An ASABE Meeting Presentation

Paper Number: 064043

Assessment of Chemical and Physical Characteristics of Bottom, Cyclone, and Baghouse Ashes from the Combustion of Manure

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**Written for presentation at the
2006 ASABE Annual International Meeting
Sponsored by ASABE
Portland Convention Center
Portland, Oregon
9 - 12 July 2006**

Abstract. *A large energy company has proposed to build an ethanol plant in Hereford, Texas. Energy from manure will be extracted in a fluidized bed combustion process and be provided to the plant as steam. The steam plant will produce about 28 tons (25,000 kg) of ash per hour.*

Characterization and classification of the ash material was determined from physical and chemical testing. In addition to particle size analysis and engineering classification, Atterberg limits, including plastic limit, liquid limit, and plasticity index, along with compaction characteristics relating bulk density to moisture content, were determined. To determine the preliminary suitability of the ash as a building material, Portland cement was mixed with the ash and compression testing was performed.

Results of the physical testing show that the ash is suitable as a subgrade material for road construction. Also, an ash-cement product could be used for feedlot surfacing, road base, and other structural building projects. However, the ash is not self-cementing. Results from chemical analysis show that the ash is classified as a Nonhazardous Industrial Class 2 waste according to Texas state regulations.

Keywords. Manure, ash, soil, cement, physical, chemical, cattle, compressive strength, feedyard.

Introduction

A proposed ethanol plant will be powered by an adjacent manure-fired steam plant. The steam plant will produce about 28 tons (25,000 kg) of ash per hour. During a recent pilot plant burn test, three different combinations of cattle manure and cotton gin trash were burned and representative ash samples from each combination were collected:

100% Cattle Manure
75% Cattle Manure/25% Cotton Gin Trash
50% Cattle Manure/50% Cotton Gin Trash

At the request of the energy company, this preliminary research focused solely on the ash from the 100% cattle manure.

Before removing the ash, chemical tests will determine whether or not the ash is hazardous. If it is, it will be characterized as ignitable, reactive, corrosive, or toxic (TCEQ, 2005). Disposal fees would likely shut the project down.

If the ash is nonhazardous, it will be classified as Class 1, Class 2, or Class 3 Nonhazardous Industrial waste. Class 1 is considered potentially threatening to human health and environment and therefore requires special handling. Class 2 is considered non-threatening but possibly reactive. Class 3 includes demolition debris and related materials which are non-threatening and do not react with other materials, and do not decompose. Most landfills will accept Class 2 and Class 3 materials, but disposal fees and waste transport will still cost the company to remove the ash.

Physical testing is used to determine the characteristics of the ash as a building material, road base, or surface additive. If testing concludes that the ash is suitable for such cases, it may provide both an inexpensive alternative for consumers and a profitable removal for the energy company.

Literature Review

Fluidized Bed Combustor (FBC) ash was tested by Iowa State University for use as a soil stabilizer in feedlots (Greenlees et al., 1998). FBC ash results from burning high-sulfur coal. The combustion takes place in a “bed” of coal and limestone that is fluidized by forcing atmospheric air into the combustion chamber from the bottom of the unit. The University’s physical plant was estimated to produce between 25,000 and 30,000 tons of this ash each year, and because disposal of the ash requires a waste fee, finding uses for the ash is ideal. In general, soil stabilized with FBC ash was stronger than untreated soil. Field results showed that soil strength could be improved 200% to 300% using standard farm equipment to stabilize the soil. The cost of using this method could be less than one-tenth that of paving the feedlot with concrete. This same method of combustion was used to burn manure for powering the ethanol plant.

Meanwhile, the use of soil cement and fly ash-cement has also been investigated for hard-surfacing feedlot pens to increase animal performance and increase manure quality (Parker et al., 2004; Cox, 2003; Kalinski et al., 2005). Soil cement is a mixture of soil, Portland cement, and water compacted to a high density (Adaska, 1990). Cement concentrations vary between 4% and 16% of dry soil weight. Water content is determined through compaction testing by finding the point of maximum dry density and taking its moisture content (ASTM. 1996d).

