Fly Ash Composites, A Step toward Pond Ash Composites
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ABSTRACT
The study demonstrated that ash composites made from polyurethane and fly ash are able to reduce leaching of coal ash inorganics to less than one-third of the U.S. Environmental Protection Agency's maximum containment limit even after being exposed to an accelerated water circulation system for 14 months. Even if these ash blocks are broken, the composite is safe. The concept of encapsulation of fly ash into ash composite by using a polar polymer to bind the fine inorganic particles, as nature has done in the original unburned coal, makes the ash composite safe. The ash composites have compression and flexure strength and surface hardness suitable for many engineering applications such as building products and infrastructure products that are more valuable than the use of ash in concrete. The application examples demonstrated include reusable ash storage blocks, utility pole crossarms, and building products such as chair railings, base boards, and decorative moldings. In addition, there are many more positive attributes of ash composites such as insect and fire resistance, low electrical and thermal conductivities, and low coefficient of thermal expansion. These properties need to be assessed further. This fly ash-composite technology will be expanded to include pond ash that is very abundant in the United States and worldwide and has very limited use. Other applications such as electric poles, railroad ties, and highway sound barriers are the products for exploration.

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1. Introduction
One of the great challenges in today’s world is the ever-expanding accumulation of waste (created by humans and industrial processes) and its consequences on humans, nature, and the planet. One such waste considered here is the coal ash produced by coal-burning electric power plants. During 2000–2013, the United States alone produced an average of 90 million metric tons of coal ash per year (Jacobs, 2015); about 50% was used in beneficial applications such as concrete (the largest use of coal fly ash), aggregates, bricks, metal–matrix composites, and fillers, and the rest was stored in ponds and landfills. The amount of stored ash in the United States exceeds a billion tons and much more worldwide.

When coal is pulverized and burned in a steam plant, it results in coal ash residuals (about 5–15% of the coal weight based on the coal source and the combustion chamber performance) that consist of fly ash, bottom ash, boiler slag, and sometimes gypsum (Figure 1). Among the four residuals, fly ash is the largest component, representing 60% of the weight, and because of its fine size it can cause air and water pollution. Otherwise, the fly ash consists of oxides of Si, Al, Fe, Ca, Zn, and Ti (>85% by weight) that are among the most chemically and thermally stable, hard, and insect- and fire-resistant materials on earth; they are also considered to be useful raw materials for many products (Shivakumar, 2016; Shivakumar et al., 2017). The secondary elements in fly ash such as As, Sb, B, Be, Mo, Se, Ti, and V are more hazardous for human consumption and can dissolve or be suspended in water (because of their submicrometer size) and can cause groundwater contamination due to seepage and runoff. Some of these contaminants potentially exceed the...
U.S. Environmental Protection Agency’s maximum contaminant limit (US EPA MCL) (Thorneloe et al., 2009) in groundwater, and if consumed can cause human health problems (US EPA, 2017; Clean Air Task Force, 2018). Electric power plants are located near rivers, streams, and lakes where the economically weaker communities live, and ground well water is their only source for household use. The drinking water issue became known after the 2014 Dan River coal ash spill in Eden, NC. The state of North Carolina then mandated that by 2019 the power companies must close some ash ponds, with the ash then being excavated, transported to uninhabited areas, and stored in lined landfills with continuous monitoring and safe disposal of leachates. Recently, many other southeastern states have initiated the inspection of coal ash pond sediments as well. US EPA studies in 2006–2008 (Thorneloe et al., 2009) have shown that coal ash sediments leach out potentially toxic or hazardous inorganics such as arsenic, antimony, barium, boron, cadmium, chromium, molybdenum, selenium, thallium, and vanadium and that these inorganics can contaminate groundwater, in some cases exceeding the US EPA MCL (Thorneloe et al., 2009). Many peer-reviewed articles, the US EPA (2017) and others (Bradley, 2012; Clean Air Task Force, 2018), have reported that the above-listed inorganics exceeding the US EPA MCL in drinking water have caused chronic health problems. At present, there are no simple solutions to this enormous leaching problem of coal ash.

