically contain a number of dissolved chemicals that

could interact with the stabilizer or soil, so the use of untreated tap water is undesirable. As is generally recommended for geotechnical testing practice, the use of distilled or de-ionized water is recommended here to prevent the introduction of unknown chemical species.

Summarized Comments:

- *The use of distilled or de-ionized water is good for the laboratory test experiments; however, the use of potable or on-site water may be used in the field.*
- *Distilled and/or de-ionized water is not a problem.*

Response to Comments:

The use of distilled or de-ionized water is an acceptable, practical choice that minimizes potential variability in the experimental data that could be related to water chemistry. Naturally, this should not be interpreted to mean that distilled or de-ionized water is required for field applications.

Step 1. Using a specified compaction test method, determine the optimum water content (OWC) for compaction of the untreated soil.

The optimum water content (OWC) for compaction shall be determined for the untreated soil using an appropriate, standardized compaction test. In this study, a modified Proctor compaction test method (ASTM D 1557, AASHTO T-180) will be used. Other standardized tests, such as the standard Proctor compaction test method (ASTM D 698, AASHTO T-99) or the TxDOT compaction test methods (Tex-113-E and Tex-114-E) may be more appropriate for a given project. The soil is to be prepared in accordance with the test specification, which may include screening out oversized particles.

Note that the OWC determined for the untreated soil will be used as the target water content for compaction of the treated soil specimens. A separate compaction curve is not determined for the soil when treated with a given stabilizer. Hence, all test specimens, including both untreated control samples and treated samples, should have about the same water content and degree of compaction. Preparing all test specimens in this manner ensures that any observed changes in soil properties can be attributed to the action of the chemical stabilizer and not to substantial differences in the density or fabric of the soil, which will result from different compaction conditions.

Summarized Comments:

- *The optimum conditions of the treated soil should be re-evaluated. The addition of lime may change the optimum conditions as determined using the untreated soil.*
- *ASTM D 698 should be used in lieu of D 1557, AASHTO T180 or Tex-114-E. The higher compactive effort will make it difficult to trim the samples. Samples should be compacted at OWC +5%.*
- *One problem with using the OWC for the untreated soil for compaction of the treated soil is that this approach assumes that the water will be used only to evenly distribute the chemical*

stabilizer throughout the soil. Thus, none of it will be used up by a possible chemical reaction with the chemical stabilizer added to the soil. If this assumption is incorrect, then the addition of the necessary water to reach OWC of the treated soil will yield a different moisture content; a different density will result from a difference in moisture content.

- *Screening out the larger particles is not representative of real soil. Using a 4-inch mold does not account for the large rock (aggregate).*
- *Need to determine optimum moisture content for compaction with each stabilizer product. Each will affect OWC differently.*
- *For "EMC SQUARED", higher compaction efforts are recommended. In Texas, at least 95% of the maximum density from Tex-113-E is recommended, but the higher energy obtained from ASTM D 1557 is preferred.*

Response to Comments:

Several respondents have pointed out the need to determine the OWC for the soil after treating with the stabilizer. In particular, the observation that some water could be used in a chemical reaction with the stabilizer is appropriate. Moreover, conventional practice with lime treated soils (such as that embodied in Tex-121-E) calls for determining the OWC for the treated soil. Hence, this part of the revised protocol has been modified so that the OWC will be determined for the soil treated at the recommended AMR. Some additional testing is warranted to investigate how much these stabilizers may affect the optimum moisture content for compaction.

For evaluating lime treated soils, TxDOT method Tex-121-E specifies the use of the Tex-113-E compaction method. This laboratory compaction test is also acceptable for use with liquid chemical stabilizers. Hence, to maintain consistency, a note has been added to recommend the use of the Tex-113-E method for TxDOT projects. Other compaction test methods should be equally acceptable.

Other issues, such as a preference for using higher or lower compaction energies, increasing the water content at which the specimens are compacted, and screening out large particles, should be addressed elsewhere. These issues also impact the performance of the untreated soil, and they should be treated separately in a comparative study to measure the relative effectiveness of a chemical additive.

Step 2. Determine the recommended application mass ratio (AMR) for the stabilizer product.

The rate of field application recommended by many stabilizer suppliers can be somewhat difficult to translate into an equivalent application rate for preparing laboratory samples. For example, assume one gallon of a product is diluted 1:500 by volume in water in the field. This diluted product is then sprayed over an area of 5,000 square feet, mechanically mixed with the base material, and compacted to a final thickness of six inches. Hence, such a product is applied at a rate of one gallon of concentrated product per 2,500 cubic feet of moist, compacted soil. Knowing the density of the concentrated chemical product and the dry density of the compacted soil, it is possible to convert the supplier's recommended application rate to the application mass ratio (AMR).

There are several advantages to expressing the application rate in terms of the AMR. First, using the AMR simplifies the conversion of recommended field application rates to equivalent values for preparing laboratory test specimens. More importantly, using the AMR clarifies that the critical aspect of determining an appropriate application rate is to consider the ratio of stabilizer chemical to dry soil solids. Although the dilution mass ratio (DMR) is relevant for determining how much water to mix with a product prior to use on a construction site, it is the AMR that expresses how much stabilizer is present in the treated soil. Therefore, AMR is of greater relevance. It is worth noting that conventional lime or cement soil stabilization is also usually specified in terms of lime or cement contents that are computed on the basis of dry soil weights. Finally, using AMR in combination with OWC, it is clear how to handle the soil water when computing application rates.

To convert recommended field application rates (e.g., 1 gallon of product diluted in 500 gallons of water, sprayed on 5,000 square feet of soil, and then mixed 6 inches deep), the dry density of the soil is needed. In making these conversions, a representative dry unit weight of about 100 lbs/ft^3 will be assumed for this study. This represents a typical dry density of a well-compacted clayey soil.

