# Evaluation of the Mechanical and Environmental Performance of Biofuel Co-Product Stabilized Unpaved Roads

Final Report August 2018

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#### 16. Abstract

More than 50% of roadways in Iowa are classified as unpaved. The performance and long-term sustainability of such roads are dependent on the quality of the surfacing material, which varies considerably by location. The large, unbound particles form an unstable road surface that becomes rough, developing potholes and corrugations as the "floating" material is scattered by vehicles or washed away by rain. As a result, such roads require more frequent maintenance and reconstruction, which becomes very expensive for Iowa counties. Therefore, it is important to construct unpaved roads with materials that can sustain their performance for a considerable amount of time with less maintenance. This problem can be addressed economically in locations that are close to sources of considerable amounts of biofuel co-products (BCPs).

Loess soil was mixed with four different biofuel co-products: lignosulfonate, glycerin bottoms, crude glycerine, and glycerin 95. The soil was mixed with 4, 8, 12, and 16% BCP by weight. Results of the study showed that lignosulfonate improved the unconfined compressive strength (UCS) of the loess soil to some extent, while such trends were not observed for the mixtures prepared with glycerin products. Leaching tests focused on the pH and leaching of metals such as Cr, Al, Fe, As, and Zn from soil mixtures. The addition of the BCP did not influence the pH of the loess soil, and none of the mixtures leached metals that were above the detection limit of the equipment. These results indicate that BCPs do not pose any environmental threat when used as a dust control or stabilizing agent in unpaved roads.

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## EVALUATION OF THE MECHANICAL AND ENVIRONMENTAL PERFORMANCE OF BIOFUEL CO-PRODUCT STABILIZED UNPAVED ROADS

#### Final Report August 2018

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#### **EXECUTIVE SUMMARY**

Generation of fugitive dust is one of the major problems related to unpaved roads, and it affects road safety, health and living standards, and vegetation. But there are mechanical and chemical methods (e.g., filters and dust suppressants) to control this problem. Several waste materials such as glycerin and lignosulfonate have been used as dust suppressants in field applications. Glycerin is a co-product of biodiesel production with a low economic value, and lignosulfonate is a waste product from the paper-making industry. These waste materials are stored in tanks; hence, creating an area to reuse these materials provides several benefits such as minimizing landfill requirements and waste disposal costs. On the other hand, dust suppressants should not affect the engineering properties of soil in a negative way. But any negative effect from dust suppressants on unpaved roads could cause road failure, loss of life, and property damage due to accidents.

In this study, a loess soil, which is locally available in Iowa, was mixed with three types of glycerin (glycerin bottoms, crude glycerin, and glycerin 95) of varying content levels and a lignosulfonate material to investigate the effect of dust suppressants on the strength of loess soil. The loess soil was also mixed with two types of corn oil (an ethanol co-product) obtained from different sources to investigate its effects, since several oil-type materials such as soybean oil have been used as dust suppressants in the past.

Unconfined compression (UC) tests were performed on 1-day and 7-day cured 2 by 2 in. specimens to observe whether the stated materials affected the strength of the loess soil considerably. It was observed from the UC test results that the lignosulfonate improved the unconfined compressive strength (UCS) of the loess soil to some extent because of the natural binding property of lignin present in the lignosulfonate and dispersion of the clay fraction due to the presence of lignin. Since the lignosulfonate used in this study was in a liquid form, it was concluded that an optimum amount of lignosulfonate should be used to prevent an excessive amount of liquid in the soil matrix. Although glycerin has been used as a dust suppressant to minimize the dust problem of unpaved roads, it was observed that using glycerin bottoms, crude glycerin, or glycerin 95 decreased the UCS of the loess soil regardless of glycerin content. Similar to the trend observed with using the glycerin materials, it was observed that using corn oil decreased the UCS of the loess soil. Crack formations were observed in all specimens containing a relatively higher amount of each material due to a reduction in the bonding between the loess soil particles. Additionally, all specimens turned into mud if excessive amounts of the materials were used.