A wealth of publications exist on coal ash. They cover topics such as types of coal (bituminous, semi-bituminous, and lignite) and residuals, mineral content, particle size classification and packing density, and applications in cement concrete, bricks, metal matrix, and fillers (American Coal Ash Association [ACAA], 2009, 2013; Liu et al., 2009; Uhlenhuth, 2014; Brown et al., 2015; Yao et al., 2015). Oberlink and associates published papers relating to sequestration of hazardous elements in coal ash (Oberlink et al., 2011; Robl and Oberlink, 2011). But only limited work was done on encapsulation of hazardous inorganic minerals in coal ash and the use the resulting material to manufacture value-added products (Shivakumar, 2016; Shivakumar et al., 2017).

Another challenge of fly ashes is that they are heavy, with a bulk density between 74 lb/ft³ (1.2 g/cc) and 87 lb/ft³ (1.4 g/cc). This weight is a disadvantage to its use in lightweight products, where an overall density range of 12 lb/ft³ (0.2 g/cc) to 74 lb/ft³ (1.2 g/cc) is needed. A simple rule of mixtures predicts that any polymer-based fly ash composite will have a density of 99 lb/ft³ (1.6 g/cc) to 112 lb/ft³ (1.8 g/cc), the range of concrete. Therefore, a foaming composite technology was explored to bring down the overall density to a range of 25 lb/ft³ (0.4 g/cc) to 74 lb/ft³ (1.2 g/cc), preferably to a range of 6 lb/ft³ (0.1 g/cc) to 74 lb/ft³ (1.2 g/cc). The challenges of this research are encapsulation of hazardous inorganic minerals to reduce leaching into water, production of controlled density of the composite and its mechanical characterization, and demonstration of value-added products. We have chosen a two-part thermoset polyurethane because of its potential for high loading of ash and foaming to produce closed cell foam. This polymer-based composite technology will add a new method of beneficial use of coal ash (Figure 2).

The objectives of the study were to minimize the leaching of inorganic materials from fly ash composites to below the US EPA MCL, demonstrate the density control by the amount of reactant water, and demonstrate the manufacturing of higher value-added products.

2. Materials and Methods

2.1. Materials

Fly ash freshly generated by Belews Creek steam plant located in Stokes County, NC, was used in this study. The ash was supplied by Ash Ventures LLC. This ash is referred as BC ash or source 1 ash. The properties of the ash were medium gray color, pH 4.8 (acidic), and tap and solid densities of 86 lb/ft³ (1.38 g/cc) and 142 lb/ft³ (2.29 g/cc). The major mineral contents were SiO₂ (52.5%), Al₂O₃ (25.9%), and Fe₂O₃ (9.6%), for a total 88.0% of the ash weight. The other minerals included CaO, MgO, N₂O, K₂O, and SO₃ between 1% and 3% each. The loss on ignition was 2.3% (less than the ASTM standard for concrete use) and classified as F class. The moisture content of ash when it left the power plant was about 0.12% and was found to be stabilized to about 0.5% after many days of exposure to ambient conditions. The mean particle diameter was about 45 μm. In addition to the source 1 ash, source 2 (South Carolina) fly ash was also used in assessing the minerals in leachates. The polyurethane resin system consists of two parts: Rubinate M supplied by Huntsman (Huntsman Corp., The Woodlands, TX) and polyols supplied BASF Corporation (Tarrytown, NY). The mixing ratio of the Rubinate M to polyol was approximately 1:1. Polyol consist of Pluracol GP-430 (rigid) and Pluracol GP-730 (semi-rigid). The mixing ratio of the GP-430 to GP-730 used was 2:1. In addition, two surfactants were used: one was for the ash (BYK-W 969, 1 w%
of composite), supplied by BYK USA Inc. (Wallingford, CT), and the other was for the polymer (DABCO DC197, 0.2% of composite), supplied by Air Products and Chemical Inc. (Allentown, PA). DABCO T-120 (0.03% of resin), supplied by Air Products and Chemical Inc., was used as a catalyst. DABCO DC197 helps mixability of the two polymers and BYK-W 969 improves the ash loading and adhesion with polymer. Short glass fiber (M-300) was also added to improve toughness.