Summarized Comments:

- *Develop an equation to determine the actual application rate.*
- *The OWC determined as part of the compaction test on each soil should be used to provide a more accurate calculation of the unit dry weight for a specific soil.*

Response to Comments:

In determining the AMR, the difficulty lies in the different ways different suppliers express their recommended "application rate." Hence, it is not possible to develop a single equation for making this conversion. Rather, with AMR clearly defined, the project engineer can make appropriate assumptions and conversions to get an AMR from the supplier-provided recommendations. Much potential confusion can be eliminated if the product supplier reports the recommended application rate in terms of the AMR.

The recommended AMR should not change with variations in dry density. Hence, the second comment above is not relevant. The assumed dry unit weight of 100 pcf is used to compute the AMR only in that situation in which the supplier's recommendations are not specific enough to determine the equivalent AMR.

Step 3. Dilute the concentrated stabilizer product to the recommended dilution mass ratio (DMR).

Nontraditional chemical soil stabilizers are typically sold as concentrated liquids that are diluted in water on the project site before application. In this step, a sufficient quantity of stabilizer is prepared by diluting the concentrated product in distilled or de-ionized water.

The dilution ratio is usually specified by the supplier on a volumetric basis. For example, the product might be diluted to a ratio of one gallon of concentrated chemical per 500 gallons of

water. Knowing the specific gravity or mass density of the chemical, this ratio can be converted to the mass-based DMR, which is more convenient to use in subsequent calculations.

Note that the DMR is *not* the ratio of the chemical product to water in the compacted soil, because the diluted product is added to soil that is already wet with water.

Summarized Comments:

- *From examination and research of other products on the market, many manufacturers advertise dilution rates of 200/300/1,000 to 1. Without the presence of solids going into the soil, there is nothing there but water. Many people get fooled because you can get compaction with water alone. The solids content (binder) is very important.*

Response to Comments:

Agreed. The separate definitions of DMR and AMR help to distinguish this issue. The dilution ratio is meaningless unless one also knows how much of the diluted solution is applied to the soil. When it comes to understanding how much product is mixed with the soil, the AMR is the key parameter.

Step 4. Pre-moisten the test soil to an initial water content of IWC = OWC – (AMR/DMR).

Begin with a sufficient quantity of soil for the planned testing program. Screen out oversized particles in accordance with the chosen compaction procedure followed in Step 1. Next, adjust the water content to a point dry of the OWC determined in Step 1. This may involve either air drying the soil over a period of time or spraying distilled or de-ionized water onto the soil as it is thoroughly mixed.

The objective at this step is to mix the soil to an initial water content (IWC) just below the OWC, so that the OWC is attained when the diluted stabilizer is added in Step 7. Recall that the stabilizer chemical, diluted to the DMR, is added to the soil in sufficient quantities to achieve the desired AMR. Adding stabilizer diluted in water will therefore change the water content of the treated soil by this amount:

$$\text{change in water content} = \frac{\text{mass of water added with stabilizer}}{\text{mass of dry soil}}$$

$$\Delta w = \frac{(M_w)_c}{M_s} = \left(\frac{M_c}{DMR}\right)\left(\frac{1}{M_s}\right) = \frac{AMR}{DMR}$$

Hence, if the IWC is set at:

$$IWC = OWC - \frac{AMR}{DMR}$$

then the water content should be equal to the OWC when the diluted chemical is added in Step 7. Note that this calculation assumes no water loss owing to evaporation during sample preparation. Depending on laboratory procedures, the IWC may need to be adjusted as discussed under Step 10.

For a typical stabilizer product, the value of (AMR/DMR) is on the order of 3%. Hence, the soil would be pre-moistened to a water content 3% below the OWC at this step.

Summarized Comments:

- *It is suggested that the moisture content should be OWC +5%. Experience indicates the chemical stabilizer will not work properly or will work intermittently without an elevated moisture content. This step is most important. If the moisture content is beyond this percentage, the sample must be elevated to allow the free water to discharge from the sample.*

Response to Comments:

Compacting untreated samples 5% wet of optimum will yield a soil with a lower dry density and lower undrained strength and stiffness. Hence, this recommendation is questionable. Also, there is no way to "elevate" the soil to ensure discharge of the excess water in the field. Perhaps some of this water is used in the stabilization reaction, and the actual OWC of the treated soil is higher than the OWC of the untreated soil. If so, this effect will be accounted for by determining the OWC for the treated soil, as discussed earlier and recommended in the revised protocol.

In ASTM D 4609 (paragraph A2.1), it is suggested that 0.5 to 3.0% in the soil water content is typically lost in mixing soil samples for compaction. These are reasonable numbers that can be used as the basis for estimating the additional water that should be added to compensate for expected evaporation during preparation. Accordingly, this step has been modified to suggest mixing the soil to a water content 2% higher.

Step 5. Allow the pre-moistened soil to mellow for 16 hours in a sealed container.

The pre-moistened soil will then be sealed in a container and allowed to sit at least 16 hours (overnight) at room temperature. This mellowing period is needed to ensure that the pore water becomes completely and uniformly dispersed into the soil.