#### INTRODUCTION

Total public road length is around 4.1 million miles (including 1.4 million miles of unpaved road) in the US (FHWA 2011). Billions of dollars are spent keeping these roads in good and serviceable condition. Thus, having an efficient and long-lasting maintenance plan is a priority at the national level. Several waste materials such as construction debris, biofuel co-products (BCPs), and highway paving materials have been used as alternatives to conventional materials in unpaved road construction. These waste materials are often thrown in landfills or stored in tanks. Hence, creating an area to reuse these materials provides several benefits: protecting the natural material resources, minimizing landfill requirements and waste disposal costs, and obtaining an added value from the waste materials. In addition, it also helps the economic growth of the nation in the long term (Cetin et al. 2010).

In general, locally available soils are used for unpaved road construction to reduce construction costs. However, saving money this way may lead to road failures such as potholes, cracks, and permanent deformation because the locally available soils may not always be suitable for construction. There is a variety of techniques to improve the performance of roadways such as excavation and replacement and physical (e.g., compaction) and chemical (e.g., using calciumbased stabilizers such as lime and fly ash) soil stabilization.

One of the major problems that is commonly seen in unpaved roads is the generation of fugitive dust (Yan and Hoekman 2012). This generation of dust may affect road safety by reducing the visibility distance for drivers and pedestrians and by causing a loss of surface material, which may cause ruts and potholes (Addo et al. 2004). It also may affect the health and living standards of people located in surrounding areas by causing air pollution. In addition, dust may damage vegetation by clogging the pores of plants and covering their surfaces (the shading effect) (Addo et al. 2004). Mechanical and chemical methods are the two main dust suppression methods (Yan and Hoekman 2012). Several waste materials such as glycerin (Yan and Hoekman 2012) and lignin derivatives (Addo et al. 2004) have been used as dust suppressants in field applications.

The lignin derivative most commonly used as a dust suppressant is lignosulfonate (sulfite lignin or lignin-sulfonates), which is a waste material from the paper-making industry (Gopalakrishnan et al. 2013). The lignin compound is a natural binder that holds the fiber of wood together (Addo et al. 2004). Not only can it reduce fugitive dust, but an increase in the strength of unpaved road surfaces can be observed with the use of lignosulfonate because of its binding properties (Nicholls and Davidson 1958, Addo et al. 2004). In addition, it was reported by Nicholls and Davidson (1958) that higher strength values can be observed with extended air curing. The presence of lignin material in the clay soil matrix leads to a dispersion of the clay fraction (Gow et al. 1961, Davidson and Handy 1960). The dispersion of the clay fraction reduces the void ratio, which leads to an improvement in particle packing (Gow et al. 1961).

Glycerin is a co-product of biodiesel production and has a low economic value. Crude glycerin was used in a study performed by Kinast (2003), and it was determined that crude glycerin provides effective dust control. The effectiveness of the glycerin application can last longer than

the water spray method. In addition, glycerin can be preferable over other dust suppressants in cold regions due to its lower freezing point (Yan and Hoekman 2012).

While reducing the dust problem is important for unpaved roads, dust suppressants should not affect the engineering properties of soils in a negative way. Any negative effect of the dust suppressants on unpaved roads may cause road failures, loss of life, and property damage due to accidents. In this study, a loess soil was mixed with glycerin and lignosulfonate materials to investigate the effect of dust suppressants on the strength of loess soil. In addition, the effect of using corn oil, an ethanol co-product, on the strength of the loess soil was also investigated since several oil-type materials such as soybean oil have been used as dust suppressants in the past (Gillies et al. 1999). Unconfined compression (UC) tests were performed to observe whether the stated materials affected the strength of the soil considerably.

#### **MATERIALS**

The loess soil was collected from the Loess Hills in western Iowa. It was air-dried to obtain a water content in the range of 1.5 to 2%. Any visible foreign and organic materials were removed physically. The soil was sieved and any particles smaller than 3/4 in. were removed. Sieve and hydrometer analyses (ASTM 2007) and an Atterberg limits test (BSI 1990, ASTM 2010) were performed. It was determined that the soil contained 0% gravel, 1% sand, 87% silt, and 12% clay-sized particles (see Figure 1).

