Fifth Quarterly Report
April 1, 1999 through June 30, 1999

Utilization of Low NO_X Coal Combustion By-Products

DE-FC26-98FT40324--03

Submitted by:

Institute of Materials Processing
Michigan Technological University
1400 Townsend Drive
Houghton, MI 49931

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1.0 INTRODUCTION

The project has switched focus this quarter from pilot plant operations to product testing. Last quarter extensive pilot plant work had occurred and testing objectives had been met. Also last quarter technology demonstrations were also performed for Potomac Electric Power, Virginia Power, and Wisconsin Electric. We had reported that groundbreaking for the PEPCo fly ash treatment facility was to begin in August. Recent conversations with the technology's licensee, Mineral Resource Technology, have resulted in changes. Long term contract negotiations between MRT and Potomac Electric Power have caused delays. Most recent estimates are that contract negotiations should be finished in August, detailed engineering is to begin in September, and groundbreaking to begin in early Spring. The commercialization of the technology is going forward, just not as fast as we or MRT had anticipated.

As this is being written we have received inquiries from Plastics Technology Magazine about fly ash utilization in plastics. We are anticipating working with one of their editors to provide an upcoming article.

Task 1.0 Test Plan

Completed.

Task 2.0 Laboratory Characterization

Completed.

Task 3.0 Pilot Plant Testing

Completed.

Task 4.0 Product Testing

<u>4.1 Concrete Testing</u>

The focus of concrete testing this quarter was on using clean fly ash produced during closed loop testing. By using material produced in closed loop, the process water becomes saturated with calcium. Theoretically, therefore, more of the pozzolanic qualities of the fly ash should be seen in the closed loop clean fly ash. This should result in strength values somewhere between as-received fly ash and open circuit processed clean fly ash.

Table 1 depicts the compoarisons of 35S control mixture, PEPCo as-received fly ash, PEPCo open circuit clean fly ash, and PEPCo closed circuit clean fly ash. The 91 day strengths for the PEPCo closed circuit clean fly ash testing are not completed.

The data for 7 day strength values are quite interesting. Using 10% fly ash for cement replacement the closed loop clean fly ash values lie between as-received and open circuit clean fly ash. At 20% cement replacement the closed loop clean fly ash out performs as-received fly ash and open circuit clean fly ash, but slightly less than control values. The 30% replacement values have the same patterns as the 20% value, however, the difference in strength values are not as great, but the value is greater than 3500 psi quality specification.

For 28 day strength values, closed loop clean fly ash produced lower values than control, as-eceived fly ash, and open circuit clean fly ash at all replacement levels of 10, 20, and 30 percent. The values are lower but still within reasonable limits of the other materials.

As mentioned previously the 91 day values are not complete for the closed loop clean fly ash. Historically, high strength values at 91 days has not been a problem, and there has been little difference between as-received and open loop clean fly ash. The complete story will be available at the end of next quarter on the effectiveness of closed loop clean fly ash.

4.2 Fly Ash as Coke

Formcoke samples have been calcined and testing on specific density, porosity, compression strength and ash content this quarter. Table 2 shows the densities and porosities of the samples. Table 3 presents the compression strengths of the formcoke samples. Table 4 lists their carbon and ash contents.

The samples show fairly low densities and large porosities, which are preferred traits of formcoke. The samples, prepared from twelve recipes, show very different strengths after calcinations. This is similar to the green compression strengths which were determined last quarter. We will analyze the strength data and other properties to derive an optimum recipe. The sulfur analysis for the sample is currently being performed. The major drawback at this time is the high ash content of the samples.

