# Biofuel Co-Product Uses for Pavement Geo-Materials Stabilization

**Final Report**
**April 2010**

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**Institute for Transportation**

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The preparation of this (report, document, etc.) was financed in part through funds provided by the Iowa Department of Transportation through its "Agreement for the Management of Research Conducted by Iowa State University for the Iowa Department of Transportation," and its amendments.

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## Technical Report Documentation Page

| | | |
|---|---|---|
| **1. Report No.**<br>IHRB Project TR-582 | **2. Government Accession No.** | **3. Recipient's Catalog No.** |

| | |
|---|---|
| **4. Title and Subtitle**<br>Biofuel Co-Product Uses for Pavement Geo-Materials Stabilization | **5. Report Date**<br>April 2010 |
| | **6. Performing Organization Code** |
| **7. Author(s)**<br>Kasthurirangan Gopalakrishnan, Halil Ceylan, and Sunghwan Kim | **8. Performing Organization Report No.**<br>InTrans Project 08-316 |
| **9. Performing Organization Name and Address**<br>Institute for Transportation<br>Iowa State University<br>2711 South Loop Drive, Suite 4700<br>Ames, IA 50010-8664 | **10. Work Unit No. (TRAIS)** |
| | **11. Contract or Grant No.** |
| **12. Sponsoring Organization Name and Address**<br>Iowa Highway Research Board<br>Iowa Department of Transportation<br>800 Lincoln Way<br>Ames, IA 50010 | **13. Type of Report and Period Covered**<br>Final Report |
| | **14. Sponsoring Agency Code** |

**15. Supplementary Notes**

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**16. Abstract**

The production and use of biofuels has increased in the present context of sustainable development. Biofuel production from plant biomass produces not only biofuel or ethanol but also co-products containing lignin, modified lignin, and lignin derivatives. This research investigated the utilization of lignin-containing biofuel co-products (BCPs) in pavement soil stabilization as a new application area. Laboratory tests were conducted to evaluate the performance and the moisture susceptibility of two types of BCP-treated soil samples compared to the performance of untreated and traditional stabilizer-treated (fly ash) soil samples. The two types of BCPs investigated were (1) a liquid type with higher lignin content (co-product A) and (b) a powder type with lower lignin content (co-product B). Various additive combinations (co-product A and fly ash, co-products A and B, etc.) were also evaluated as alternatives to stand-alone co-products. Test results indicate that BCPs are effective in stabilizing the Iowa Class 10 soil classified as CL or A-6(8) and have excellent resistance to moisture degradation. Strengths and moisture resistance in comparison to traditional additives (fly ash) could be obtained through the use of combined additives (co-product A + fly ash; co-product A + co-product B).

Utilizing BCPs as a soil stabilizer appears to be one of the many viable answers to the profitability of the bio-based products and the bioenergy business. Future research is needed to evaluate the freeze-thaw durability and for resilient modulus characterization of BCP-modified layers for a variety of pavement subgrade and base soil types. In addition, the long-term performance of these BCPs should be evaluated under actual field conditions and traffic loadings. Innovative uses of BCP in pavement-related applications could not only provide additional revenue streams to improve the economics of biorefineries, but could also serve to establish green road infrastructures.

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|---|---|
| **17. Key Words**<br>biofuel co-product—pavement—soil stabilization—sustainability | **18. Distribution Statement**<br>No restrictions. |

| | | | |
|---|---|---|---|
| **19. Security Classification (of this report)**<br>Unclassified. | **20. Security Classification (of this page)**<br>Unclassified. | **21. No. of Pages**<br>52 | **22. Price**<br>NA |

**Form DOT F 1700.7 (8-72)** **Reproduction of completed page authorized**

# BIOFUEL CO-PRODUCT USES FOR PAVEMENT GEO-MATERIALS STABILIZATION

**Final Report**
**April 2010**

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Sponsored by
the Iowa Highway Research Board
(IHRB Project TR-582)

Preparation of this report was financed in part
through funds provided by the Iowa Department of Transportation
through its research management agreement with the
Institute for Transportation,
InTrans Project 08-316.

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# TABLE OF CONTENTS

# LIST OF FIGURES

# LIST OF TABLES

# ACKNOWLEDGMENTS

The authors gratefully acknowledge the Iowa Highway Research Board (IHRB), Grow Iowa Values Fund (GIVF) seed grant program, Iowa State University (ISU), and ISU's Bioeconomy Institute for financial supporting this study. The authors are also grateful to Dynamotive Energy Systems (Canada) and Grain Processing Corporation (GPC) of Muscatine, Iowa, for supplying the experimental bio-oil and ethanol by product materials investigated in this study. The Chief Operating Officer of Dynamotive Energy Systems, Mr. Thomas Bouchard's assistance towards this project is greatly appreciated. The authors would also like to acknowledge the special administrative support of the Institute for Transportation (InTrans) at Iowa State University throughout the entire project.

The authors are grateful to Prof. Robert C. Brown and Ms. Jill E. Euken from ISU's Bioeconomy Institute and Prof. Hans van Leeuwen of CCEE for their thoughtful discussions and input. The invaluable guidance and input provided by the Technical Advisory Committee (TAC) members Ms. Melissa A. Serio, Messrs. Edward J. Engle, Stephen J. Megivern, Robert L. Stanley, Mark J. Dunn, and Dean F. Herbst of Iowa Department of Transportation (Iowa DOT), the Buchanan County Engineer Mr. Brian Keierleber, and Ms. Lisa Rold from the Federal Highway Administration (FHWA) Ames, Iowa office throughout this project is also greatly appreciated.