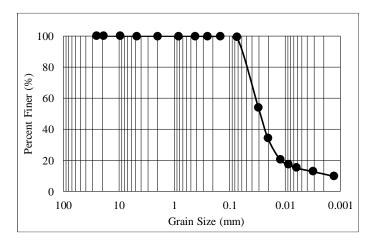


Figure 1. Grain size distribution of the loess soil

In addition, the soil's liquid limit (LL) and plastic limit (PL) were determined to be 37.3 and 26.9, respectively. Thus, it was classified as having low plastic silt (ML) according to the Unified Soil Classification System (USCS) (ASTM 2011) and A-4 soil according to the American Association of State Highway and Transportation Officials (AASHTO) Soil Classification System (AASHTO 2004).

The lignosulfonate was obtained from Blue Flame Propane in Letts, Iowa, and three different glycerin materials (glycerin bottoms, crude glycerin, and glycerin 95) were obtained from the Renewable Energy Group in Ames, Iowa. The chemical contents of the glycerin materials are shown in Table 1.

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Property	Limit	<b>Glycerin Bottoms</b>	Crude Glycerin	Glycerin 95
<b>Glycerin Content</b>	% wt, min	40	78	95
Moisture	% wt, max	0.5	13	2
Methanol	% wt, max	0.3	0.3	0.1
Ash	% wt, max	30	7	0.3
<b>Total Fatty Acid</b>	% wt, max	15	1	1
pН	Range	_	4.0–7.5	4.0–7.5

It was determined that glycerin bottoms contained the lowest glycerin content (at 40%) compared to the other two glycerin materials. The glycerin contents of the crude glycerin and glycerin 95 were determined as 78% and 95%, respectively. Two different corn oil materials were obtained from Absolute Energy in St. Ansgar, Iowa, and POET in Sioux Falls, South Dakota.

#### **METHODS**

#### **Standard Proctor Test**

A standard proctor test was conducted in accordance with ASTM D698 Method A (ASTM 2012) to obtain a relationship between the moisture content and the dry unit weight of the compacted loess soil. An automatic proctor device was used for the tests (see Figure 2).



Figure 2. Automatic proctor device

Target moisture content values (8, 12, 14, 16, and 18%) were determined and a sufficient amount of water was added to the air-dried loess soil (moisture content in the range of 1.5 to 2%) to reach each target moisture content value. After completing the tests, the maximum dry unit weight and the optimum moisture content (OMC) values of the loess soil were obtained from the compaction curve.

#### **Specimen Preparation**

The dust suppressants were mixed with the loess soil at different percentages (4, 8, and 12% by weight) (see Figure 3). In addition, loess only specimens were also prepared.



Figure 3. Mixing the loess soil with lignosulfonate

The  $2\times2$  in. cylindrical specimens were prepared at the OMC of the loess soil for the UC tests in accordance with the method described by O'Flaherty et al. (1963). For the loess soil/lignosulfonate mixtures, specimens that contained moisture contents of 4% (OMC–4%) and 8% (OMC–8%) lower than the OMC of the loess soil were also prepared. A five pound hammer was dropped from a 12 in. height for the compaction (see Figure 4).

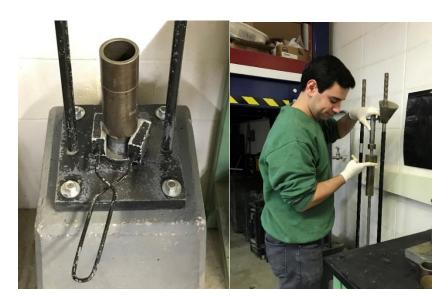


Figure 4. Compaction apparatus for the  $2\times2$  in. specimens

In total, seven blows were applied to compact the specimens. After compaction, the specimens were extracted from the mold with a hydraulic jack (see Figure 5).



Figure 5. Hydraulic jack for extracting the specimens

The prepared 2×2 in. specimens are shown in Figure 6.



Figure 6. Extracted 2×2 in. specimens

As the last step, the specimens were wrapped in a plastic film and then in aluminum foil to keep their moisture contents constant (see Figure 7). The specimens were cured for 1 day and 7 days at room temperature prior to UC testing.