Table 1. Comparative Concrete Data of PEPCo As-Received Fly Ash, Open Circuit Clean Fly Ash and Closed Loop Clean Fly Ash										
Samples	**Control**	**PEPCo As-Rec'd. Fly Ash**			**PEPCo Clean Fly Ash - Open Circuit -**			**PEPCo Clean Fly Ash - Closed Circuit -**		
		10%	**20%**	**30%**	**10%**	**20%**	**30%**	**10%**	**20%**	**30%**
Cement, lb.	52.19	46.97	41.75	36.53	46.97	41.75	36.53	46.97	41.75	36.53
Fly Ash, lb.	--	5.22	10.44	15.66	5.22	10.44	15.66	5.22	10.44	15.66
Water, lb.	27.43	25.25	25.10	25.05	25.05	25.00	24.85	25.50	24.90	24.10
W/C Ratio (%)	52.56	48.38	48.09	48.00	48.00	47.90	47.61	48.86	47.71	46.18
Fine Agg., lb.	106.42	106.42	106.42	106.42	106.42	106.42	106.42	106.42	106.42	106.42
Coarse Agg., lb.	170.70	170.70	170.70	170.70	170.70	170.70	170.70	170.70	170.70	170.70
Slump, in.	3.50	3.25	2.75	3.12	2.75	3.50	3.50	3.0	3.38	3.0
Air, %	2.4	2.0	1.9	1.85	2.0	2.0	1.90	1.80	1.90	2.1
Density, #/Ft.^{3}										
7		154.52	153.99	153.66	153.89	153.53	153.47	153.42	152.87	153.20
28	153.78	154.73	154.51	153.91	153.88	153.50	153.38	153.16	152.91	153.17
91	154.10	154.66	153.79	154.28	153.96	153.70	153.51			
7 day strength (psi)										
	4210	4386	3749	3395	4156	3678	3555	4219	4011	3388
	3979	4032	3802	3430	4138	3572	3395	4046	4096	3562
	4280	4350	3731	3430	3802	3537	3440	3967	3700	3700
Avg.	4126	4256	3760	3419	4032	3596	3463	4077	3936	3550
28 day strength (psi)										
	5880	6101	5632	5062	5889	5310	4978	5174	5351	4916
	5898	5960	5234	4956	5800	5517	5234	5542	4994	4976
	5730	5906	5500	5022	5553	5517	5340	4323	5107	4651
Avg.	5836	5989	5458	5013	5747	5448	5184	5346	5151	4848
91 day strength (psi)										
	7143	7137	6864	6570	6944	6621	6603	N/A	N/A	N/A
	7032	7484	6934	6452	7011	6726	7099	N/A	N/A	N/A
	6959	7148	7098	6440	6791	6804	6989	N/A	N/A	N/A
Avg.	7045	7256	6965	6487	6915	6717	6897	N/A	N/A	N/A
N/A = Not available										

Table 2. Density and Porosity of Formcoke Samples Calcined at 1000°C for One Hour Carbon theoretical density = 2.0 g/cm^3, Handbook of Applied Engineering Science, p180						
Sample #	**Height, mm**	**Dia., mm**	**Vol., cm^3**	**Mass, g**	**Density, g/cm^3**	**Porosity, %**
1	15.34	28	9.441	7.558	0.801	60
1	15.36	28	9.453	7.335	0.776	61
1	15.16	28	9.330	7.35	0.788	61
1	15.86	28	9.761	7.491	0.767	62
1 avg.	15.43	28	9.496	7.434	0.783	61
2	17.42	28	10.721	8.286	0.773	61
2	17.24	28	10.610	8.179	0.771	61
2	17.57	28	10.813	8.256	0.764	62
2	17.46	28	10.746	8.348	0.777	61
2 avg.	17.42	28	10.723	8.267	0.771	61
3	19.98	28	12.296	9.217	0.750	63
3	19.73	28	12.143	9.424	0.776	61
3	20.09	28	12.364	9.247	0.748	63
3	19.98	28	12.296	9.337	0.759	62
3 avg.	19.95	28	12.275	9.306	0.758	62
4	15.70	28	9.662	8.163	0.845	58
4	15.98	28	9.835	8.221	0.836	58
4	15.49	28	9.533	8.205	0.861	57
4	15.49	28	9.533	8.304	0.871	56
4 avg.	15.67	28	9.641	8.223	0.853	57
5	17.91	28	11.023	8.163	0.741	63
5	18.26	28	11.238	8.231	0.732	63
5	18.13	28	11.158	8.287	0.743	63
5	18.41	28	11.330	8.360	0.738	63
5 avg.	18.18	28	11.187	8.260	0.738	63
6	17.32	28	10.659	8.467	0.794	60
6	17.32	28	10.659	8.434	0.791	60
6	16.99	28	10.456	8.390	0.802	60