Summarized Comments:

- *Extend the mellowing period for evaluation up to seven days. The additional mellowing time may yield different results.*
- *Extend the mellowing period if the soil is sulfate rich. Procedures such as using a three-day mellowing period at 5% above optimum moisture content has been used for sulfate soils mixed with lime. This time allows the sulfates to be soluble and for ettringite to form.*
- *Change the pre-moistened soil to mellow for 16 to 24 hours depending on the type of soil under evaluation. The time of 16 hours would not be sufficient for cohesive soils (subgrades), but for non-cohesive soils (caliche, limestone, or RAP) a mellowing period of 16 hours or less would work.*
- *Initial moisture content should be about 5% above OWC.*
- *The field conditions should be used to determine mellowing period.*

Response to Comments:

Some of these comments are apparently confusing the mellowing period (hours) of the mixed material prior to compaction with the curing period (days) of the compacted specimens prior to testing. The appropriate curing period is discussed under Step 9 below.

Although it would be good to match the field mellowing period, actual times during construction will vary significantly even on the same project, making that approach impractical. The 16-hour mellowing period is based on the requirements set forth in ASTM D 698 and ASTM D 1557 for preparing fine-grained soils for laboratory compaction (shorter mellowing periods are permitted for silty or clean sands and gravels). For lime treated soil samples, Tex-121-E requires only a 12-hour mellowing time prior to compaction. Given that there is less experience with liquid chemical stabilizers, the slightly longer mellowing period of 16 hours is warranted. However, based on the experience embodied in ASTM D 698, a longer mellowing period of 24 hours is unnecessary.

The comment regarding appropriate mellowing periods for sulfate-rich soils is intriguing. However, more research with sulfate-rich soils is needed to study the effects of a longer mellowing period.

Step 6. Measure out the mass of diluted stabilizer needed to achieve the recommended application mass ratio (AMR) and optimum water content (OWC) in the treated sample.

On the basis of the mass of dry solids (M_s) in the sample, determine the mass of concentrated chemical (M_c) that must be added to achieve the desired AMR. Measure out a sufficient quantity of the diluted stabilizer to obtain the required mass of chemical concentrate.

If the stabilizer is diluted properly to the DMR in Step 3 and the soil is moistened to the correct IWC in Step 4, then the water content of the treated soil will be equal to the OWC (less any losses due to evaporation).

No comments were received.

Step 7. Thoroughly mix the diluted stabilizer with the soil sample, and then allow to stand for 1 hour in a sealed container.

The soil will be thoroughly and completely mixed using a mechanical mixer. Care will be taken to limit evaporation losses and to maintain the desired values of AMR and OWC in the mixed soil.

Immediately following mixing, the sample will be sealed in a container and allowed to stand for one hour. This standing time is intended to allow the stabilizer chemicals to achieve a more homogeneous diffusion into the soil. Longer standing times will be avoided to prevent excessive stabilizer curing prior to compaction. The one-hour delay is also meant to reflect a typical time delay between the initial application and mixing of a product and the final compaction of a roadbed in the field. The sample must be sealed during the standing time to prevent excessive loss of moisture.

The one-hour standing time is also required prior to compaction of untreated control samples.

Summarized Comments:

- *One hour is insufficient, particularly in heavy clay. The term "complete mixing" should be defined.*
- *Based on independent studies, a standing period longer than one hour is necessary to see the swell reduction in the lab that is observed in the field. Standing periods, after mixing, of one and two weeks have been used for compaction. After a one-week standing period, a slight swell reduction was measured between treated and untreated soil. After a two-week period, a 40% swell reduction was measured, a value similar to that which has been measured in the field on TxDOT projects for chemically injected sites.*
- *Thoroughly mix the diluted stabilizer with the soil samples and then allow one sample to stand for one hour in a sealed container. Allow one sample to stand at room temperature, after mixed in a mixing bowl. One hour is normally sufficient, but one to three hours may be needed based on material being mixed (i.e., less time for base than subgrade).*

Response to Comments:

In ASTM D 4609 (paragraph 7.2), sufficient mixing of chemically stabilized soil samples is described as "Blend thoroughly (normally for about 5 min) to produce a high degree of homogeneity." Similar wording has been added to this step of the revised protocol.

It should be noted that for lime treated soil samples, Tex-121-E does not require any minimum standing time prior to compaction. Here, one hour is allowed to permit more complete mixing of the liquid chemicals. Much longer times are avoided because some products may work by bonding soil particles together. If significant bonding occurs during a long standing time prior to compaction, the bonds may be broken during compaction, and the improvements to the soil may be lost.

The suggestion of allowing one to two weeks prior to compaction is clearly not representative of typical field conditions. Moreover, the reported reduction in swell potential might have resulted from pre-swelling of the soil under wet conditions prior to compaction. In any case, more research would be needed to justify a standing time in excess of a few hours.

Step 8. Compact the soil with the specified compaction method, extrude from the mold, and seal in a container.

Immediately following the one-hour standing time, the soil will be compacted following the same standard procedures used in Step 1. The specimens will then be extruded, sealed in containers, and cured according to the procedures in Step 9.

Summarized Comments:

- *Store the sealed containers in a moist curing room.*
- *Compact the soil with the specified compaction method, extrude from the mold, then place one sample in a sealed container and one sample out to air dry at room temperature.*

Response to Comments:

As noted and discussed under Step 9 below, the samples should be cured at constant water content. In addition to using "sealed containers," placing the specimens in a moist curing room will ensure that the samples do not dry out if the container seal is less than perfect. Hence, storing the compacted samples in a moist curing room has been added to the revised protocol.

Step 9. Cure the compacted soil in sealed container at room temperature for 7 days.