When manure is scraped from feedlot pens every six to twelve months, it is usually applied as a fertilizer to agricultural lands. Manure scraped from a hard-surfaced pen has a lower soil content than manure scraped from an earthen pen, so the manure has greater agronomic value. And because hard-surfaced pens drain water faster, they are less prone to become muddy and sluggish, saving the cattle energy and the rancher feed. Likewise, odor emissions are significantly reduced. The cost of soil cement for hard-surfacing is about one-fifth that of hard-surfacing with concrete (Parker et al., 2004).

Soil cement has also been used successfully for road- and airport runway-surfacing (Adaska, 1990; PCA, 2002). Additionally, soil cement is big in Texas water projects (Hansen, 1991). Toledo Bend Dam, for instance, uses 125,000 yd^3 of soil cement for slope protection. The Barney M. Davis Power Station in Corpus Christi uses a 6.2 mile (9.9 km) long soil cement embankment to encircle an 1100-acre cooling water reservoir. A south Texas nuclear plant uses more than 1.2 million yd^3 of soil cement slope protection for the exterior embankment of a 7000-acre cooling water reservoir and interior dykes that guide cooling water around the reservoir.

These established uses of FBC ash-treated soil and soil cement show the relevance of such materials for construction purposes. If manure ash has similar physical properties, or if the ash can be economically and feasibly made to have such properties, the energy company can not only avoid disposal fees, but it may have a profitable way to remove the ash and assure the long-term viability of the plant.

Materials and Methods

Physical Testing

Physical testing was performed on four types of ash: baghouse ash, cyclone ash, bottom ash, and "clinker" material. Baghouse ash could be described as very fine, similar to talcum powder. Cyclone ash was similar to very fine sand mixed with a small amount of baghouse ash. Bottom ash was similar to coarse sand. The clinker material was also a bottom ash product, but was similar to small gravel.

Tests for particle size were performed according the ASTM Standard D 422-63, *Standard Test Method for Particle-Size Analysis of Soils*. The mass of material retained on a sieve was added to all material retained on larger sieves and subtracted from 100 to determine the percent of material finer than the given sieve size. Baghouse ash was tested using a hydrometer and dispersing agent (sodium hexametaphosphate). After mixing the dispersing agent with water at a concentration of 40 g/L, 125 mL of the solution was stirred into 50g of ash and allowed to soak for 16 hours. After soaking, the slurry was transferred to a sedimentation cylinder and water added to obtain a total 1000mL volume. After thoroughly mixing the 1000 mL slurry, the hydrometer was inserted and readings taken at 2, 5, 15, 30, 45, and 60 minutes. Once the particle-size analysis was complete, the ashes were classified according the ASTM Standard D 2487-93, *Standard Classification of Soils for Engineering Purposes (Unified Soil Classification System)*.

Testing to find Atterberg limits was performed according to ASTM Standard D 4318-95, *Standard Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils* (Figure 1). Only baghouse and cyclone ashes had a consistency that allowed them to be tested. Cyclone ash was processed to remove a very small amount of material retained on the 0.167-inch (425μm) sieve. Liquid limit was found using a mechanical device consisting of a brass cup suspended from a carriage and designed to drop onto a hard base. A soil sample with a maximum depth of approximately 0.394 inches (10mm) was spread into the brass cup. A grooving tool was then used to cut a groove through the sample from the back to the front. Finally, the cup was allowed to drop and hit the base. Soil samples with incremented moisture contents were tested until 20-30 drops caused the groove to close along a distance of 0.512 inches (13mm). Liquid limit was calculated as the water content multiplied by a correction factor, k, which varied as the number of required drops drifted away from 25. (At 25 drops, k = 1.000). Plastic limit was found by taking a small elliptical soil sample and rolling it into a ribbon 0.126 inches (3.2mm) in diameter. This ribbon was broken, reformed and rolled again. When the ribbon crumbled at a diameter greater than 0.126 inches (3.2mm), the moisture content was determined. This value is the plastic limit. The plastic limit was subtracted from the liquid limit found earlier to calculate the plasticity index. Figure 1 shows how the plot of liquid limit versus plasticity index can help determine soil classification.