2.2. Processing

There are several ways the ash composites can be produced. The ash composite that we used in the laboratory is essentially produced by mixing dry fly ash with liquid polymer ingredients and additives. The difficulty of incorporating a high weight percent of ash was accomplished by using two-part liquid polyurethane, surfactants, and catalysts. Both ash and polymer were mixed in a high-speed mixer (KitchenAid, model KL26M1XSL, Macy’s, Greensboro, NC) and poured in a controlled volume rubber mold. The mold pressure required was between 25 to 65 psi (170–440 kPa), depending on the density requirement of the composite part. The two-part polyurethane reacts, generates heat, and cures itself. The whole process takes about 30–45 minutes, depending on the amount and type of curing agent used. The part was removed from the mold and postcured under ambient conditions for a week or in a tunnel furnace at 80°C for about 30 minutes. Steps of the processes are shown in Figure 3a. The rubber mold was changed for different parts, sizes, and shapes. Figure 3b shows the flat panel, decorative mold, and composite blocks fabricated by the current process. Flat plates were made for mechanical testing by using a laboratory-sized, high-speed electric mixer with a high shear blade. The ash weight was varied from 60% to 75% of the composite. The ambient moisture content (0.5%) in the ash was used to control the foaming and density of the composite. A U.S. patent was filed on this technology (Shivakumar and Brown, 2017).

The process is versatile and can be adapted into a broad range of products with relatively minor changes to the formulation. For example, to produce very light-weight parts, water is added to the formulation, and the water reacts and produces carbon dioxide that foams the material and can greatly reduce the density. Heavy parts such as the ash storage blocks, with 90 lb/ft³ (1.50 g/cc) density, or flame-resistant lightweight foam, with 1 lb/ft³ (0.015 g/cc) density, can be produced, both with high ash loadings. For increased toughness, strength, and stiffness, chopped glass or other fibers can be added. Similarly, mass colorants and weathering stabilizers can be mixed in the formulation or can be added after the part is made by using spray or roller-applied paint.

2.3. Leach test and analysis

Two types of leaching tests were performed: the US EPA M1313 tumbling test (Leaching Environmental Assessment Framework, 2018) for fly ash and an in-house-built continuous circulating water tank test that simulates river flow similar to US EPA M1315. The M1313 test was limited to distilled water (pH 7) because of quick validation of encapsulation concept and limited availability of resources. The test equipment used for US EPA M1313 is shown in Figure 4, and the water circulation test is shown in Figure 5. In both cases, the solid-to-distilled water ratio used was 1:10 by weight. The M1313 test was performed for 24–72 hours, depending on the size of the ash particle. The leachate samples (100 mL) were collected and analyzed by inductively coupled plasma (ICP) and inductively coupled plasma mass spectrometry (ICPMS) analyses at the Chemical Analysis Laboratory, North Carolina Department of Environment Quality (NC DEQ), and the results are expressed in parts per billion (or micrograms per liter). Similarly, leaching from coal ash powder and source 2 ash were tested for comparison. The distilled water was tested alone as a control.
The circulating water system consisted of a food-grade, high-density polyethylene tank that holds 25 L of water. A pump (Cobalt E-X-T 800, Cobalt Aquatics, Rock Hill, SC) with tube diameter of 15.6 mm (0.625 in.) and pumps 800 L/h was plumbed into the tank so that it collects water from the bottom of the tank and delivers it to the top. Two polypropylene (PP) grids with a thickness of 1.6 mm (0.063 in.), a hole diameter of 3 mm (0.125 in.), and an open area fraction of 40% were placed on a polyethylene stand so the sample is above the upper level of the bottom outlet tube. A fine grid PP sheet with a thickness of 4.8 mm (0.188 in.), hole diameter of 4.8 mm (0.188 in.), and open area fraction of 32% is placed under the sample so that fine ash particles do not fall to bottom of the tank. A sample (ash or composite piece) of about 500–2000 g is immersed in the distilled water of 10 times the weight of the sample (per US EPA guidelines), and the water is circulated continuously. A 100-mL water sample was collected and replaced with the same amount of fresh distilled water at the required interval. The sample was tested for leachates using ICP and ICPMS at the NC DEQ.