Compacted samples, including both treated and untreated specimens, will be cured at constant water content by placing them in sealed nonreactive containers (such as sealed plastic bags). Curing at constant water content has been selected to make it possible to discern the effects of a given product on the measured properties of the soil. A constant overall water content eliminates the effect of changing water content on the observed soil strength and stiffness. That is, simply wetting or drying an untreated, unsaturated soil will lead to changes in the measured shear strength in an undrained triaxial test. To observe how much the strength may change owing to the presence of the stabilizer, one needs to eliminate variations in water content as a possible cause of these measured changes. We also feel that this procedure will effectively represent the curing conditions in a chemically stabilized, compacted roadway subgrade. Although the very top of a compacted base material may have free access to air during the curing period, soil just below the surface does not have open ventilation and will remain moist.

Secondly, the soil will be cured at room temperature, which is a reasonable and convenient compromise between the extremes of hot or cold temperatures that could be encountered in the field.

Finally, a curing time of seven days is specified to allow sufficient time for the stabilizer product to completely react with the soil. During the curing period, the compacted samples will

be out of the molds, sealed, and kept at room temperature, as described above. The seven-day cure period is based on the recommendations of the various stabilizer suppliers, is consistent with typical curing periods used in the evaluation of lime and cement soil stabilization, and is convenient for sequencing a laboratory test program.

Summarized Comments:

- *A 28-day cure is standard for lime and cement. Curing the samples at room temperature is a good idea because an accelerated cure can produce inaccurate results in some soils. Compacting the samples at the same moisture content is a good idea as long as that will be the moisture content at which the samples are compacted in the field.*
- *Most dry stabilizers gain all their strength in the first seven days. Top-Seal will continue to gain strength up to 28 days. This is a function of the slower curing process. Thus, it is suggested that the sample not be placed in a sealed container for seven days. Top-Seal needs the air to cure. When testing it for permeability, some labs have even placed the samples in an oven.*
- *Cure the compacted soil, one in a sealed container and one unsealed container, for 3, 7, and 28 days in order to simulate similar field conditions.*
- *Curing time should be two to three days only. If the chemical hasn't worked within 72 hours, it will not work at all. Also, a longer than necessary curing time that is established at this point will create undue delays in field applications/testing.*
- *Could the test be extended to a 14-day break and a 28-day break? We noted on the University of Arkansas test of our "BASE-SEAL", considerable strength was realized in added days.*
- *Seven days seems a little excessive. Most chemical stabilizers that I have read about say that changes to a treated soil occur rapidly after initial introduction of the chemical stabilizer. As a result, if a manufacturer states that only three days is needed to fully cure the soil, then the time for curing should be set at three days instead of seven. It might be better to state that the maximum allowed curing time of the treated and compacted specimens should be seven days. The curing time can be less if recommended by the manufacturer.*
- *Curing the compacted soil for seven days is long for real-life situations. The roadway is generally opened within a few hours after compacting, especially for secondary roads.*
- *A "dry back" period, with air dry curing, is necessary for "EMC SQUARED" to be effective. Keeping the soil moist will stop the curing process. The lab procedures should reflect field conditions with a dry back period as part of curing. We recommend a 48- to 72-hour period of air drying. After the dry back period, the samples may be placed in bags if cracking is visible on the surfaces of the curing specimens.*

Response to Comments:

Of all the comments received, the issue of what is an appropriate curing procedure received the most attention. The respondents recommended curing periods from 2 to 28 days. The apparent motivation for recommending longer curing periods is to obtain the maximum possible change in soil properties. On the other hand, shorter curing periods would permit a faster evaluation.

Data recently published by Santoni et al. (2002) show that for two polymer stabilizers, about half of the 28-day increase in unconfined compressive strength was achieved in about seven days. In those tests on silty sands treated at very high application rates, changes in the soil strength were clearly detectable after seven days. However, part of the observed strength gain was due to drying of the soil specimens during the curing period.

Overall, it seems that a seven-day cure is a reasonable compromise. Clearly, one might see continued changes in soil properties beyond the seven-day cure. However, if no significant change in soil properties is observed in the first week, it is unlikely that significant changes would occur in the ensuing weeks. Shorter curing periods might be considered, but only after much more experience is gained with these products. Note that for lime treated soils, the Tex-121-E procedure involves a seven-day cure at room temperature.

Some suppliers recommend a period of air drying during the curing cycle. Our objection to this suggestion, as outlined above, centers around the fact that changing the water content will by itself change the observed soil properties. Drying out a compacted soil specimen increases the matric suction pressures in the pore water that, in turn, increases the strength and stiffness of the soil. Hence, it would not be possible to distinguish the positive effects of the stabilizer chemical from the effects of drying the soil. Likewise, an accelerated cure in an oven is undesirable for similar reasons.

Step 10. Trim the sample to an appropriate size for testing and determine the specimen water content. If the water content is not within acceptable limits for compaction, prepare new specimens using an adjusted initial water content (IWC).

At the end of the seven-day curing period, the soil samples will be removed from the sealed containers and trimmed to an appropriate size for testing.

The water content of the trimmed specimen should now be checked to determine whether significant water has been lost to evaporation during sample preparation. This can be evaluated easily by measuring the water content of the specimen trimmings. The amount of evaporation loss could vary considerably, depending on a number of factors such as the relative humidity and temperature of the laboratory where the soil is mixed.

If the water content of the trimmed specimen is too low, then new specimens should be prepared using a higher IWC in Step 4. For example, suppose that the compaction specification calls for compacting soil in the field at a water content within ±2% of optimum. If the specimen water content measured in Step 10 was found to be 3% below the OWC (outside the acceptable range), then new specimens should be prepared starting with IWC = OWC – (AMR/DMR) + 3%.