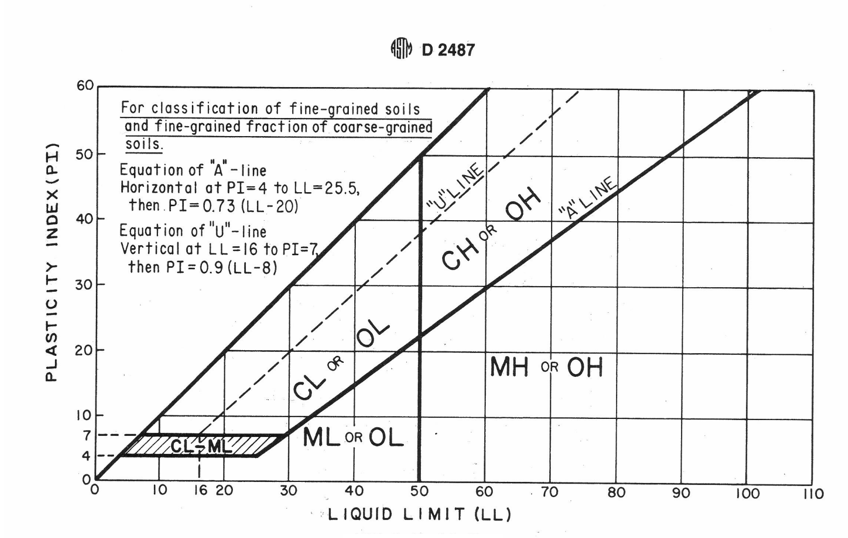

Figure 1. Plasticity Chart from ASTM Standard D 2487-93 *Classification for Engineering Purposes (Unified Soil Classification System).*

Compaction tests were performed according to ASTM Standard D 698-91, *Test Method for Laboratory Compaction Characteristics of Soil Using Standard Effort (12,400 ft-lbf/ft^3 (600 kN-m/m^3))*. Proctor tests are used to determine the optimum moisture content for compacting a soil and the maximum dry density of that soil. Five compactions were made for each test, each with increased moisture content. Each compaction was made by adding the soil sample to a mold with a height of 4.584 inches (116.4mm) and a diameter of 4.0 inches (101.6mm). The soil sample was compacted by raising a 5.5-pound (2.49-kg) rammer 12 inches (304.8mm) inside its guide sleeve and releasing it. The rammer was dropped 25 times. Then more of the soil sample was added and the rammer dropped another 25 times. This procedure was repeated a third time, until three layers had been compacted. The amount of soil sample added to the mold each time was estimated such that each layer would be approximately of equal height. A straightedge was used to clear away the excess height of the final layer after the test was performed. After the mass of the compacted specimen was recorded, a sample was extracted and the moisture content of that sample was determined. These numbers were then used to calculate dry density of the specimen, where dry density equals moist density (specimen mass divided by compaction mold volume) divided by one plus the water content. Finally, plots of moisture content versus dry density were constructed and are presented in Figures 3.1 - 3.3.

For compression testing, specimens were prepared according to ASTM Standard D 698-91, Test *Method for Laboratory Compaction Characteristics of Soil Using Standard Effort (12,400 ft-lbf/ft^3 (600 kN-m/m^3))*. Forty-eight samples were prepared with 35% moisture content. Four samples were made for each condition: baghouse ash, cyclone ash, and a mixture of 70% cyclone/30% baghouse ashes, each prepared with cement contents of 5%, 10%, 15%, and 20%. Samples were allowed to cure in an air-tight, high-humidity environment for 28 days. Compression testing was performed per ASTM Standard D 1633-84, *Standard Test Method for Compressive Strength of Molded Soil-Cement Cylinders*. A hydraulic compression-testing machine was used to fracture the samples to a failure point of 20% below the maximum load. Loading was kept constant at a rate of 200 lb/s (16 psi/s). After each sample was fractured, the average maximum load and the average stress were calculated from the four samples for each varying specimen.