The authors would like to thank the Research Engineer Mr. Robert F. Steffes of the National Concrete Pavement Technology Center (CP Tech Center) for obtaining the soil samples from the field project sites and the Geotechnical Engineering Laboratory Technician Mr. Donald Davidson for helping the operation of laboratory equipments. The authors also appreciate the undergraduate hourly students from the Civil, Construction and Environmental Engineering (CCEE) including Ms. Danielle Abbott, Messrs. Dong-Hyun Chung, Forrest Hasty, Shiyun (Simon) Wang, and Teng (Alex) Wang for their assistance to conduct the portions of the laboratory tests. The project would not have been completed smoothly without all of the above-mentioned support and help.

# EXECUTIVE SUMMARY

Sustainable use of biomass as a renewable source to produce energy could be an alternative solution to the problems of the cost of fossil-based energy and global warming. The production and use of biofuels as renewable energy has increased. Biofuels produced from plant biomass creates not only biofuels or ethanol, but also co-products that contain lignin, modified lignin, and lignin derivatives. The use of lignin in soil stabilization has been studied over the past decades. However, most of the previous lignin-related soil stabilization studies investigated sulfite lignins (lignosulfonates) derived from the paper industry, while the lignins obtained from biofuel or ethanol production are sulfur-free. Newer uses of biomass-derived lignin need to be developed to provide additional revenue streams to improve the economics of the bio-based products and the bioenergy business. The present study aims to investigate the innovative utilization of biofuel co-products (BCPs) containing sulfur-free lignin in pavement soil stabilization.

Laboratory tests were conducted to evaluate the strength performance and the moisture susceptibility of two types of BCP-treated soil samples to compare to the performance of untreated and traditional stabilizer-treated (fly ash) soil samples. Two types of BCPs investigated were (1) a liquid type BCP with higher lignin content (co-product A) and (2) a powder type BCP with lower lignin content (co-product B). Various additive combinations (co-product A + fly ash, co-products A + B, etc.) were also evaluated as alternatives to stand-alone additives. The unconfined compression strength (UCS) experimental test was used to evaluate strength performance. Atterberg limits and standard Proctor compaction tests were also conducted as engineering properties tests. Additive and moisture contents and curing periods were incorporated as variables into the strength property test factorial. The experimental test program for evaluating moisture susceptibility consisted of UCS tests after “dry” and “wet” conditioning as well as visual observations of soaked specimens (so-called soaking tests). The UCS tests were conducted on both dry and wet specimens to evaluate the strength loss due to moisture intrusion. Each specimen was also fully soaked in water over a period of time to examine how long it could withstand moisture damage and whether it would fail due to moisture effects.

Performance test results indicate that BCPs are effective in stabilizing the Iowa Class 10 soil classified as CL or A-6(8). Moisture susceptibility test results indicate that the BCPs also have excellent resistance to moisture degradation. Co-product A with higher lignin content is more effective at providing resistance to moisture damage than co-product B with lower lignin content and traditional additive (fly ash). The use of combined additives (co-product A + fly ash, co-products A + B) could be a promising alternative to the use of co-product A alone to obtain strengths and moisture resistance comparable to traditional additive (fly ash).

Utilizing BCPs as a soil stabilizer appears to be a viable use of these bio-based products. Because much more BCP is disposed of rather than utilized, making more productive use of BCPs would have considerable benefits for sustainable development. BCPs used in this experiment demonstrated excellent potential for stabilizing low-quality materials for use in pavement foundation systems. These products could be used to stabilize existing subgrade materials to provide a stable working platform and to improve the strength of undesirable soil materials for use as the load-bearing layer within the pavement system. From an economic perspective, the change to renewable energy from fossil-based energy could result in less

production and higher costs of fly ash, which is a byproduct from coal-fired power plants. However, this change could also result in higher production and lower costs of BCPs, making them comparable to traditional soil stabilizers. While fly ash has the potential to leach heavy metals that would result in soil contamination (FHWA 2005), BCPs could be beneficially used without adversely impacting the environment because the feedstock of biofuels and co-products is natural biomass and they are considered to be biodegradable. However, this needs to be further researched.

Future research is needed to evaluate the freeze-thaw durability and for resilient modulus characterization of BCP-modified soils. In addition, the long-term performance of these BCPs should be evaluated under actual field conditions and traffic loadings. Because only one soil type was investigated in this study, any future work should investigate the use of lignin-based BCPs for a variety of soils that are being used as pavement subgrade and base layers in highway infrastructure systems. Considering the wide range of pavement-related applications in which modified lignins have already been used, such as concrete admixtures, dust suppressants, and potentially pavement base layer treatment agents and joint and crack sealants, the utilization of BCPs containing sulfur-free lignin in these applications should be investigated in the future. Newer uses of BCPs in pavement-related applications could not only provide additional revenue streams to improve the economics of biorefineries, but could also serve to establish green road infrastructures.