Table 2. Density and Porosity of Formcoke Samples Calcined at 1000°C for One Hour Carbon theoretical density = 2.0 g/cm^3, Handbook of Applied Engineering Science, p180						
Sample #	**Height, mm**	**Dia., mm**	**Vol., cm^3**	**Mass, g**	**Density, g/cm^3**	**Porosity, %**
6	16.80	28	10.339	8.399	0.812	59
6 avg.	17.11	28	10.529	8.423	0.800	60
7	14.90	28	9.170	7.645	0.834	58
7	15.32	28	9.429	7.842	0.832	58
7	15.25	28	9.385	7.577	0.807	60
7	15.12	28	9.305	7.718	0.829	59
7 avg.	15.15	28	9.322	7.696	0.825	59
8	18.61	28	11.453	9.321	0.814	59
8	18.37	28	11.306	9.239	0.817	59
8	18.89	28	11.626	9.246	0.795	60
8	18.46	28	11.361	9.259	0.815	59
8 avg.	18.58	28	11.436	9.266	0.810	59
9	21.53	28	13.250	9.425	0.711	64
9	21.15	28	13.017	9.337	0.717	64
9	21.25	28	13.078	9.332	0.714	64
9	21.41	28	13.177	9.314	0.707	65
9 avg.	21.34	28	13.130	9.352	0.712	64
10	17.06	28	10.499	7.912	0.754	62
10	17.52	28	10.783	7.893	0.732	63
10	17.36	28	10.684	8.086	0.757	62
10	16.66	28	10.253	7.892	0.770	62
10 avg.	17.15	28	10.555	7.946	0.753	62
11	17.24	28	10.610	7.615	0.718	64
11	17.32	28	10.659	7.537	0.707	65
11	16.06	28	9.884	7.187	0.727	64
11	17.75	28	10.924	7.653	0.701	65
11 avg.	17.09	28	10.519	7.498	0.713	64
12	15.16	28	9.330	7.258	0.778	61

Table 2. Density and Porosity of Formcoke Samples Calcined at 1000°C for One Hour Carbon theoretical density = 2.0 g/cm^3, Handbook of Applied Engineering Science, p180						
Sample #	**Height, mm**	**Dia., mm**	**Vol., cm^3**	**Mass, g**	**Density, g/cm^3**	**Porosity, %**
12	13.57	28	8.352	9.511	1.139	43
12	14.68	28	9.035	6.898	0.764	62
12	15.22	28	9.367	7.303	0.780	61
12 avg.	14.66	28	9.021	7.743	0.858	57

Table 3. Compression Strengths of Formcoke Samples Calcined at 1000°C for One Hour 0.5 inch/min crosshead speed			
Sample #	**Dia., mm**	**Load, lbs.**	**Compression Strength, psi**
1	28	144.50	151.48
1	28	117.60	123.28
1	28	146.60	153.68
1	28	167.80	175.90
1 avg.	28	144.13	151.08
2	28	296.10	310.40
2	28	308.30	323.19
2	28	231.80	242.99
2	28	314.50	329.69
2 avg.	28	287.68	301.57
3	28	633.00	663.57
3	28	611.00	640.51
3	28	572.60	600.25
3	28	542.00	568.17
3 avg.	28	589.65	618.12
4	28	386.60	405.27
4	28	654.80	686.42
4	28	428.20	448.88

Table 3. Compression Strengths of Formcoke Samples Calcined at 1000°C for One Hour 0.5 inch/min crosshead speed			
Sample #	**Dia., mm**	**Load, lbs.**	**Compression Strength, psi**
4	28	514.60	539.45
4 avg.	28	496.05	520.00
5	28	282.70	296.35
5	28	300.50	315.01
5	28	298.40	312.81
5	28	304.20	318.89
5 avg.	28	296.45	310.77
6	28	441.60	462.93
6	28	400.50	419.84
6	28	508.50	533.06
6	28	370.30	388.18
6 avg.	28	430.23	451.00
7	28	478.90	502.03
7	28	553.60	580.33
7	28	458.80	480.96
7	28	390.30	409.15
7 avg.	28	470.40	493.12
8	28	838.10	878.57
8	28	927.00	971.77
8	28	807.00	845.97
8	28	906.00	949.75
8 avg.	28	869.53	911.51
9	28	564.30	591.55
9	28	457.20	479.28
9	28	614.20	643.86
9	28	517.30	542.28