Chemical Testing

Samples of baghouse, cyclone, and bottom ashes were collected and sent to ASK Laboratories, Inc. in Amarillo, Texas to determine their chemical properties. For the October 31st analysis, a baghouse composite was made with 0.165-pound (75-gram) samples from each container (containers numbered 3, 4, 9, 10, 11, 12, 19, 27, 36, 44, 48, 53, 56, and 60); a cyclone composite was made with 0.165-pound (75-gram) samples from every other container (containers numbered 1, 7, 15, 17, 25, 32, 34, 37, 39, 41, 46, 49, 52, 55, and 58); and a bottom composite was made with 1.10-pound (500-gram) samples from each container (containers numbered 42, 43).

For the November 28th analysis, a baghouse composite was made with 0.165-pound (75-gram) samples from each container (containers numbered 3, 4, 9, 10, 11, 12, 19, 27, 36, 44, 48, 53, 56, and 60); a cyclone composite was made with 0.165-pound (75-gram) samples from every other container (containers numbered 2, 8, 16, 18, 26, 33, 35, 38, 40, 45, 47, 50, 54, 57, and 59); and a bottom composite was made with 1.10-pound (500-gram) samples from each container (containers numbered 42, 43). The lab used 2.20 pounds (1000) grams of each composite sample for testing.

According to the Texas Commission on Environmental Quality "Guidelines for the Classification and Coding of Industrial and Hazardous Wastes," a waste can be either Hazardous or Nonhazardous. Distinguishing between these two classifications was performed using the toxicity characteristic leaching procedure (TCLP) (40 CFR Part 261). The TCLP leaches 0.22 pounds (100 grams) of solid sample with 4.41 pounds (2,000 grams) of leaching solution, and the resulting leachate is analyzed for a list of 39 constituents. Only eight of these constituents (i.e. the heavy metals) are likely to be present in ash material. These metals and their maximum TCLP concentrations are presented in the Tables 1.1 – 1.4. The ash was nonhazardous according to the TCLP results.

In addition to the hazardous waste classification tests, the "7-Day Distilled Water Leachate Test" was conducted in order to classify as a Class 1, 2, or 3 waste per regulatory guidelines (TCEQ, 2005). The pH of the ash was also measured by mixing with an equivalent weight of water (1:1 ratio).

The following chemical tests were performed:

1) TCLP metal analysis per 40 CFR Part 261
2) 7-day distilled water leachate test (TCEQ, 2005)
3) Analysis of salts in the 7-day leachate extraction
4) Total Metal Analyses

Table 1.1. Results of 7-day leachate extraction, presented in mg/L. Numbers highlighted are in exceedence of the limit for classification as a Class 3 waste.