# INTRODUCTION

## Background

Bio-based energy production derived from biomass is often advocated as a significant contributor to possible solutions to our need for a sustainable transportation fuel. The burning of fossil fuels is a major contributor to atmospheric carbon dioxide (IPCC 1996). As an alternative, the United States Department of Energy (U.S. DOE) and private enterprises are developing a fermentation process for producing ethanol from high-cellulose biomass (Dipardo 2000; Hettenhaus et al. 2000). Corn stover and other plant materials with a high concentration of cellulose have potential to be used for biofuels (e.g., ethanol production). Using biofuels may partially offset energy requirements currently fulfilled by fossil fuels (Paustian et al. 1998). A complete life-cycle analysis of this process includes comparing possible economical and environmentally sound uses for the byproduct remaining after the fermentation of corn stover, such as production of electricity or use as a soil amendment (Johnson et al. 2004).

The byproduct remaining after fermentation of corn stover contains 600 to 700 g lignin $kg^{-1}$ and 20 g N $kg^{-1}$ (NREL 2002). “Stover” refers to the plant parts remaining in the field after harvesting corn. Biofuels are made from corn residue (stalks and leaves) in a process similar to the production of biofuels from corn grain. The corn stover biofuels byproduct has three times the concentration of nitrogen as the original cornstalks. It consists of stalk parts too tough to be digested by alcohol fermentation microbes and has a compost-like consistency. After using stover for biofuels production, the remaining byproduct of fermentation has up to 60% to 70% lignin. There have been continued research efforts focusing on the development of market opportunities for lignin, modified lignin, and lignin derivatives.

Traditional uses for lignin and modified lignin include concrete admixtures, binders, well drilling mud, dust control, vanillin production, and dispersants (Gargulak and Lebo 1999; Sundstrom et al. 1983). However, the amount of lignin that will become available from biorefineries will easily saturate these markets, and new uses for lignin need to be developed to provide additional revenue streams to improve the economics of biorefineries (Gopalakrishnan et al. 2010). Lignin has been studied as an extender in asphalt to help reduce the use of petroleum and has been found to have no adverse effects on performance (Khandal 1992; Sundstrom et al. 1983). Recent research studies are also focusing on evaluating the lignin derived from agricultural co-products as an antioxidant in asphalt. The addition of lignin as a potential antioxidant for asphalt is anticipated to increase the service life of our nation’s highways (Guffey et al. 2005; Williams and McCready 2008).

Lignin has also been implicated as having a role in soil stabilization (Nicholls and Davidson 1958; Kozan 1955; Johnson 2003). The impact of lignin could be direct, or lignin may contribute to the formation of humic acid, which increases soil stability. It has been hypothesized that since the biofuel co-product (BCP) is high in lignin, which is thought to play a role in stabilizing soil, incorporation of the co-product into soil may help maintain or improve soil structure and stability (Gantzer et al. 1987; Johnson et al. 2004). Johnson et al. (2004) conducted the first study to evaluate the impact of corn stover–derived BCP on biological, chemical, and physical properties of soils.

Natural soils rarely possess the necessary engineering properties for road construction. Thus, adding chemicals to soil to improve the road soil properties, termed "soil stabilization," has become a common practice in constructing both paved and unpaved roads. For unpaved roads, the application of dust suppressants for the purpose of controlling fugitive dust generation has been noted to produce changes in the road soil characteristics that influence soil stabilization. Many factors influence soil stabilization. The most notable factors are the physical and chemical properties of the soil and the chemical additive. The stabilization effect of a soil additive is measured in terms of the increase in shear strength of the soil-additive mixture.

It has been demonstrated that lignosulfonates introduce better improvement for ground modification than non-organic stabilizers (Palmer et al. 1995). Lignosulfonates are also used in combination with other chemicals to achieve soil improvement (Puppala and Hanchanloet 1999). Lignin as a soil additive causes dispersion of the clay fraction of some soils, resulting in the shear strength increase of the soil (due to particle rearrangement) (Addo et al. 2004). Many studies have already confirmed that sulfur-containing lignin derivatives act as effective dust suppressants on unpaved roadways.

The utilization of lignin-based BCPs in pavement geo-materials stabilization needs to be investigated because it is hypothesized that stronger geo-materials stabilization may be achieved, possibly reducing the need for as much geo-material through this innovative approach.

## Objective

This research aims to investigate the utilization of BCPs containing lignin in pavement geo-materials stabilization. In the first phase of this research, the following objectives were evaluated: (1) the ability of lignins that are currently available or that are anticipated to become available in the future to act as an effective soil-stabilizing agent and (2) the effect of lignin on the engineering properties of soil-lignin mixtures for Iowa conditions. Based on the research findings, recommendations were made regarding the beneficial effects of using co-products containing lignin as soil stabilizer. It is anticipated that an extended and rigorous evaluation of this concept will be conducted both in the lab and in terms of field performance during the next phase of this research.

## Report Arrangement

This report first presents a literature review (Section 2) that focuses on the past use of natural lignin or sulfite lignin (lignosulfonates) mostly derived from the pulp industry in soil stabilization. Soil stabilization is explained, along with the ways co-products improve soil strength. Section 3 explains the experimental plan and procedures used to analyze lignin-containing co-products as soil performance enhancers. The results and statistical analysis of the experiments for performance evaluation are illustrated and summarized in Section 4. Section 5 is dedicated to explaining the ways co-products improve soil strength and improve the moisture resistance of subgrade soil. The final section summarizes the experimental findings. Recommendations are made, along with suggestions for future work to determine the course of the next phase of this research.