Table 3. Compression Strengths of Formcoke Samples Calcined at 1000°C for One Hour 0.5 inch/min crosshead speed			
Sample #	**Dia., mm**	**Load, lbs.**	**Compression Strength, psi**
9 avg.	28	538.25	564.24
10	28	281.20	294.78
10	28	231.50	242.68
10	28	279.80	293.31
10	28	315.90	331.16
10 avg.	28	277.10	290.48
11	28	71.30	74.74
11	28	67.20	70.45
11	28	58.60	61.43
11	28	79.60	83.44
11 avg.	28	69.18	72.52
12	28	738.00	773.64
12	28	816.10	855.51
12	28	702.30	736.21
12 avg.	28	752.13	788.45

Table 4. Carbon and Ash Contents of Formcoke Samples Calcined at 1000°C for 24 hrs					
Sample #	**Sample Wt., g**	**Sample & Crucible, g**	**After Firing, g**	**Carbon Content, %**	**Ash Content, %**
1	1.106	35.847	35.401	40.325	59.675
2	1.068	35.443	34.993	42.135	57.865
3	1.585	35.963	35.189	48.833	51.167
4	1.133	35.624	35.143	42.454	57.546
5	1.101	35.310	34.833	43.324	56.676
6	1.430	36.075	35.455	43.357	56.643
7	1.290	32.679	32.092	45.504	54.496
8	1.233	36.515	35.889	50.770	49.230
9	1.253	36.681	36.066	49.082	50.918
10	1.256	36.007	35.482	41.799	58.201
11	1.142	35.850	35.355	43.345	56.655
12	1.275	35.694	35.182	40.157	59.843

4.3 Fillers - Plastic Fillers

During this reporting period, the injection molding and compression molding of thirty batches of plastic compounds was performed. The plastic compounds utilized were nylon, ABS, and polycarbonate. The fillers that were employed were calcium carbonate, fine clean ash, and calcined fine clean ash. Mechanical testing on the injection and compression molding specimens will be completed next quarter.

A pilot plant size classification of PEPCo clean fly ash was conducted last quarter which generated 3 , 5 , 10 , and 15 fly ash in 28 batches. The classified material was characterized for particle size distribution last quarter. This quarter these materials were characterized for LOI, loose density, tap density, and moisture content. The results are displayed in Table 5.