7-Day Leachate Extraction		Baghouse Ash		Cyclone Ash		Bottom Ash	
Test	Limit	10/31/05	11/28/05	10/31/05	11/28/05	10/31/05	11/28/05
Arsenic	0.050	0.010	0.048	<0.005	<0.005	<0.005	0.007
Barium	1.000	0.416	0.530	0.222	0.502	0.037	0.041
Benzene	0.005	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Cadmium	0.005	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Carbon Tertachloride	0.005	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Chlordane	0.002	<0.00005	<0.00005	<0.00005	<0.00005	<0.00005	<0.00005
Chlorobenzene	0.100	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Chromium	0.100	0.117	0.194	0.023	0.055	0.023	0.019
2, 4-D	0.070	<0.008	<0.008	<0.008	<0.008	<0.008	<0.008
Dibromochloropropane	0.0002	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002
o-Dichlorobenzene	0.600	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
p-Dichlorobenzene	0.075	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
1, 2-Dichloroethane	0.005	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
1, 1-Dichloroethylene	0.007	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Trans-1, 2-Dichloroethylene	0.100	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
1, 2-Dichloropropane	0.005	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Ethylbenzene	0.700	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Heptachlor	0.0004	<0.00005	<0.00005	<0.00005	<0.00005	<0.00005	<0.00005
Heptachlor Epoxide	0.0002	<0.00005	<0.00005	<0.00005	<0.00005	<0.00005	<0.00005
Lead	0.050	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Mercury	0.002	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002
Methoxychlor	0.040	<0.00005	<0.00005	<0.00005	<0.00005	<0.00005	<0.00005
Pentachlorophenol	0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Selenium	0.050	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Silver	0.050	<0.005	<0.015	<0.005	<0.015	<0.005	<0.015
Styrene	0.100	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Tertachloroethylene	0.005	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
1, 1, 1-Trichloroethane	0.200	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Trichloroethylene	0.005	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Toluene	1.000	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Toxaphene	0.003	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
2, 4, 5-TP (Silvex)	0.050	<0.020	<0.020	<0.020	<0.020	<0.020	<0.020
Vinyl Chloride	0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
M, P-Xylenes	10.000	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
O-Xylene	10.000	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Total Dissolved Solids	500	8345	9626	2115	2176	1310	893

Table 1.2. Results of TCLP Metal Analyses. Results presented in mg/L. (10/31/2005)

Metal	Limit	Baghouse	Cyclone	Bottom
Arsenic	5.0	<0.20	<0.20	<0.20
Barium	100.0	0.15	<0.10	0.51
Cadmium	1.0	<0.05	<0.05	<0.05
Chromium	5.0	0.06	<0.05	<0.05
Lead	5.0	<0.05	<0.05	<0.05
Mercury	0.2	<0.05	<0.05	<0.05
Selenium	1.0	0.31	<0.20	<0.20
Silver	5.0	<0.05	<0.05	<0.05

Table 1.3. Results of salt analyses on the 7-day leachate. Results presented on wet weight basis show amount of salt per unit of solution, mg/L. Results presented on a dry basis show amount of leachable salt per weight of ash, mg/kg. (11/28/2005)

	Baghouse Ash		Cyclone Ash		Bottom Ash	
Test	Wet Basis	Dry Basis	Wet Basis	Dry Basis	Wet Basis	Dry Basis
Calcium	1170	4680	396	1584	120	480
Chloride	2970	11880	187	748	53	212
Magnesium	<10	<40	<10	<40	<1	<4
Nitrate + Nitrite as Nitrogen	<10	<40	<10	<40	<1	<4
Ortho-Phosphate as Phosporus	<50	<200	<10	<40	<1	<4
Potassium	2450	9800	506	2024	48	192
Sodium	497	1988	90	360	15	60
Sulfate	2120	8480	1040	4160	451	1804
Total Alkalinity	338	1352	210	840	68	272
Total Phosporus	<0.5	<2	<0.5	<2	<0.5	<2
Specific Conductance	11900 µmhos/cm		3020 µmhos/cm		1070 µmhos/cm	

Table 1.4. Results of pH and total metals analyses, RCRA metals. Results presented on a total basis, mg/kg. (11/28/2005)

Test	Baghouse Ash	Cyclone Ash	Bottom Ash
Arsenic	<1.0	<1.0	<1.0
Barium	267	220	100
Cadmium	1.2	<1.0	<1.0
Chromium	28.6	18.1	5.6
Lead	59.9	8.0	2.8
Mercury	<0.2	<0.2	<0.2
Selenium	<1.0	<1.0	<1.0
Silver	<1.0	<1.0	<1.0
pH	10.6	10.0	8.0

Results and Discussion

Particle size distribution curves are presented in Figures 2.1 - 2.4. The resulting USCS classifications for each ash are as follows (ASTM, 1996c):

Cyclone Ash:
USCS Classification = *Silty Sand (SM)*— 75% fine sand; 23% silty fines; LL = 58; PI = 6