2.4. Mechanical properties

Three panels with different ash percentages (60%, 70%, and 75%) were prepared for mechanical characterization. All panels were produced with the same processing conditions but with different amounts of ash, and the density was controlled by the addition of water. The panel size was 203 × 203 × 12.5 mm (8 × 8 × 0.5 in.). The panels were tested after postcuring under ambient conditions for 1 week. Several ash composite blocks measuring 150 × 150 × 75 mm (6 × 6 × 3 in.) were made. One sample was tested for 14 months of leaching, the second sample for 12 months, and the third sample had the outer surface of the ash-composite block removed by hand sanding using 3M 426U, 180 grade sand paper to expose the inner surface of the block and was tested for 12 months. The last sample replicates the condition of a broken sample. Three types of mechanical tests were conducted: (1) Shore D hardness, (2) compression, and (3) flexure, according to ASTM D2240, ASTM D695, and ASTM D790, respectively (ASTM International 2015a, 2015b)}
The specimen configuration and test loading for each case are shown in Figure 6.

2.4.1. Hardness test

The surface hardness of the ash composites was conducted using type D Shore durometer (Instrument & Mfg. Co., Inc., Freeport, NY) according to ASTM D2240 (ASTM International, 2015a). Twenty-five equally spaced 5 × 5 grids were selected (Figure 6a), and then Shore D hardness was measured using the durometer. The average and standard deviation of each panel were calculated.

2.4.2. Compression test

Five circular cylindrical specimens (Figure 6b) with a 25-mm (1-in.) diameter and a 12.5-mm (0.5-in.) height were cut at selected locations of the panel, and then compression tested using an MTS universal test machine according to ASTM D695 (ASTM International, 2015a). Tests were performed in displacement control mode with a load cell capacity of 44.4 kN (10 kip) at room temperature. Crosshead speed of the test machine was 1.25 mm/min (0.05 in./min). The compression strength was calculated by dividing the maximum load by the cross-sectional area. The average strength and standard deviation for each ash percentage panel were calculated.

2.4.3. Flexure test

Flexure tests were performed according to ASTM D790 (ASTM International, 2017) by an MTS test machine using a three-point bend fixture. Tests were performed in displacement control mode with load cell capacity of 1.8 kN (400 lb) at room temperature. Crosshead speed of the test machine was 2.5 mm/min (0.10 in./min). Figure 7c shows the flexure specimen configuration and loading. The specimen size was 203 × 19 × 12.5 mm (8 × 0.75 × 0.5 in.). Three specimens were tested for each panel. Flexural strength was calculated from the maximum load \( P_{\text{max}} \) and based on the following equation:

\[
\sigma_f = \frac{3P_{\text{max}}S}{2wt^2}
\]

where \( \sigma_f \) is flexural strength (psi), \( P \) is maximum or failure load (lb), \( S \) is span (6.5 in.), \( w \) is width (0.75 in.) of the specimen, and \( t \) is thickness (0.50 in.) of the specimen.

3. Results

3.1. Leach test results

Table 1 compares the leachates from the two ashes (source 1 and source 2), coal powder (not ash) of source 1, and distilled water.

<table>
<thead>
<tr>
<th>Mineral</th>
<th>US EPA MCL</th>
<th>Distilled water</th>
<th>Coal powder (source 1)</th>
<th>Fly ash</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sb by ICP (μg/L)</td>
<td>6</td>
<td>10 U</td>
<td>10 U</td>
<td>37</td>
</tr>
<tr>
<td>As by ICP (μg/L)</td>
<td>10</td>
<td>2 U</td>
<td>3.4</td>
<td>160</td>
</tr>
<tr>
<td>B by ICP (μg/L)</td>
<td>7000</td>
<td>50 U</td>
<td>50 U</td>
<td>3800</td>
</tr>
<tr>
<td>Ba by ICP (μg/L)</td>
<td>2000</td>
<td>29</td>
<td>150</td>
<td>180</td>
</tr>
<tr>
<td>Be by ICP (μg/L)</td>
<td>4</td>
<td>5 U</td>
<td>5 U</td>
<td>5 U</td>
</tr>
<tr>
<td>Cd by ICP (μg/L)</td>
<td>5</td>
<td>0.5 U</td>
<td>0.5 U</td>
<td>1.3</td>
</tr>
<tr>
<td>Cr by ICP (μg/L)</td>
<td>100</td>
<td>5 U</td>
<td>5 U</td>
<td>49</td>
</tr>
<tr>
<td>Cu by ICP (μg/L)</td>
<td>1300</td>
<td>2 U</td>
<td>17</td>
<td>2 U</td>
</tr>
<tr>
<td>Hg245.1 (μg/L)</td>
<td>2</td>
<td>0.2 U</td>
<td>0.2 U</td>
<td>0.2 U</td>
</tr>
<tr>
<td>Mn by ICP (μg/L)</td>
<td>50</td>
<td>10 U</td>
<td>47</td>
<td>10 U</td>
</tr>
<tr>
<td>Mo ICP (μg/L)</td>
<td>200</td>
<td>10 U</td>
<td>10 U</td>
<td>300</td>
</tr>
<tr>
<td>Pb by ICP (μg/L)</td>
<td>15</td>
<td>2 U</td>
<td>4.8</td>
<td>2 U</td>
</tr>
<tr>
<td>Se by ICP (μg/L)</td>
<td>50</td>
<td>1 U</td>
<td>15</td>
<td>510</td>
</tr>
<tr>
<td>Ti by ICP (μg/L)</td>
<td>2</td>
<td>2 U</td>
<td>2 U</td>
<td>2 U</td>
</tr>
<tr>
<td>V by ICP (μg/L)</td>
<td>200</td>
<td>10 U</td>
<td>10 U</td>
<td>150</td>
</tr>
<tr>
<td>Zn by ICP (μg/L)</td>
<td>5000</td>
<td>10 U</td>
<td>14</td>
<td>10 U</td>
</tr>
</tbody>
</table>