## LITERATURE REVIEW

### Use of Agricultural Biomass for Sustainable Development

Sustainable development has been globally recognized under depleting non-renewable resources (petroleum, natural gas, coal, minerals, etc.), regulations for using synthetic materials, growing environmental awareness, and economic considerations (Kamm and Kamm 2004). Sustainable development requires safe, sustainable resources for various industrial applications (Kamm and Kamm 2004). Even though various alternative raw materials (wind, sun, water, biomass, nuclear fission and fusion) may replace fossil-based energy, biomass, in particular plant biomass, is one of the most economical recourses, and its transformation into "bio-based products" and "bioenergy" through "biorefineries" in new production plants has the potential to replace petroleum-based refineries (Kamm and Kamm 2004).

The United States government has pushed significant industrial developments for using biomass as an alternative energy resource (U.S. President 1999; U.S. Congress 2000). It is expected by 2020 that more than 90% of the consumption of organic chemicals in the United States and up to 50% of liquid fuel needs would be covered by bio-based products (National Research Council 2000; Kamm and Kamm 2004). The European Union (EU) has also requested member states to define national guidelines for a minimal amount of biofuels and other renewable fuels (with a reference value of 2% by 2005 and 5.75% by 2010, calculated on the basis of the energy content of all petrol and diesel fuels for transport purposes) (Kamm and Kamm 2004).

The agricultural biomass is lignocellulosic material consisting of three main constituents: (1) hemicellulose/polyoses, a sugar polymer of predominantly pentoses; (b) cellulose, a glucose polymer; and (c) lignin, a polymer of phenols. Biorefineries combine the essential technologies that have developed and are developing to transform these lignocellulosic materials into various industrial applications, as shown in Figure 1 (Kamm and Kamm 2004). Agricultural byproducts are annually renewable and available in abundance and at present have limited value (Reddy and Yang 2005). Primary lignocellulosic agricultural byproducts having economical effectiveness are corn stover, wheat, rice, barley straw, sorghum stalks, coconut husks (coir), sugarcane bagasse, and pineapple and banana leaves (Reddy and Yang 2005).

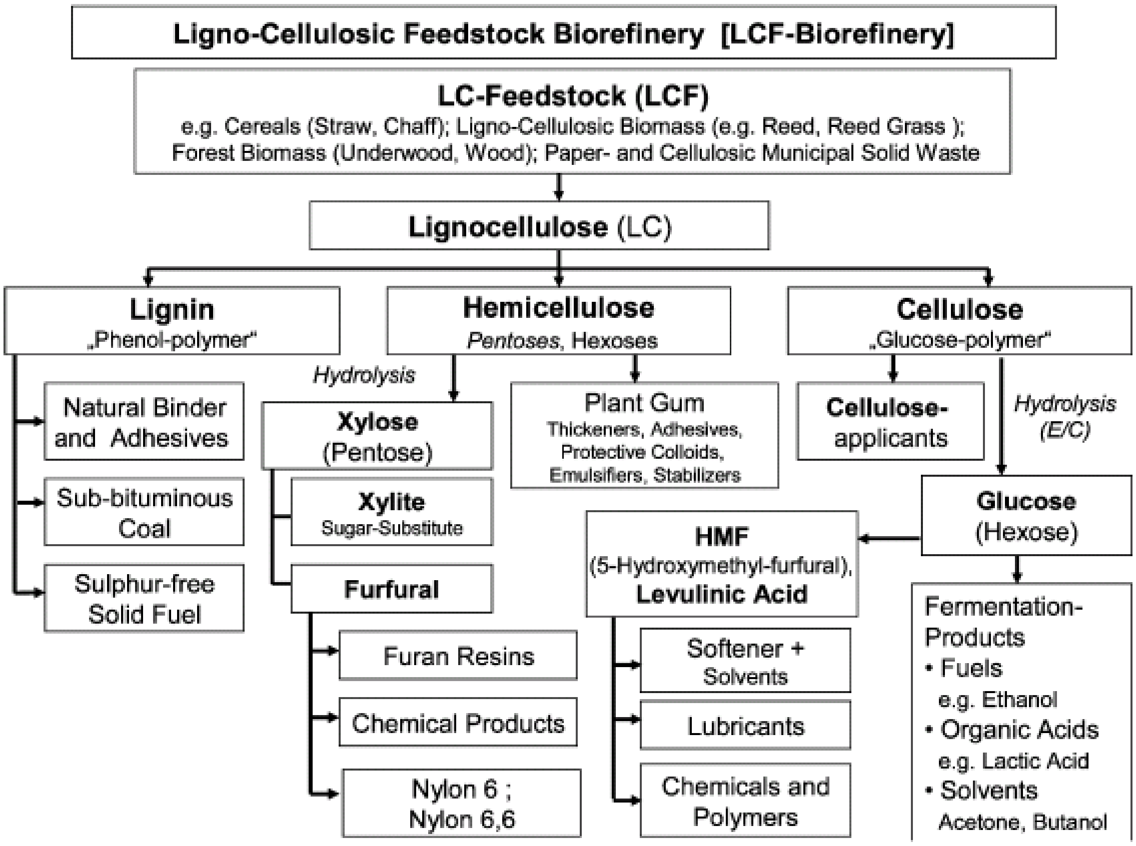


**Figure 1. Industrial applications from lignocellulosic materials (Kamm and Kamm 2004)**

Biofuels or ethanols are the most developed and widely used industrial application in the market from lignocellulosic agricultural materials, such as corn products. Biofuels have several advantages over fossil fuels. Biofuels are renewable, environmentally friendly, provide energy security, and present a large economic development potential in the world (Demirbas and Balat 2006).