<u>Bottom Ash:</u>
USCS Classification = *Poorly Graded Sand (SP)*— 4% coarse sand; 75% medium sand; 21% fine sand; nonplastic

<u>Clinker:</u>
USCS Classification = *Poorly Graded Gravel (GP)*— 5% coarse gravel; 95% fine gravel; nonplastic

<u>Baghouse Ash:</u>
USCS Classification = *Silt (ML)*— 100% silt; LL = 42; PI = 1

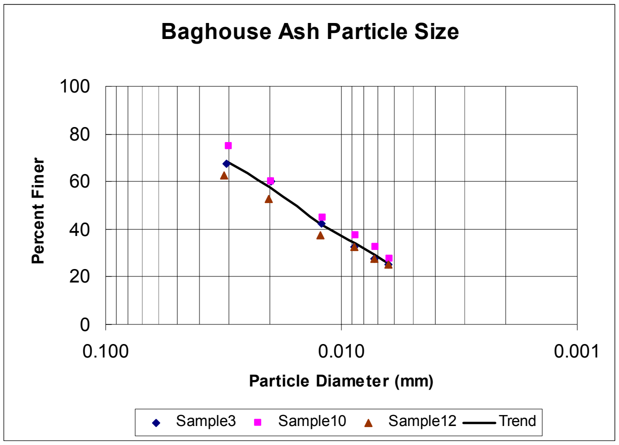

Figure 2.1. Particle size distribution curve for baghouse ash.

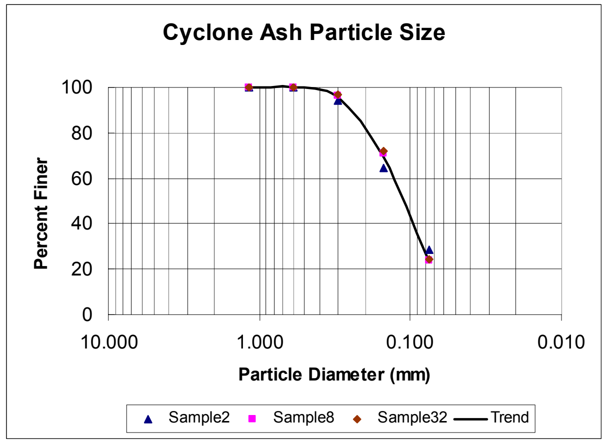

Figure 2.2. Particle size distribution curve for cyclone ash.

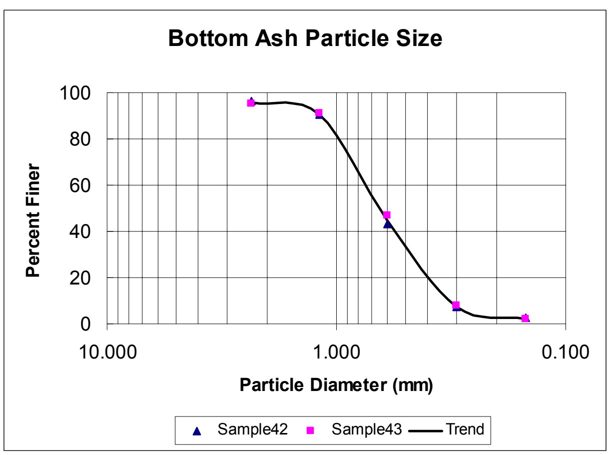

Figure 2.3. Particle size distribution curve for bottom ash.