Summarized Comments:

- *Trimming should be performed immediately after compaction, at the time that the samples are extruded from their molds. Some stabilizers, notably lime and cement, cannot be trimmed after being cured, and the test procedure should be consistent for all agents.*
- *Starting the process all over again by preparing new samples with significantly lower moisture content would be very time consuming. Instead of having results after about eight or nine days of testing, it will take at least 16 days to get test results. It is also assumed that any significant loss of water would be due to evaporation. The water might be lost for other*

reasons. If so, starting again with an adjusted water content may not correct for the significant water loss of the compacted specimens during the seven days of curing.

Response to Comments:

Trimming after the cure period is generally more convenient, because the lab technician can trim away areas damaged in handling. Specimens trimmed before the curing period are unlikely to have the perfect shape (straight cylinder, flat and square ends) needed later to perform a good quality triaxial or swell test. If the specimen cannot be trimmed after the cure, the specimens could be trimmed prior to curing with little anticipated impact on the test results.

As discussed under Step 4 above, the revised protocol has been changed to suggest mixing the soil to an initial water content 2% above optimum to account for typical evaporation. However, this will not eliminate the need to confirm that the final water content of the specimen is acceptable at this stage of the procedure.

Overall Comments on the Proposed Protocol.

Comment:

- *The samples should be prepared at the same unit dry weight because it has a large effect on both the swell and strength results. Also, the strength tests should be performed at saturated undrained conditions, which represent the likely scenario of pavements. Because the routine strength test performed on soil samples is some test other than triaxial (e.g., unconfined compression), perhaps the testing procedure should rely on another means of measuring strength. For comparison, the study should evaluate samples treated with traditional methods, such as lime and water. The water control would allow for comparison with simple mechanical compaction, and lime treated soils would provide a comparison to the state-of-the-practice.*

Response:

Compacting the soil at OWC with the same compaction energy will produce specimens with the same dry unit weight, with some variation due to experimental error. The other comments are related to testing of the prepared specimens and are not relevant to the preparation protocol under discussion.

Comment:

- *Three things must take place when installing a liquid stabilizer:*
    a. *the presence of solids (binder in the product),*
    b. *adequate and even distribution into the soil, and*
    c. *good compaction of the soil.*
- *The testing procedure protocol is right in line with ASTM D 4609 for liquid chemical stabilizers and all other related ASTM specifications.*

Response:

Agreed. These issues have been addressed in the protocol.

Comment:

- *In the definition of AMR, the phrase oven-dry soil should be replaced with dry unit weight.*

Response:

Wrong. The AMR is a ratio of weights, not unit weights (density).

Comment:

- *It is difficult to obtain consistent samples (density) using standard dynamic compaction equipment. More uniform results may be obtained by utilizing a hydraulic press to compact the samples for trimming. A smaller sample would also result in a better moisture distribution within the sample.*

Response:

A hydraulic press may produce a more uniform soil density, but it also tends to form layers in the specimen and does not induce significant kneading action. The widely used impact compaction test methods are thus preferred.

Comment:

- *The basic procedure is very sound, with a few changes are proposed with regard to the use of "Perma-Zyme 11X" enzyme soil stabilizer:*
  (a) Since the enzymes in "Perma-Zyme 11X" act to both assist water penetration into the soil mass and to chelate dissolved minerals in the soil matrix, it is very important to initially add "Perma-Zyme" to the soil along with the required water. Therefore, at least for "Perma-Zyme 11X", we recommend using the calculated amount of "Perma-Zyme" prediluted with the necessary water to initially bring the soil sample up to optimum moisture. Therefore, the mixing process should take place in Step 4 instead of Step 7, with a soaking period of at least 16 hours afterward (combine Step 6 into Step 4).
  (b) Although initial curing takes place within a four- to seven-day period, full curing with "Perma-Zyme 11X" occurs, similar to Portland cement concrete over a 28-day period. The enzymes in "Perma-Zyme 11X" act to chelate the dissolved soil minerals into a weak mortar cement bond. Therefore, prior to any strength tests (i.e., CBR), the core should be fully cured.
  (c) We note that there is no mention of soil characteristics. For the use of "Perma-Zyme 11X", we recommend soils with a slight plasticity ($0 < PI < 4$). Also, we have learned through working with companies such as ____ that the total organics in the soil need to be less than 10% by mass.

Response:

The protocol is general and can be applied to any chemical stabilizer. To permit proper comparisons of performance, special provisions should not be introduced to favor any particular type of product. If the mechanism described in (a) is significant, then this product will alter the OWC for compaction. Consideration for this possible effect has been incorporated into the revised protocol where the OWC is determined for the treated soil. The longer curing period suggested in (b) has been addressed under Step 9 above. Finally, characteristics of soils that can be effectively treated with any given chemical, as pointed out in (c), are not addressed in this protocol. Rather, this protocol is designed for use in laboratory investigations attempting to answer whether a particular soil can be treated effectively.

Comment:

- *The total time required by the procedure is lengthy. The project timeline may not allow for this amount of time to be spent to start the roadbed preparation.*

Response:

Yes, the procedure is lengthy in that one to two weeks of work are required to prepare test specimens. However, without more experience with these materials, it is difficult to simplify or accelerate the process. At present, careful work in the laboratory is essential to quantify the potential benefits of these unconventional products.

Addendum: Wetting and drying cycles.

For evaluating the effectiveness of treating soils with lime, the Tex-121-E procedure used by TxDOT involves preparing test specimens in this manner:

- Cure compacted specimens at room temperature in a sealed container for seven days.
- Dry specimen in an oven to remove one third to one half of the water.
- Subject the specimen to capillarity for ten days while under a confining pressure of 1 psi.
- Test in unconfined compression.

This procedure thus tests the durability of the treatment through one drying and wetting cycle, but adds ten days to the testing cycle.