Table 5. Characterization Results of PEPCo Fine Clean Fly Ash														
Sample	**Tare, g**	**Sample, g**	**Tot. Burnt, g**	**Sample Burnt, g**	**% LOI**	**Mass**	**Loose Vol.**	**Tap Vol.**	**Loose Den. g/ml**	**Tap Den. g/ml**	**Tare, g**	**Sample, g**	**Dry, g**	**Moist., %**
1-f	14.1690	1.0007	15.1492	0.9802	2.049	42.260	55.0	45.0	0.7684	0.9391	12.6670	5.0391	17.6745	0.627
1-c	12.0914	1.0000	13.0861	0.9947	0.530	58.075	50.0	42.0	1.1615	1.3827	14.6890	5.0660	19.7442	0.213
2-f	14.6199	1.0018	15.6004	0.9805	2.126	45.554	56.0	47.0	0.8135	0.9692	11.7460	5.0156	16.7322	0.586
2-c	12.3813	1.0017	13.3771	0.9958	0.589	55.415	49.0	40.0	1.1309	1.3854	14.3751	5.0948	19.4605	0.185
3-f	12.0994	1.0018	13.0812	0.9818	1.996	26.865	34.0	28.5	0.7901	0.9426	12.7848	5.0416	17.7978	0.567
3-c	13.2641	1.0002	14.2616	0.9975	0.270	51.044	45.0	32.5	1.1343	1.5706	13.2708	5.0049	18.2639	0.236
4-f	11.7444	1.0012	12.7316	0.9872	1.398	47.275	51.0	45.0	0.9270	1.0506	12.6584	5.0742	17.6387	1.851
4-c	12.1097	1.0013	13.1064	0.9967	0.459	58.200	47.0	40.0	1.2383	1.4550	11.7462	5.0807	16.8207	0.122
5-f	14.3748	1.0002	15.3654	0.9906	0.960	45.048	47.5	40.0	0.9484	1.1262	12.6671	5.0890	17.7371	0.373
5-c	13.8122	1.0010	14.8081	0.9959	0.509	62.060	52.0	43.0	1.1935	1.4433	14.1650	5.0296	19.1895	0.101
5-f 10 min	13.8850	1.0015	14.8712	0.9862	1.528	44.570	50.0	41.0	0.8914	1.0871	13.6358	5.0972	18.7054	0.541
5-f 30 min	13.8928	1.0002	14.8818	0.9890	1.120	46.950	51.0	44.0	0.9206	1.0670	12.0965	5.0579	17.1345	0.393
5-c 30 min	14.6187	0.9995	15.6143	0.9956	0.390	57.732	48.5	40.0	1.1904	1.4433	12.1105	5.0800	17.1828	0.152
6-f	12.0911	1.0005	13.0821	0.9910	0.950	52.782	50.0	42.5	1.0556	1.2419	13.8856	5.0261	18.8975	0.283
6-c	13.2652	1.0016	14.2627	0.9975	0.409	62.850	52.0	45.0	1.2087	1.3967	14.6927	5.0880	19.762	0.368
7-f	14.6888	1.0000	15.6835	0.9947	0.530	59.446	53.0	47.0	1.1216	1.2648	13.6355	5.0493	18.6744	0.206
7-c	12.0994	1.0012	13.0927	0.9933	0.789	59.271	49.0	43.0	1.2096	1.3784	13.8155	5.0014	18.8009	0.320
8-f	13.6778	1.0005	14.6744	0.9966	0.390	54.438	47.5	41.0	1.1461	1.3278	11.8172	5.0146	16.8305	0.026
8-c	12.3119	1.0021	13.3092	0.9973	0.479	56.278	50.0	45.0	1.1256	1.2506	12.7870	5.0348	17.8088	0.258

Table 5. Characterization Results of PEPCo Fine Clean Fly Ash														
Sample	**Tare, g**	**Sample, g**	**Tot. Burnt, g**	**Sample Burnt, g**	**% LOI**	**Mass**	**Loose Vol.**	**Tap Vol.**	**Loose Den. g/ml**	**Tap Den. g/ml**	**Tare, g**	**Sample, g**	**Dry, g**	**Moist., %**
9-f	13.9104	1.0018	14.9110	1.0006	0.120	54.675	48.0	41.0	1.1391	1.3335	14.1703	5.0845	19.2466	0.161
9-c	12.3817	1.0012	13.3820	1.0003	0.090	56.782	47.5	43.0	1.1954	1.3205	13.8860	5.0214	18.9025	0.098
10-f 10 min	12.0924	1.0022	13.0755	0.9831	1.906	40.674	49.5	41.5	0.8217	0.9801	12.0964	5.0378	17.1087	0.506
10-f 20 min	13.8135	1.0015	14.7994	0.9859	1.558	48.565	56.0	47.0	0.8672	1.0333	13.2649	5.0150	18.2556	0.485
10-c 30 min	14.6886	1.0004	15.6832	0.9946	0.580	57.310	48.5	40.0	1.1816	1.4328	14.1645	5.0386	19.1960	0.141
10-f 1 hr	13.8846	1.0069	14.8773	0.9927	1.410	42.333	49.0	42.0	0.8639	1.0079	14.1700	5.0036	19.1491	0.490
10-c 1 hr	11.7445	1.0011	12.7403	0.9958	0.529	56.781	47.0	39.0	1.2081	1.4559	14.3750	5.0229	19.3903	0.151
10-f 2 hr	13.6779	1.0018	14.6576	0.9797	2.206	44.908	52.0	42.0	0.8636	1.0692	13.2657	5.0038	18.2490	0.410
10-c 2 hr	13.2645	1.0001	14.2582	0.9937	0.640	59.940	51.0	42.5	1.1753	1.4104	12.1100	5.0052	17.1082	0.140

The data clearly shows differences in LOI and density between the fine and coarse fractions after each classification (identified by test number followed by -f for fine or -c for coarse). The fine fractions have higher LOI values and lower densities, which is believed to be caused by residual carbon in the clean fly ash. The working principle of the air size classification was based on the differences in weight of different sized homogeneous fly ash particles. Since there exists some residual carbon in the clean fly ash material, the coarser carbon will have the same separation tendency of finer more dense fly ash. A magnetic test check indicated that the coarse fraction had more magnetic particles than the fine fraction. Conversely these iron rich more dense particles even though fine would want to report to the coarse fraction of the fly ash. It is expected that the slight uneven distribution of carbon and iron particles will not cause serious quality problems for plastic filler applications.