1 US EPA MCL = U.S. Environmental Protection Agency’s maximum contaminant limit; ICPMS = inductively coupled plasma mass spectrometry; U = instrument limit; ICP = inductively coupled plasma.
2 All tests were conducted in distilled water.
3 Drinking water equivalent level (DWEL).

![Fig. 7. Variation of Shore D hardness with ash-composite density.](image-url)
used in the test. The US EPA MCL and drinking water equivalent level (DWEL) are also listed. Only the elements that the US EPA considers important from the health risk aspect are included here. As anticipated, Sb, As, and Se from source 1 and, in addition, V from source 2 exceeded the USEPA MCL. Also notice that these leachates from coal powder (again, not ash) are much less than the US EPA MCL. This indicates that the semi-bituminous coal, unburned, does not leach out hazardous inorganic minerals to water. Also note that, as expected, the distilled water used is contaminant free (the values are below the instrument limit).

Table 2 compares the leachates from the fly ash alone (source 1) and from the ash-composite block made from the same ash. The leachates were from 1-, 6-, and 14-month samples from the ash-composite block in the circulating water system. The leachate from the block is far less than the ash and does not reach the USEPAMCL. After 14 months, the maximum contaminant is less than one-third of the US EPA MCL and in most cases, the leachates were undetectable (below the instrument limit “U”). In some cases, leaching decreased with time, e.g., Zn, and some cases it increased, e.g., B. However, unexpectedly, Mn, Cu, and Zn showed up in the composite block, whereas they were undetectable in the ash. These elements may be from the metallic parts of the pump. This leaching test demonstrates that the ash composite is a safe way of storing the fly ash.

Table 3 compares 1- and 12-month leachates from the same fly ash.
ash-composite blocks (source 1), one with the surface as molded and the other with the outer surface machined off. The leachate results were similar for both blocks, and all identified hazardous minerals at even 12 months were much below the US EPA MCL in the circulating water system. The above-mentioned leachate results of fly ash composite clearly demonstrate that the fly ash composite is safe even if its outer surface is removed.

3.2. Mechanical test results

Results of hardness, compression, and flexure tests for three different ash loadings (60%, 70%, and 75%) are summarized (Figures 7–9). The three ash loadings of 60%, 70%, and 75% yielded densities of 29 lb/ft³ (0.46 g/cc), 41 lb/ft³ (0.66 g/cc), and 52 lb/ft³ (0.84 g/cc), respectively. Because the density is the primary controlling parameter of the composite, all results are assessed with respect to composite density. The ash content is also shown on the plots.

3.2.1. Hardness test results

Figure 7 shows the variation of surface hardness with density of the ash composites. The hardness generally increases with the density of the composites and the ash loading. The circle, square, and diamond represent the average hardness values, and the bar represents the standard deviation. The Shore D hardness values for 60%, 70%, and 75% ash were 38.4, 53.3 and 60.1, respectively.