Figure 7. Sealed test specimens in plastic film (left) and aluminum foil (right)

#### **UCS Test**

The UC test is a special case of an unconsolidated undrained (UU) triaxial test where the confining pressure is zero relative to atmospheric pressure. It is used to determine the unconfined compressive strength of soils. The UC tests were conducted in accordance with ASTM D2166 (ASTM 2016). A GeoJAC Digital Load Actuator and Sigma-1 computer program were used for the tests (see Figure 8).

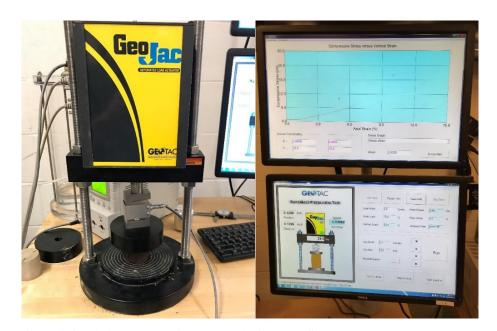


Figure 8. GeoJAC Digital Load Actuator (left) and Sigma-1 computer program (right)

The tests were conducted on the 1-day and 7-day cured specimens with a strain rate of 2%/min. (see Figure 9).

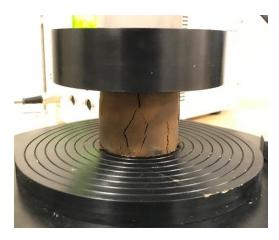


Figure 9. Failure of a specimen during the UC test

When a peak loading with a corresponding axial strain value between 0 to 15% was observed, the peak loading was recorded as the UCS of the specimen. When a continuous increase in the loading was observed, the tests were ended when the deformation of the specimen reached an axial strain of 15%. The loading value corresponding to the 15% axial strain was recorded as the UCS of the specimen.

#### Water Leach Test (WLT)

Batch water leach tests were conducted on the soil and soil-BCP mixtures in accordance with ASTM D3987. A constant liquid-to-solid (L/S) ratio of 20:1 was used for all materials. The airdried soil was crushed and sieved using a U.S. No. 4 sieve (4.75 mm), and the soil was mixed homogeneously with BCPs at different percentages. Each specimen was cured for 7 days in plastic bags in a moisture-controlled humidity chamber (21°C and 100% relative humidity). After curing, 2.4 g of soil mixture was added to a 50 mL plastic centrifuge tube followed by 48 mL leachant (i.e., the 0.1 M NaBr solution). The soil mixtures were rotated continuously on a rotator at 29 revolutions per minute at room temperature (~22°C) for 18 hours for equilibration. After reaching an equilibrium, the specimens were settled for 5 minutes and placed in a Beckman GPR centrifuge machine. The mixtures were centrifuged at 3,000 rpm for 20 minutes. Next, the suspended solids were filtered through the 0.2  $\mu$ m pore size, 25 mm diameter membrane disk filters fitted in a 25 mm easy pressure syringe filter holder by using a 60 mL plastic syringe. The filtered samples were subjected to pH measurements and then acidified to pH < 2 using high-purity nitric acid and stored in 15 mL high density polyethylene centrifuge tubes at 4°C. Triplicate WLTs were conducted on all fly ashes, soils, and soil mixtures.

#### **Toxicity Characteristic Leaching Procedure (TCLP)**

The soils and their BCP mixtures prepared for the TCLP tests were the same ones prepared for the WLTs. Duplicate tests were conducted on each specimen. EPA Method 1311 was followed during the TCLP tests. The soil mixtures were sieved through an U.S. No. 9.5 mm sieve. A L/S ratio of 20:1 was used for all test specimens. An acetic acid solution with a pH of 5 was used as an extraction fluid and was added only once at the start of the extraction. The pH and electrical

conductivity measurements were recorded immediately after sample collection. The protocol for sample preparation and preservation followed those employed in the WLTs, except for the filtration procedure. The samples were vacuum filtered through TCLP glass fiber filters, and the filtered leachates were acidified to pH < 2 with a 2% HNO<sub>3</sub> acid solution and preserved in 4°C for chemical analysis.