Agricultural biomass can be converted into biofuels or ethanols by hydrolysis and subsequent fermentation (Hamelinck et al. 2005). In hydrolysis, the cellulosic part of the biomass is converted into fermentable sugars. To increase the yield of hydrolysis, a pretreatment step that softens the biomass and breaks down cell structures to a large extent is required. The pretreatments not only make the cellulose component susceptible to saccharification, but also have the potential of generating sulfur-free lignin with the hemicellulose as a residue (Hamelinck et al. 2005). Figure 2 presents the generalized biomass-to-ethanol process.

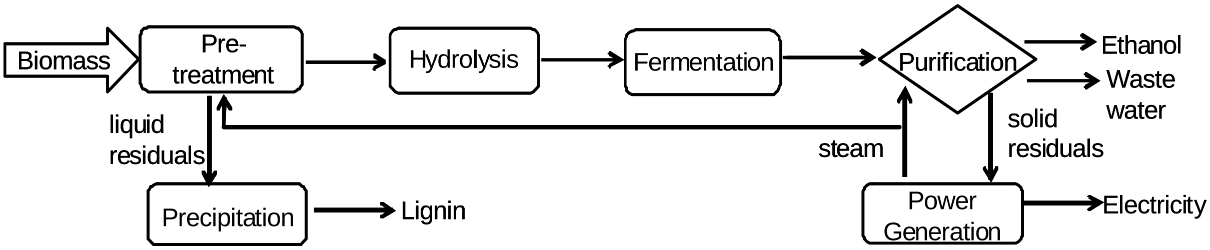


**Figure 2. Generalized biomass to ethanol process diagram (Hamelinck et al. 2005)**

The pretreatment methods can be classified into physical, chemical, or biological. The physical pretreatment is to clean and size the biomass and destroy its cell structure to make it more accessible to further chemical or biological treatment. Desired sizes of the biomass after physical treatment vary from a few centimeters (Wooley et al. 1999) to 1 to 3 mm (Lynd 1996). Common chemical pretreatment methods use dilute acid, alkaline, ammonia, organic solvent, sulfur dioxide, carbon dioxide, or other chemicals. Biological pretreatments use fungi to solubilize/degrade the lignin (Graf and Koehler 2000). Biodelignification is the biological degradation of lignin by microorganisms. Biological pretreatment has the advantage of low energy use and mild environmental conditions. However, the very low hydrolysis rate impedes its implementation (Sun and Cheng 2002). Biological treatments can sometimes be used in combination with chemical pretreatments (Graf and Koehler 2000).

Biofuel production creates many different co-products that have many unexplored uses (Bothast and Schlicher 2005). The type of co-products produced depends on the method of biofuel production and co-products recovery technique and the biomass source. Among many different co-products, lignin, which represents the third largest fraction of lignocellulosic biomass, has been considered as a waste material or a low-value co-product, with its utilization predominantly limited to use as a fuel in the production of octane boosters and in bio-based products and chemical production (Stewart 2008). It is therefore extremely important to recover and convert biomass-derived lignin into high-value product for the profitability of the bio-based products and the bioenergy business (Gopalakrishnan et al. 2010).

## Soil Stabilizer

A good road (paved or unpaved) requires a suitable foundation, which in turn requires soil stability. The degree of stability is primarily a function of the road material resistance to lateral movement or flow (U.S. DOT 1976). Different types of road material employ different mechanisms for resisting lateral movement. In general, granular soils count on their particle sizes, angularity, and interlocking ability to develop the internal friction required to resist lateral flow. However, in fine-grained soils such as clay soils, the stability is very much moisture-dependent.

There are many varieties of soil available for road construction. Unfortunately, many of the soil deposits do not naturally possess the requisite engineering properties to serve as a good foundation material for roads and highways. As a result, soil-stabilizing additives or admixtures are used to improve the properties of less-desirable road soils (ARBA 1976). When used, these stabilizing agents can improve and maintain soil moisture content, increase soil particle cohesion, and serve as cementing and waterproofing agents (ARBA 1976; Gow et al. 1961). Unpaved road dust suppressants are considered soil additives because they produce changes in soil characteristics that influence soil stabilization (Gow et al. 1961; Ross and Woods 1988). Many factors influence soil stabilization. The most notable factors are the physical and chemical properties of the soil and of the chemical additive. The stabilization effect of a soil additive is measured in terms of the increase in shear strength of the soil-additive mixture.

According to the technical manual of the U.S. Departments of the Army and Air Force (JDAAF 1994), "Stabilization is the process of blending and mixing materials with a soil to improve certain properties of the soil. The process may include the blending of soils to achieve a desired gradation or the mixing of commercially available additives that may alter the gradation, texture or plasticity, or act as a binder for cementation of the soil." Kezdi (1979) says, "Soil stabilization means increase of the shear strength of that soil corresponding to the given requirements, and its stabilization [is] independent of the weather conditions or the traffic turnover."