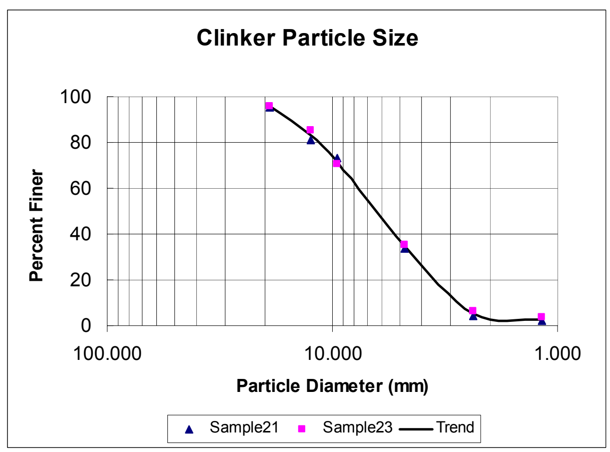

Figure 2.4. Particle size distribution curve for clinker (bottom) ash.

Baghouse ash was found to have a plastic limit between 41 and 42. Liquid limit ranged from 40 to 42, leaving plasticity index to be from zero to one. Therefore, the baghouse ash will be classified as a non-plastic, or cohesionless, soil (ASTM, 1996b).

Cyclone ash was tested and found to have a plastic limit ranging from 56 to 62. The liquid limit was 50 to 56. The plasticity index was calculated at an average of 6. Therefore, cyclone ash has low plasticity and may again be compared to a cohesionless soil.

Third-order polynomials were added to the compaction data plots to show the trends of each ash type. The equation of each line is given and can be used to calculate the maximum dry density and the moisture content at that point. The "R squared" value is also given to show how well the equation fits the data set (the closer to 1.000, the better the equation fits the plotted points).

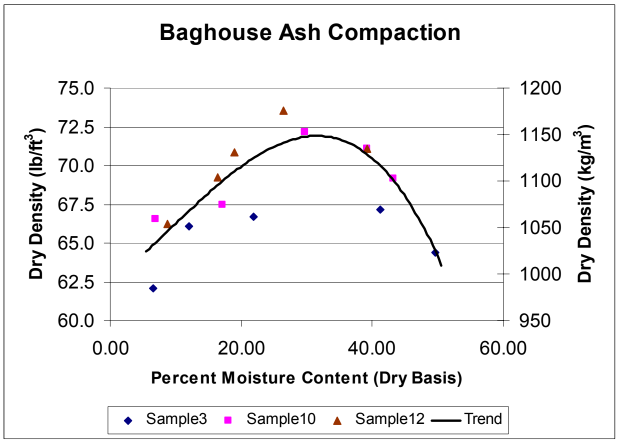

Figure 3.1. Moisture-density relationship for baghouse ash.
$y = -0.0003x^3 + 0.0073x^2 + 0.3567x + 61.796$(lb/ft^3)
$R^2 = 0.7052$
Maximum dry density is 71.9 lb/ft^3 (1148 kg/m^3) at 31.4% moisture content.

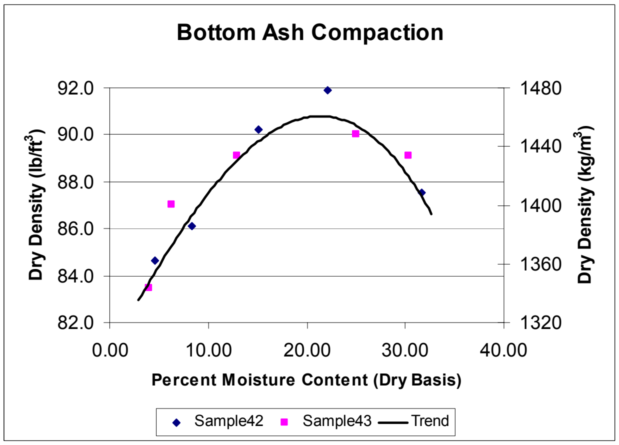

Figure 3.2. Moisture-density relationship for bottom ash.
$y = -0.0003x^3 - 0.0083x^2 + 0.7978x + 81.078$ (lb/ft^3)
$R^2 = 0.9276$
Maximum dry density is 91.4 lb/ft^3 (1460 kg/m^3) at 21.4% moisture content.