#### **Chemical Analysis**

The pH levels of the leachate samples collected from the WLTs and TCLP tests were determined following the methods outlined in ASTM D 1293. The pH of the fly ash was determined by using SW-846 Method 9045. Three replicate samples were measured for each sample and the mean values were reported. The metals selected for analysis were Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Sb, Si, Sr, TI, V, and Zn, based on total elemental analyses. The concentrations of all metals were determined by inductively coupled plasma optical emission spectroscopy (ICP-OES) using a Varian Vista-MPX CCD Simultaneous ICP-OES instrument. Minimum detection limits (MDLs) for the ICP-OES were determined for each metal and a set of calibration standards according to the U.S. Code of Federal Regulations Title 40. The MDLs for Al, Cr, Fe, Mn, Sb, and V were determined as 2.5, 0.5, 3.2, 0.05, 3, and 0.1 µg/L, respectively.

#### **TEST RESULTS**

#### **Standard Proctor Test Results**

The compaction curve of the loess soil is provided in Figure 10.

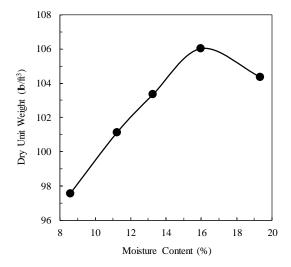


Figure 10. Compaction curve of the loess soil

It was determined that the optimum moisture content and maximum dry unit weight of the loess soil were 16.2% and 106.1 lb/ft<sup>3</sup>, respectively.

#### **UCS Test Results**

Effect of Lignosulfonate on the UCS of the Loess Soil

Summaries of the UC test results of the specimens prepared with the loess soil and lignosulfonate are provided in Table 2 and Figure 11.

Table 2. Effect of lignosulfonate on the UCS of the loess soil

<b>Moisture Content</b>	<b>Lignosulfonate Content (%)</b>	1-Day UCS (psi)	7-Day UCS (psi)
	0	21.64	22.13
OMC	4	25.18	29.98
OMC	8	17.69	21.80
	12	12.68	14.12
OMC – 4%	4	23.43	29.04
	8	26.01	31.03
	12	16.43	17.91
	4	18.46	24.98
OMC - 8%	8	17.55	27.06
	12	21.02	23.29
	16	17.36	17.78

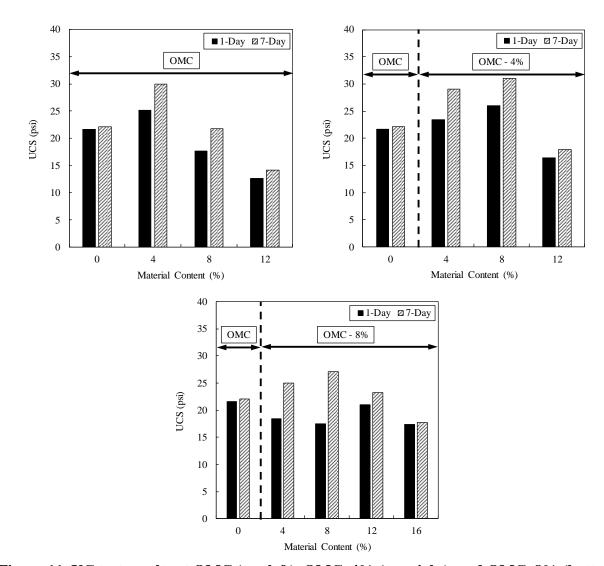


Figure 11. UC test results at OMC (top left), OMC-4% (top right), and OMC-8% (bottom)

As seen in the top left bar graph in Figure 11, the use of 4% lignosulfonate at the OMC of the loess soil increased the UCS of the loess soil around 16% and 39% after 1 day and 7 days of curing, respectively. A decreasing trend in the UCS of the loess soil was observed in the specimens containing more than 4% lignosulfonate (at the OMC of the loess soil).

As seen in the top right bar graph in Figure 11, it was observed that a 4% reduction in the OMC of the loess soil (OMC–4%) provided a further increase in the UCS of the loess soil in the cases where 4% and 8% lignosulfonate were used. However, the use of 12% lignosulfonate decreased the UCS of the loess soil.