Petry and Little (2002) presented an excellent review of the advances of soil stabilization over the past 60 years, the state of the practice, and the research needs. Based on the mechanism of soil stabilization, they divided a variety of available stabilizers into three groups:

- Traditional stabilizers: hydrated lime, portland cement, and fly ash
- Byproduct lime stabilizers: cement kiln dust, lime kiln dust, and other forms of byproduct lime
- Nontraditional stabilizers: sulfonated oils, potassium compounds, ammonium chloride, enzymes, polymers, and so on

The primary mechanisms for the traditional and byproduct stabilizers are calcium exchange and pozzolanic reactions to effect stabilization. The nontraditional stabilizers the authors identified rely on a different stabilization mechanism, such as hydrogen ion penetration of sulfonated oils into a clay lattice (Petry and Little 2002). A large quantity of studies completed regarding the application of traditional stabilization additives is available in the literature (TRB 1987; ACI 1990; ACAA 1995). However, little independent research has been documented pertaining to the use of nontraditional stabilizer (Santoni et al. 2002).

### Lignin as a Soil Stabilizer

Lignin has been implicated as having a positive role in soil stabilization (Nicholls and Davidson 1958; Kozan 1955; Johnson et al. 2003). Adding lignin to clay soils increases the soil stability by causing dispersion of the clay fraction (Gow et al. 1961; Davidson and Handy 1960). According to Gow et al. (1961), the dispersion of the clay fraction benefits stability of the soil-aggregate mix by (1) plugging voids and consequently improving water tightness and reducing frost susceptibility; (2) eliminating soft spots caused by local concentrations of binder soil; (3) filling voids with fines, thus increasing density; and (4) increasing the effective surface area of the binder fraction, which results in greater contribution to strength.

Lignin is also used in combination with other chemicals to achieve soil improvement (Puppala and Hanchanloet 1999). Lignin as a soil additive causes dispersion of the clay fraction of some soils, resulting in the shear strength increase of the soil (Addo et al. 2004). Various studies on lignin as a soil additive have concluded that lignin is primarily a binding agent (Landon et al. 1983; Ingles and Metcalf 1973; Woods 1960). In most of these studies, sulfite lignin (lignosulfonates) has been utilized.

Laboratory methods as well as onsite testing have been done to quantify soil stabilization using chemical additives, including ligninsulfonate. In one such study, Lane et al. (1984) used laboratory methods to measure soil cohesion increases resulting from the addition of some commercially available chemical additives. The laboratory methods included the unconfined compression test (UCS) (ASTM C 39) and a modified wet sieve analysis test (ASTM C 117). The testing was performed at three sample-drying conditions: 24-hour air-dried, 24-hour bag cured, and immediate sample testing. Figure 3 shows the resulting cohesive strength measured for the 24-hour air-dried test conditions. These results indicate that each additive tested varies in cohesive strength with a range of 4 to 55 psi. The calcium ligninsulfonate at each of the initial aggregate moisture contents (4%, 6%, and 8%) showed a higher cohesive strength than the petroleum-based additives.

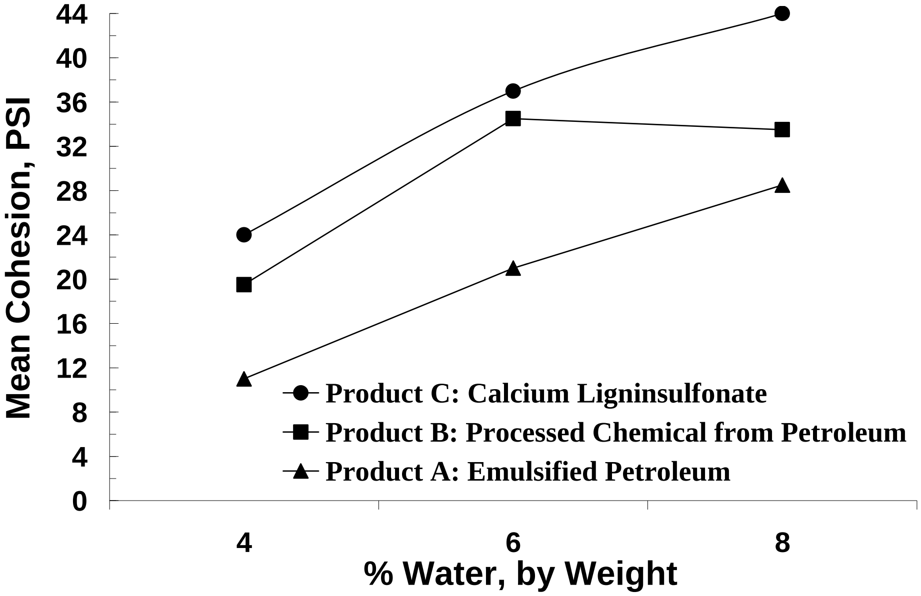


**Figure 3. Effect of moisture content on cohesion of treated aggregate, 24-hour air-dried test condition (adapted from Lane et al. 1984)**

In the past, several studies have been conducted at Iowa State University (ISU) on the use of ligninsulfonate as a stabilizing agent on Iowa's silty loam and loess soils (Sinha et al. 1957; Nicholls and Davidson 1958; Demirel and Davidson 1960; Hoover et al. 1959; Gow et al. 1961). Sinha et al. (1957) found that lignins used alone as admixtures do not show much promise as stabilizing agents for loess or loess-derived soils. However, their investigations indicated that lignins should be much more effective as stabilizing agents for granular soils or soil aggregate mixtures.

The Quebec Department of Roads conducted laboratory tests comparing the engineering properties of lignin-treated aggregate with those of raw aggregate and clay-mixed gravel (Hurtubise 1953). The bearing capacity of the aggregate treated with 1.2% lignin was higher than that of the raw aggregate soil and clay-mixed aggregate. Cohesive strength increased with the addition of 2% lignin. The strength increase was also found to be nearly linearly proportional to the amount of lignin used. Water absorption tests indicated that water absorption through capillary action was substantially reduced. Moisture density relationship tests showed that an increase in the amount of lignin added to the soil increased the density and reduced the optimum moisture content.