A similar procedure might be warranted for evaluating the durability of liquid chemical soil stabilizers. However, much more experience with these products is needed before a simplified procedure, such as that used for lime treated soils, can be justified. For now, it is recommended that no attempt to simulate wetting and drying, or other durability effects, should be included in this protocol for soils treated with liquid chemical stabilizers.

APPENDIX Q

RECOMMENDED PROTOCOL FOR PREPARING LABORATORY TEST SPECIMENS OF SOILS TREATED WITH LIQUID CHEMICAL SOIL STABILIZERS

To evaluate the efficacy of a candidate liquid soil stabilizer when treating a given soil, a standardized method is needed for preparing test specimens. Here, a protocol appropriate for preparing treated soil specimens for triaxial and swell testing is outlined. This is a revised version of a protocol used in this study, which is described in Chapter 5, with revisions made in response to TxDOT and industry comments as summarized in Appendix O.

The application under consideration for these chemicals is to stabilize roadway subgrade or base courses, where mechanical mixing of the product with the soils is possible during construction. Soil stabilization is sometimes accomplished in situ using pressure injection without the benefit of mechanical mixing. For evaluating changes in soil properties resulting from pressure injection of chemical stabilizers, modifications to this protocol may be appropriate.

DEFINITIONS

Four terms are used to describe the proportions of water and chemical stabilizer in a soil. These terms are defined here and are shown in Figure Q-1 on the next page.

- IWC = Initial Water Content = mass ratio of water to oven-dry solids in the uncompacted soil prior to the addition of the diluted stabilizer chemical.

- OWC = Optimum Water Content = mass ratio of water to oven-dry soil that yields the maximum dry density, when compacted with a specified compaction effort, of a soil that has been treated with a given stabilizer at the recommended application rate.

- DMR = Dilution Mass Ratio = mass ratio of concentrated chemical product to water, used to express the dilution recommended for construction operations. This ratio applies only to the diluted product prior to mixing with the soil and does not reflect the final concentration of product in the treated soil.

- AMR = Application Mass Ratio = mass ratio of concentrated chemical product to oven-dry soil in the treated soil.

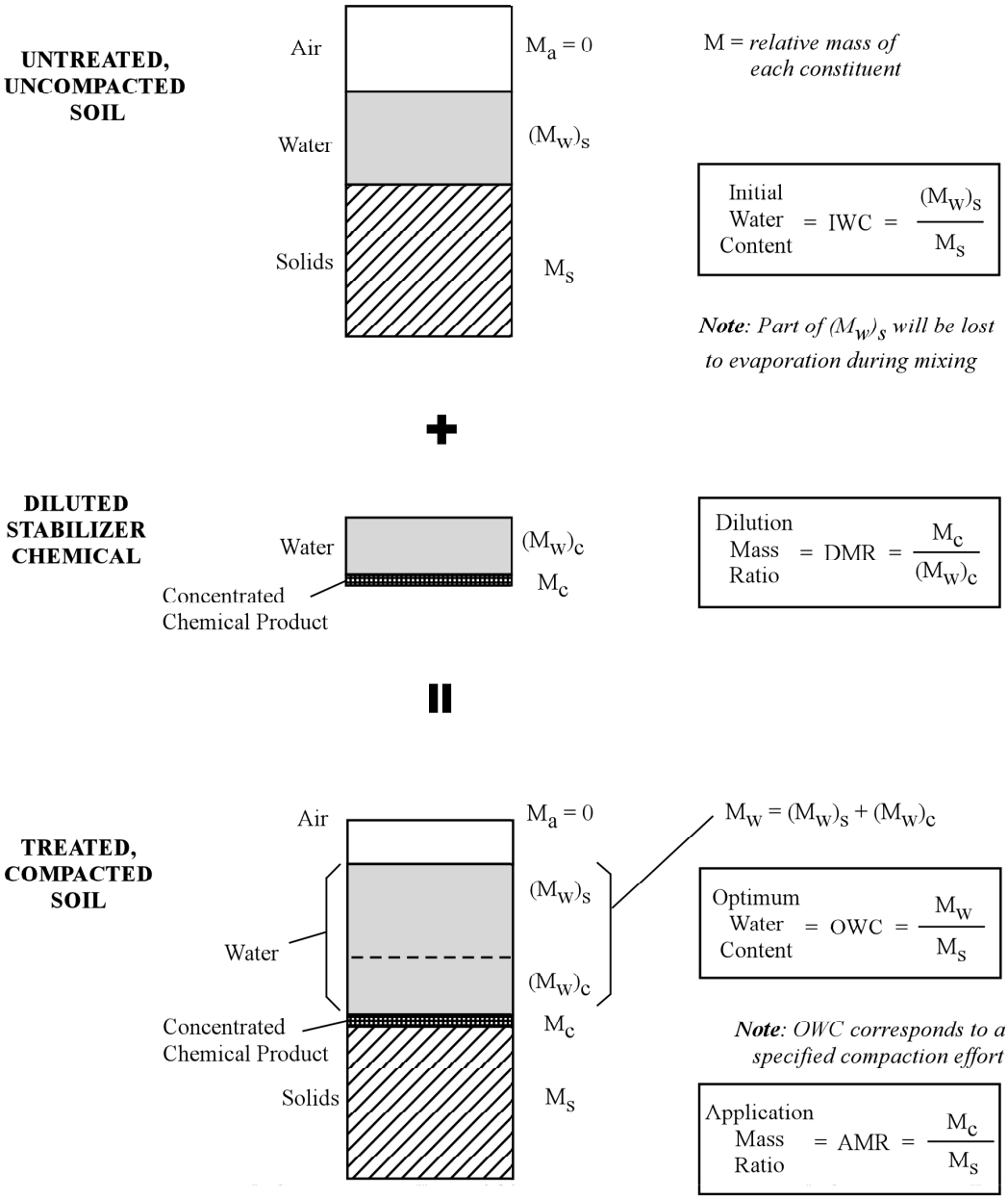

Figure Q-1. Phase diagrams for the untreated soil, diluted stabilizer, and chemically treated soil after mixing to the optimum water content

SAMPLE PREPARATION PROTOCOL

The following steps are required to prepare laboratory test specimens of untreated control soil specimens and specimens of soil treated with chemical stabilizers. In all cases, only distilled or de-ionized water shall be used to dilute the stabilizer products or to increase the water content of the test soil.