As seen in the bottom bar graph in Figure 11, a further reduction in the OMC of the loess soil (OMC–8%) to prepare the specimens did not provide any further benefit compared to the results obtained in the specimens containing 4% less moisture content. It was concluded that the highest value of the UCS of the loess soil was observed in the specimen prepared with 8% lignosulfonate at the moisture content 4% less than the OMC of the loess soil (OMC–4%). The 2×2 in. cylindrical specimens containing 4% and 12% lignosulfonate (at the OMC of the loess soil) are shown in Figure 12.

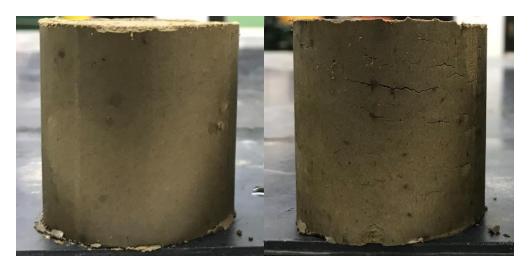


Figure 12. Specimens containing 4% lignosulfonate (left) and 12% lignosulfonate (right)

As seen here, cracks were observed in the specimens prepared with the highest amount of lignosulfonate due to a reduction in the bonding between soil particles.

Effect of Glycerin on the UCS of the Loess Soil

Summaries of the UC test results of the specimens prepared with the loess soil and glycerin materials (glycerin bottoms, crude glycerin, and glycerin 95) are provided in Table 3 and Figure 13.

Table 3. Effect of the glycerin materials on the UCS of the loess soil

Glycerin Type	<b>Glycerin Content (%)</b>	1-Day UCS (psi)	7-Day UCS (psi)
	0	21.64	22.13
Clysopin Pottoms	4	14.33	18.33
Glycerin Bottoms	8	11.38	12.66
	12	4.81	4.56
	0	21.64	22.13
Courde Clysospin	4	18.24	19.65
Crude Glycerin	8	11.23	12.83
	12	3.12	3.02
Classic 05	0	21.64	22.13
	4	13.36	15.62
Glycerin 95	8	8.63	9.51
	12	2.82	2.99

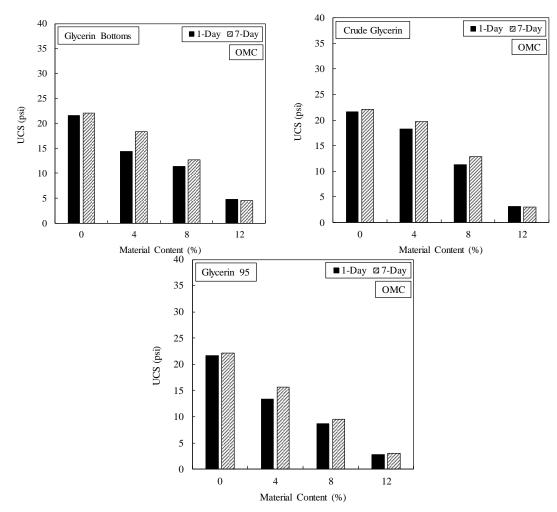


Figure 13. UC test results for glycerin bottoms (top left), crude glycerin (top right), and glycerin 95 (bottom)

As seen in Figure 13, a decreasing trend in the UCS of the loess soil was observed with an increase in the glycerin content of each glycerin type. In addition, as seen in Figure 14, cracks were observed in the specimens prepared with the highest amount of glycerin (valid for each glycerin type) due to a reduction in the bonding between soil particles.

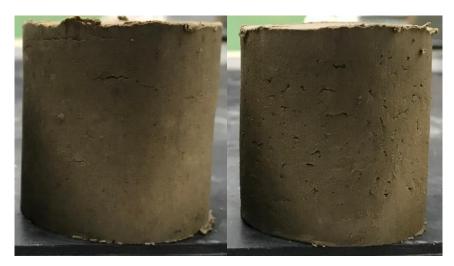


Figure 14. Specimens containing 4% crude glycerin (left) and 12% crude glycerin (right)

Effect of Corn Oil on the UCS of the Loess Soil

Summaries of the UC test results of the specimens prepared with the loess soil and the corn oil materials (from Absolute Energy and POET) are provided in Table 4 and Figure 15.