In a low-volume road study, laboratory methods were used to evaluate the strength and density modification of unpaved road soils because of chemical additives (Palmer et al. 1995). The additives tested included lignin, $CaCl_2$, and $MgCl_2$ at different concentrations. Three different road soil materials with different soil classifications were used. The seven-day air-cured samples exhibited large strength increases for the lignin-treated specimens at all concentration levels. For each of the soils tested, lignin provided the highest increase in strength, as determined by the unconfined compression tests. Figure 4 illustrates some results for soil 1B, classified as A-1-b by American Association of State Highway and Transportation Officials (AASHTO) designation M-145, and SM by the Unified Soil Classification System (USCS) with PI = 0.

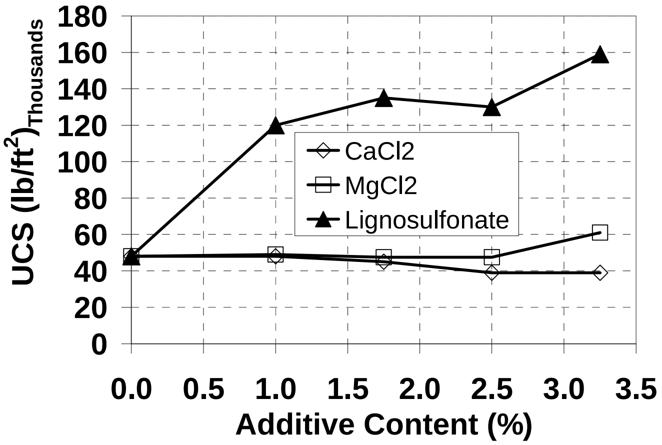


**Figure 4. Average peak UCS for specimens tested dry (adapted from Palmer et al. 1995)**

Puppala and Hanchanloet (1999) studied the effect of a new chemical treatment method using a liquid comprised of sulfuric acid and lignosulfonate stabilizer (SA-44/LS-40 or DRP) on the shear strength and plasticity characteristics of soils. Three soil types, including a silty clay (raw soil) and two types of lime-stabilized raw soils, two chemical dilution rates, and curing periods were investigated. The percent increase in UCS with the SA-44/LS-40 treatment ranged between 30% and 130% for the soils evaluated in this study. The increase in strength properties was attributed to the formation of chemical bonds between soil particles. The lignosulfonate-based chemical treatment increased the resilient moduli of soils, which is important from the point of mechanistic design of flexible pavements.

Nicholls and Davidson (1958) confirmed that lignin admixtures indeed do improve some engineering properties related to soil stability. They also reported that the strength of lignin-treated soil rapidly increases with an increase in the length of air curing. Also, lignin is considered biodegradable; therefore, its presence in the environment can be considered less harmful compared to industrial byproducts, such as fly-ash.

The U.S. Army Engineer Research and Development Center (Santoni et al. 2002; Tingle and Santoni 2003; Santoni et al. 2005) conducted a laboratory experiment to evaluate the stabilization of clay soils and silty-sand with nontraditional chemical or liquid stabilizers. Nontraditional stabilizers were evaluated in this experiment, including an acid, enzymes, a lignosulfonate, a petroleum emulsion, polymers, and a tree resin. Type I portland cement and hydrated lime were used as traditional stabilizers to provide a comparison under the same mixing, compaction, and curing conditions. The UCS test results of each additive under wet and dry condition were compared to those results of the remaining nontraditional additives, the traditional stabilization results, and a series of control specimens that were not stabilized. These studies found that lignosulfonate provide excellent waterproofing for clay soils and silty-sand. The 5% of lignosulfonate was reported as optimum additive quantity for silty-sand.

In most of the previous studies described, natural lignin or sulfite lignin (lignosulfonates) has been utilized. It has been hypothesized that since the BCP derived from plant biomass is high in lignin (sulfur-free lignin), which is thought to play a role in stabilizing soil, soil incorporation of BCP may help maintain or improve soil structure and stability (Gopalakrishnan et al. 2010). The utilization of lignin-based BCPs from biomass in pavement substructure stabilization needs to be investigated as it is hypothesized that one may achieve stronger pavement substructure stabilization, possibly reducing deterioration of pavement system. The feasibility of this approach has been studied in this research.

## PERPORMANCE EVALUATION PROGRAM

### Experimental Materials

*Soil*

Natural soils were collected from a new construction site for U.S. 20 in Calhoun County, Iowa (STA. 706 to STA.712, Project Number NHSX-20-3(102)--3H-13), as shown in Figure 5. The engineering properties of the soil samples are shown in Table 1. The collected soil samples, as shown in Figure 6, can be classified as an A-6(8) soil and CL in accordance with the AASHTO soil classification system and USCS, respectively, and as Class 10 soil per Iowa Department of Transportation (Iowa DOT) specifications (Iowa DOT 2008). The Class 10 soil is the typical excavated soil that includes all normal earth materials, such as loam, silt, clay, sand, and gravel. Based on the engineering properties and Iowa DOT specifications, the Class 10 soil can be limited in construction use under specification or should be removed.