4.4 Activated Carbon

As reported last quarter we have built a system for evaluating the mechanism for mercury adsorption on fly ash carbon. The findings to this point are that the adsorption testing is extremely sensitive to moisture. The nitrogen carrying gas therefore, has to be moisture free.

Also, when the system is purged to condition the carbon surface, it has been identified that oxygen to some degree has to be present in the purging gas.

Based on these findings we have concluded that our test system has to be redesigned for moisture minimization and dry bottled air has to be used as the purging gas. It is believed that it is crucial for surface oxygen groups to be present on the surface of the carbon for mercury adsorption to take place. We have begun revamping the test system to accommodate the requirements. It is anticipated that quality comparable data will be produced with the new system next quarter.

4.5 Additives for Powder-Based Aluminum Composites

During this reporting period, preliminary pressure casting trials of aluminum-fly ash composites were carried out and metal powders were received from Ames Laboratory (USDOE) for production of the next series of powder-metallurgy-based aluminum/fly ash metal matrix composites. The pressure casting trial was carried out at MER Corporation of Tucson, AZ. Beneficiated fly ash having a mean particle size of 5 was pressure-infiltrated with 6061 aluminum to produce a composite containing nominally 50 vol%ash. The composite was produced with little difficulty, and exhibited a 4-point bend strength of 400 Mpa (58 ksi). A micrograph of the fracture surface is shown in Figure 1. Note that there appears to be good bonding between the aluminum and the fly ash, as evidenced by the lack of clearly-visible fly ash particles on the fracture surface.

These results illustrate the potential for using squeeze casting to produce a low-cost aluminum/fly ash “composite concentrate” for remelting with additional aluminum. As aluminum/fly ash technology develops, there is likely to be a substantial potential market for cast aluminum/fly ash composites, since aluminum castings are widely used for a range of applications, including automotive parts. Current methods for producing cast aluminum/fly ash composites, however, are limited to fairly large fly ash particles, and often do not yield a uniform distribution of fly ash in the final composite.

Both of these factors limit the mechanical properties of the final cast material. The squeeze-cast "composite concentrate" approach offers the potential for using finer fly ash particles and producing a more uniform distribution, both of which are likely to enhance the mechanical properties of the final part.

Approximately 500 g of aluminum powder was received from Ames Laboratory (USDOE) at Iowa State University. This material was produced using the advanced GARS (Gas Atomized Reaction Synthesis) technology developed at that laboratory and exhibits substantially-improved sinterability over conventional commercial materials. Accordingly, it is likely to be particularly suitable for producing high-density powder-metallurgy-based aluminum/fly ash composites. During the next reporting period, aluminum/fly ash composite transverse rupture test specimens and wear testing coupons will be fabricated using this powder and evaluated.

Composites will be fabricated containing 0, 10, and 20 vol% beneficiated, sized fly ash. A blended elemental alloy having the composition Al-4.4 wt% Cu-0.8wt% Si-0.5 wt% Mg will be used as the matrix. This composition is similar to 201AB, a commercial aluminum powder metallurgy alloy currently used for bearing caps in some automotive engines. The primary anticipated advantage of fly ash additions for something like bearing caps is that the ash should improve the hardness and wear resistance of the bearing surface. The ash will be blended with matrix powders and then cold pressed to approximately 90% of theoretical density. It will then be sintered under a protective atmosphere and heat-treated to a T6 temper. Initial processing trials will be performed using sintering and heat treatment conditions similar to those used for the commercial 201AB alloy, although these conditions may eventually need to be modified slightly to compensate for the presence of the ash. Both the transverse rupture and wear testing of the composites will be done at Michigan Technological University.