Table 4. Effect of the corn oil materials on the UCS of the loess soil

Corn Oil Type	Corn Oil Content (%)	1-Day UCS (psi)	7-Day UCS (psi)
Absolute Energy	0	21.64	22.13
	4	21.44	24.74
	8	12.76	15.20
	12	4.10	3.50
POET	0	21.64	22.13
	4	19.61	21.32
	8	11.91	13.11
	12	4.50	4.20

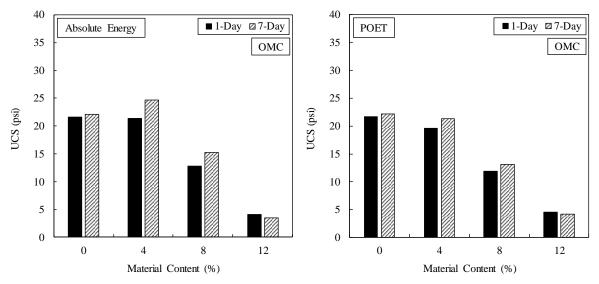


Figure 15. UC test results for Absolute Energy (left) and POET (right)

As seen in Figure 15, a decreasing trend was observed in the UCS of the loess soil with an increase in the corn oil content (valid for both corn oil materials). The only improvement in the UCS of the loess soil was observed in the specimen prepared with 4% corn oil (from Absolute Energy) and cured for 7 days. However, considering the general trend, this result was attributed to several possible errors that might have occurred during specimen preparation or during UC testing. In addition, as seen in Figure 16, cracks were observed in the specimens prepared with the highest amount of corn oil (valid for each corn oil type) due to a reduction in the bonding between soil particles.

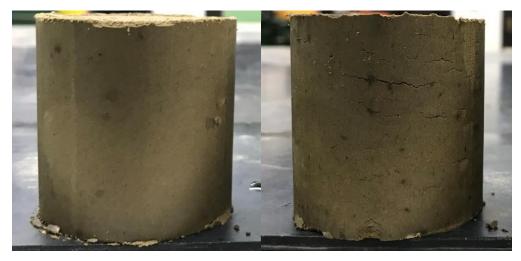


Figure 16. Specimens containing 4% corn oil (left) and 12% corn oil (right) from POET

Finally, as seen in Figure 17, the use of 16% corn oil (for each corn oil type) turned the soil into mud and the  $2\times2$  in. cylindrical specimens could not be prepared.



Figure 17. Specimens containing 16% corn oil from Absolute Energy

#### **Leaching Results from WLTs and TCLP Tests**

Both WLTs and TCLP test results showed that the addition of BCPs to the soils did not influence the leaching of metals from the loess soil. These metals included Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Sb, Si, Sr, TI, V, and Zn. These results indicated that BCPs do not cause any environmental concerns from a metal leaching perspective.

#### CONCLUSIONS

In this study, the use of lignosulfonate improved the UCS of the loess soil to some extent. Since the lignosulfonate material used in this study was in a liquid form, an optimum amount of lignosulfonate should be used to prevent an excessive amount of liquid in the soil matrix. In this study, the optimum lignosulfonate contents were determined to be 4% at the OMC of the loess soil and 8% at the moisture content, 4% less than the OMC of the loess soil (OMC–4%).

Although glycerin has been used as a dust suppressant to minimize the dust problem of unpaved roads, it was determined in this study that the use of glycerin bottoms, crude glycerin, and glycerin 95 decreased the UCS of the loess soil. And like glycerin, it was determined that the use of corn oil materials decreased UCS. Leaching studies showed that the BCPs did not leach any metal at any significant level that may pose a threat to the environment.

#### RECOMMENDATIONS

It was stated by Sinha et al. (1957) that the use of lignosulfonate may be more effective for granular soil types. Thus, not only lignosulfonate but also the glycerin and corn oil materials should be mixed with granular soils. In addition, several durability tests such as freeze-thaw (F-T) or wet-dry (W-D) tests should be performed to observe whether the materials used in this study improve the durability of the loess soil or other soil types.

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