3.2.2. Compression test results

Figure 8 shows the variation of compression strength with ash-composite density. Similar to the hardness, compression strength also increased with the density of the composites. The composite compression strength for 60%, 70%, and 75% was 4.5 MPa (651 psi), 5.9 MPa (851 psi), and 11.0 MPa (1592 psi), respectively.

3.2.3. Flexure test results

Figure 9 shows the variation of flexural strength with density of ash composites. Similar to hardness and compression strength, the

![Figure 8](image1.png)

**Fig. 8.** Variation of compression strength with ash-composite density.

![Figure 9](image2.png)

**Fig. 9.** Variation of flexural strength with ash-composite density.
The flexural strength also increased with the density of the composites. The flexural strength for 60%, 70%, and 75% was 4.7 MPa (679 psi), 5.8 MPa (834 psi), and 9.0 MPa (1317 psi), respectively. As noted in many text books (from Ashelby and Gibson), the material properties can also be controlled by the molding pressure. Higher pressure results in higher density and the associated higher strength values. This trend was noticed in our study. Details of the mechanical properties will be presented in future publications.

### 3.3. Ash-composite products

This section addresses the objective of demonstrating higher value-added products using ash composites. Figure 10 shows the different products produced in the laboratory. They include building products (Figure 10a) such as a chair mold, a base board, and a decorative mold. All products were made by 70% fly ash (source 1) by weight and demonstrated that complex shapes can be manufactured by a continuous or a batch process. Figure 10b shows three types of ash-composite foams with 0%, 30%, and 50% ash (from left to right). These products can be used for insulation applications. The density in all three foams is less than 6 lb/ft³ (0.1 g/cc). The fire resistance of ash-filled foam was found to be much better than that of no-ash foam. Figure 10c is a demonstration of storage block with ash content 75% by weight. The block has an interlocking design and a lifting mechanism to demonstrate that large-size blocks can be made to stack, if ash has to be safely stored and reused later. These blocks can also be used for flood protection, sea barriers, and many more applications where inert, low-cost, and heavy blocks are needed. Other attributes of ash composites are their electrical and thermal insulation properties combined with fire and termite resistance. These properties offer opportunities for several electric utility products such as crossarms, ground or buried utility poles, and insulators. Figure 10d shows a demonstration of 0.9-m-long, 102 × 102-mm (3-ft-long, 4 × 4-in.) crossarm with commercial insulators installed. In addition, ash composites could be used in products for buildings (e.g., siding, roof tiles, wall boards, fire doors) and infrastructures (e.g., railroad ties, Jersey barriers, highway sound barriers). Extensive product development, validation, and certification are required to bring this technology to fruition and make ash waste a valuable resource for power utility companies. The products shown in Figure 10 are for demonstration and their application needs to be validated.

The processing versatility and manufacturing adaptability are demonstrated in Figure 11: a sign board measuring 1500 × 450 × 38 mm (60 × 18 × 1.5 in.); a sandwich composite utility box cover, 300 × 300 × 25 mm (12 × 12 × 1 in.); and two composite posts, 2250 × 100 × 100 mm (90 × 4 × 4 in.). The board and two posts were combined and made a sign for a local elementary school. The utility box cover design load was 6803 kg (15,000 lb) and the maximum load was 10,205 kg (22,500 lb). The test results surpassed the maximum load, and the cover was less than one-half the weight of the cover that was by polymer concrete.

There seems to be no limit to where this technology can be used, due to its advantages: low cost (due to high coal ash loadings), insect and fire resistance, and a choice of densities, strengths, and stiffnesses. In addition, leaching of inorganic contaminants into water is greatly reduced, so these ash composites are safe. This seems an excellent starting point for developing many new products.

### 4. Conclusions

This work demonstrated that ash composites made from polyurethane and fly ash are able to reduce long-term leaching of coal ash inorganics to less than one-third of the US EPA MCL, even after being exposed to an accelerated water circulation.
The authors acknowledge the support of North Carolina senators Trudy Wade, Andrew Brock, and Bill Cook for initiating this research; the North Carolina Department of Environmental Quality (Cynthia Moseley) for funding the research; and Roy Byrd and team at the Division of Water Resources, Chemistry Laboratory (NC DEQ) for conducting the leachate analyses.

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