Step 1. Determine the recommended application mass ratio (AMR) for the stabilizer product.
- The AMR should be provided by the product supplier.

Step 2. Dilute the concentrated stabilizer product to the recommended dilution mass ratio (DMR).
- The DMR should be provided by the product supplier.

Step 3. Using a specified compaction test method, determine the optimum water content (OWC) for compaction of the soil when treated at the recommended AMR.
- For TxDOT projects, the Tex-113-E compaction test method should be used.
- In accordance with Step 5 below, allow the pre-moistened soil samples to mellow for at least 16 hours before adding the diluted stabilizer.
- In accordance with Step 8 below, allow the treated soil (after mixing at the recommended AMR) to stand for 1 hour before compaction.

Step 4. Pre-moisten or air-dry the test soil to an initial water content of

$$IWC = OWC + 2\% - (AMR/DMR)$$

Step 5. Allow the pre-moistened soil to mellow for at least 16 hours in a sealed container.

Step 6. Measure out the mass of diluted stabilizer needed to achieve the recommended application mass ratio (AMR) in the treated sample.

Step 7. Blend and mix the diluted stabilizer with the soil sample until a high degree of homogeneity is achieved.

Step 8. Allow the mixture to stand for 1 hour in a sealed container.

Step 9. Compact the soil with the specified compaction method used in Step 3.

Step 10. Extrude the compacted soil from the mold, seal in a container, and place in a moist curing room.

Step 11. Cure the compacted soil in sealed container at room temperature for 7 days in a moist curing room.

Step 12. Trim the sample to an appropriate size for testing and determine the specimen water content using the sample trimmings.

Step 13. If the specimen water content is not within acceptable limits for compaction, prepare new specimens using an adjusted initial water content (IWC).

EXAMPLE

Assume that product *X* is to be evaluated for modifying soil *Y*. The following 13 steps are followed to prepare treated test specimens.

Step 1. The supplier of product *X* recommends that one gallon of concentrated chemical can be used to treat 600 cubic feet of compacted soil *Y*. To determine the

equivalent AMR, assume the soil has a compacted dry unit weight of 100 lbs/ft^3. We also need the mass density of the concentrated product *X*, which is given as 1.45 g/ml. The AMR is then 1/5,000, calculated as follows:

Step 2. The supplier of product *X* recommends that the concentrated chemical

$$\left(\frac{1\,\text{lb}}{4.4482\,\text{kg - m/sec}^2}\right)\left(\frac{9.80665\,\text{m}}{\text{sec}^2}\right)\left(\frac{\text{kg}}{1{,}000\,\text{g}}\right)=\frac{1}{4{,}958}=\frac{1}{5{,}000}$$

$$\text{AMR}=\left(\frac{1\,\text{gal}\,X}{600\,\text{ft}^3\,Y}\right)\left(\frac{3785.4\,\text{ml}}{\text{gal}}\right)\left(\frac{1.45\,\text{g}}{\text{ml}\,X}\right)\left(\frac{1\,\text{ft}^3\,Y}{100\,\text{lbs solids}}\right)\times$$

Step 3. should be diluted with water at a volumetric ratio of 1/200. A sample of product *X* is then mixed with distilled water at this ratio.

Based on the mass density of 1.45 g/ml for concentrated product *X*, the equivalent DMR is then:

$$\text{DMR}=\left(\frac{1\,\text{ml}\,X}{200\,\text{ml water}}\right)\left(\frac{1.45\,\text{g}}{\text{ml}\,X}\right)\left(\frac{\text{ml water}}{1.00\,\text{g}}\right)=\frac{1}{137.9}$$

Step 4. Using the TxDOT Tex-113-E compaction test method, the OWC of soil *Y* is determined to be 24%, when soil *Y* is treated with product *X* to an AMR of 1/5,000.

Step 5. The target initial water content (IWC) is computed based on the OWC, AMR, and DMR determined in Steps 1 to 3. Here, 2% additional water is added to compensate for typical evaporation during mixing. Also, an allowance is made for the water that will be added with the diluted product in Step 7, which will increase the soil water content by a magnitude of (AMR/DMR). That is,

$$\text{IWC} = \text{OWC} + 2\% - (\text{AMR}/\text{DMR}) = 0.24 + 0.02 - (137.9/5000) = 0.232$$

A "dry" sample of soil *Y* has a total mass of 10.304 kg (10,304 g). The actual water content is measured to be 4.9%. The mass of dry soil solids and water in this sample is then

$$M_s = M_{total} / (1 + w) = 10{,}304 / (1 + 0.049) = 9{,}823 \text{ g}$$
$$(M_w)_{si} = M_{total} - M_s = 10{,}304 - 9{,}823 = 481 \text{ g}$$

The mass of water needed in the sample to achieve the IWC = 23.2% is

$$(M_w)_s = M_s \times IWC = 9{,}823 \times 0.232 = 2{,}279 \text{ g}$$

The difference in the two water masses is the amount of water that must be added to achieve the IWC. In this case, it is

$$\text{Mass of water to be added} = (M_w)_s - (M_w)_{si} = 2{,}279 - 481 = 1{,}798 \text{ g}$$

Remember that the density of distilled water is 1 g/ml. Hence, mix the soil thoroughly with 1,798 ml of distilled water.