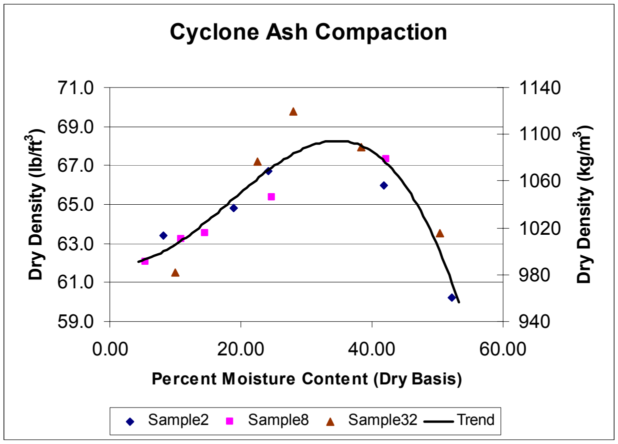

Figure 3.3. Moisture-density relationship for cyclone ash.
y = -0.0004x3 + 0.0203x2 - 0.0704x + 61.822 (lb/ft3)
R2 = 0.8597
Maximum dry density is 68.3 lb/ft3 (1095 kg/m3) at 34.9% moisture content.

After each sample was fractured, the average maximum load and the average stress were calculated from the four samples for each varying specimen. The average compressive strength for each sample is plotted in Figure 4 below. The “Mix” ash type consists of 70% cyclone ash and 30% baghouse ash.

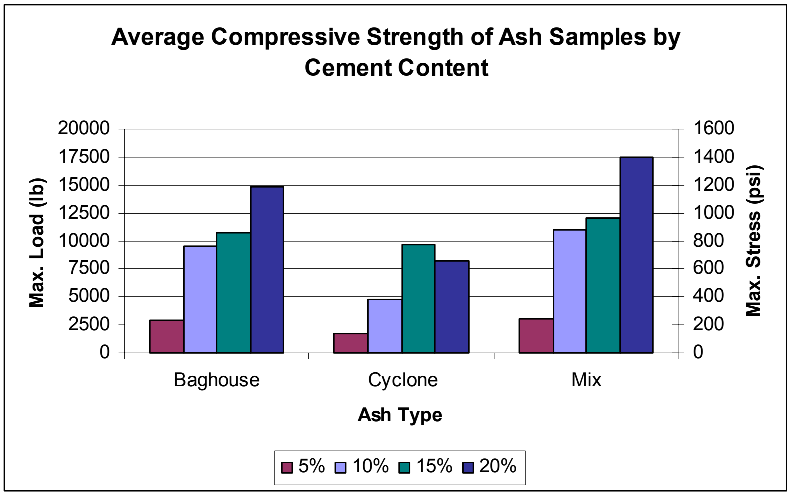

Figure 4. Results of compressive strengths with ash at Portland cement concentration of 5, 10, 15, and 20 percent.

The results of the chemical analyses are presented in Tables 1.1 – 1.4. Based on the results of the chemical testing, all of the ash samples appear to be nonhazardous (TCEQ, 2005). According to the TCEQ designation, the ash is likely a Class 2 waste, as the baghouse ash exceeds the chromium limit in the 7-day leachate test, and all the ash samples exceed the limits for total dissolved solids in the 7-day leachate test (see Table 1.1). Based on the TCEQ guidelines, there is still the possibility of a variance from the waste classification.

Conclusion

Results of the physical testing show that the ash is suitable as a subgrade material for road construction. The addition of 10 percent Portland cement to the ash produces a lightweight concrete-like material with about one-third the compressive strength of concrete (approximately 1000 psi). This ash-cement product could be used for feedlot surfacing, road base, and other structural building projects.

According to state and federal regulations, results of chemical analyses show that the ash produced from burning manure is nonhazardous per federal regulations, and is classified as a Nonhazardous Industrial Class 2 waste per state regulations.

Additional research is warranted on using the ash as a building material or as a supplement to traditional concrete. We recommend further collaboration with industry, feed yards (for pen surface paving projects), and governmental entities (i.e. state and county highway departments) for potential uses of the ash.

References

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