Step 6. The pre-moistened soil is then placed in a sealed container to mellow overnight (at least 16 hours).

Step 7. Next, we need to determine the mass of concentrated stabilizer (M_c) that must be added to the soil sample, based on the AMR and the mass of dry soil solids in our sample.

$$M_c = AMR \times M_s = \frac{1}{5{,}000} \times 9{,}823 \text{ g} = 1.965 \text{ g} = 2.0 \text{ g}$$

In Step 2, the stabilizer was diluted at the DMR of 1/137.9. To get a mass of chemical of M_c = 2.0 g, we need to measure out a mass of diluted product equal to

$$\text{Mass of diluted } X = M_c + (M_w)_c = M_c\,(1 + 1/DMR) = 2.0(1+137.9) = 278 \text{ g}$$

Step 8. Thoroughly mix 278 g of diluted product *X* with the soil from Step 5. The mass of water added to the soil at this point is

$$(M_w)_c = 278 \text{ g} - M_c = 278 - 2.0 = 276 \text{ g}$$

If there is negligible evaporation, the amount of water currently in the soil will be

$$M_w = (M_w)_s + (M_w)_c = 2{,}279 + 276 = 2{,}555 \text{ g}$$

Therefore, the sample will now contain M_s = 9,823 g, M_w = 2,555 g, and M_c = 2 g. To confirm, the target AMR and OWC have been met,

$$\text{Stabilizer content} = \frac{2 \text{ g}}{9{,}823 \text{ g}} = \frac{1}{4{,}912} \cong \frac{1}{5{,}000} = AMR$$

$$\text{Water content} = \frac{2{,}555 \text{ g}}{9{,}823 \text{ g}} = 26\% = OWC + 2\%$$

Note that the actual water content will be less than 26% due to evaporation losses and, thus, closer to the OWC desired.

Step 9. Allow one hour of standing time for the soil and stabilizer to interact prior to compaction. The treated soil is kept in a sealed container during this period.

Step 10. The stabilized soil is then compacted using the Tex-113-E compaction effort.

Step 11. Extrude the sample from the compaction mold, seal in an airtight container, and place in a moist curing room.

Step 12. The compacted treated soil sample is allowed to cure in the sealed container at room temperature for 7 days in the moist curing room.

Step 13. The cured sample is trimmed in preparation for geotechnical testing. The water content of the specimen trimmings is measured to be 24.8%.

Step 14. Because the water content of the trimmings is within ±2% of the optimum water content for compaction, the specimen is acceptable for testing.

APPENDIX R

APPLICATION GUIDELINES FOR NONTRADITIONAL LIQUID CHEMICAL SOIL STABILIZERS

In Research Project 7-1993, the reaction mechanisms and effectiveness of three nontraditional liquid chemical soil stabilizers were evaluated in detail. The three products studied were selected to represent the common types of such products currently on the market. Some evidence of the reactions between the chemicals and the soils was observed. However, no consistent, significant improvement was measured in the engineering properties of eight different clay soils, when these soils were treated with the stabilizers at the suppliers' recommended application rates and at ten times the recommended application rates. Although effective liquid chemical soil stabilizers may exist, it is prudent to view supplier claims with skepticism until the performance of such products are clearly quantified through objective laboratory testing or controlled field trials.

The findings of this study do not support the implementation of these products in the field. However, it is possible that these or other liquid chemical products may prove to be effective on other soils or at higher application rates. Potential applications of these products should be preceded by conducting standard laboratory tests to quantify the effectiveness of the treatment on a particular soil type at a given chemical application rate. Guidelines for evaluating the potential performance of nontraditional liquid chemical soil stabilizers are given in this checklist:

- ❏ Sales literature from the product suppliers and testimonials from other users should be considered inadequate and unreliable for demonstrating product effectiveness.

- ❏ An appropriate product application rate should be determined for the project-specific soils. More research is needed to determine what minimum engineering properties are needed to justify the application of a soil stabilizer in pavement applications.

- ❏ Initial estimates of appropriate application rates can be determined through micro-characterization studies of treated and untreated samples. X-ray diffraction of oriented and gylcolated samples and BET surface area analysis are useful for assessing changes in soil characteristics.

- ❏ Chemical application rates should be expressed in a consistent manner. Implementation of the application mass ratio (AMR), which is defined as the mass of concentrated chemical product per mass of oven-dry soil, is recommended.

- ❏ Laboratory investigations of the effectiveness of chemical soil treatments should include multiple tests on identically prepared specimens, with tests on both the untreated soil and soil treated at the appropriate rates. Standard, accepted test methods should be followed to measure the engineering properties of interest.

- ❑ A rational protocol for preparing test specimens should be followed. A suitable protocol, which includes control of specimen water content and a seven-day cure at constant moisture, was developed in this study.

- ❑ The shear strength of treated soils should be evaluated using standard test methods. Unconsolidated, undrained, triaxial compression tests are recommended.

- ❑ The expansiveness or potential swell of treated soils should be evaluated using standard test methods.

- ❑ Tests to measure the stiffness of untreated and treated soils, such as resilient modulus tests, should be considered.

- ❑ Field tests of soil stabilizers in pavement base or subgrade layers must include untreated control sections and quantitative measurements of performance.

- ❑ For products that are found to produce significant improvements in soil properties, additional studies will be needed to assess the permanence and long-term effectiveness of the product.