**Figure 5. New construction site for U.S. 20 in Calhoun County**

**Table 1. Engineering properties of investigated soils**

| Property | Soil |
|---|---|
| *Classification* | |
| AASHTO (group index) | A-6(8) |
| USCS group symbol | CL |
| USCS group name | Sandy lean clay |
| *Grain size distribution* | |
| Gravel (> 4.75 mm), % | 7.6 |
| Sand (0.075–4.75 mm), % | 40.4 |
| Silt and clay (< 0.075mm), % | 51.9 |
| *Atterberg limits* | |
| Liquid limit (LL) , % | 39 |
| Plasticity limit (PL), % | 16. |
| Plasticity index (PI), % | 23 |
| *Proctor test* | |
| Optimum moisture content (OMC), % | 17.7 |
| Maximum dry unit weight ($\gamma_{d\ max}$), kg/m$^3$(pcf ) | 1,691 (105.7) |

**Figure 6. Soil collected for research**

*Additives*

Two types of BCPs containing lignin were used as additives and designated as co-products A and B in this study. Co-product A, shown in Figure 7, was obtained from a commercial biomass conversion facility located in Canada. This BCP is a dark brown, free-flowing liquid fuel with a smoky odor reminiscent of the plant from which it is derived. It is formed in a process called fast pyrolysis where plant material (biomass), such as forest residues (bark, sawdust, shavings, etc.)

and agricultural residues (sugar cane, cornhusks, bagasse, wheat straw, etc.), are exposed to 400°C to 500°C in an oxygen-free environment (Dynamotive Energy Systems Corporation 2007). Recently, several qualification trial tests of co-product A for heating the Iowa Capitol Complex were conducted by the State of Iowa Department of Administrative Services-General Services Enterprise (Iowa DAS-GSE) in partnership with Dynamotive Energy Systems Corporation and Biogreen Resources (Iowa DAS 2008). Co-product A contains about 25% lignin and up to 25% water with a pH value of 2.2. The water component in co-product A to be used as liquid fuel is not a separate phase because it lowers the viscosity of the fuel. Table 2 presents the contents of component materials in co-product A.

**Figure 7. BCP A**

**Table 2. Component materials in BCP A**

| Components | % by weight |
|---|---|
| Gases | 5 to10% |
| Water | Up to 25% |
| Lignin | 25% |
| Char | 4% |
| Aldehydes | 35% to 41% |

Co-product B, shown in Figure 8, was obtained from a full-scale, wet-mill, corn-based ethanol plant of Grain Processing Corporation (GPC) of Muscatine, Iowa (GPC 2009). Alkaline-washed corn hull is obtained in the process of converting the corn into ethanol, and co-product B is a powdered version of this. Co-product B contains about 5% lignin, 50% hemicellulose, 20% cellulose, and other components. These lignin-type components are not high molecular weight lignin like those found in wood but are specific to maize.

**Figure 8. BCP B**

The Ottumwa Class C fly ash was selected as the traditional additive to compare BCPs' relative performance. The Ottumwa Class C fly ash is a coal combustion byproduct from the Ottumwa Generating Station (OGS) located near Chillicothe, Iowa. This fly ash is commonly used for soil treatment in Iowa. The chemical compositions of Ottumwa fly ash are presented in Table 3.

**Table 3. Chemical composition of Ottumwa fly ash**

| Components | % by weight |
|---|---|
| $SiO_2$ | 31.6 |
| CaO | 28.8 |
| $Al_2O_3$ | 16.2 |
| MgO | 6.81 |
| $Fe_2O_3$ | 6.03 |
| $Na_2O$ | 3.21 |
| $SO_3$ | 3.13 |
| $TiO_2$ | 1.24 |
| $P_2O_5$ | 1.02 |
| BaO | 0.89 |
| SrO | 0.51 |
| $K_2O$ | 0.32 |
| LOI | 0.3 |
| $Mn_2O_3$ | 0.02 |

## Experimental Plan

The experimental tests in this study included the UCS test as a strength property test and Atterberg limits and standard Proctor compaction tests as engineering properties tests. For comparison purposes, the primary experimental plan for the strength property test encompassed preparing and testing four broad categories of treatment types: (1) untreated soil sample (control), (2) soil sample treated with the BCP A, (3) soil sample treated with the BCP B, and (4) soil sample treated with fly ash. Table 4 lists the primary treatment group combinations evaluated for the UCS test during this study. Soil was mixed with each additive (BCPs or fly ash) at variable percentages to examine their influence. The co-product A and fly ash contents evaluated are 1%, 3%, 6%, 12%, and 15% by dry soil weight. The co-product B contents evaluated are 1%, 6%, and12% by dry soil weight. The untreated soils were also tested without the addition of any co-product.

Similarly, the moisture contents and curing periods were incorporated as variables into the test factorial. The levels of water content (WC) for the testing samples were optimum moisture content (OMC), OMC+4%, and OMC-4% of untreated soil. The curing periods primarily investigated were one and seven days after sample fabrication for strength tests.

**Table 4. Primary treatment group combinations for strength property tests**

| Moisture content level | Curing period | Additives[a], % | | |
|---|---|---|---|---|
| | | Co-product A | Co-product B | Fly Ash |
| OMC-4 | 1 day | 0, 1, 3, 6, 12, 15 | 0, 1, 6, 12 | 0, 1, 3, 6, 12, 15 |
| | 7 days | 0, 1, 3, 6, 12, 15 | 0, 1, 6, 12 | 0, 1, 3, 6, 12, 15 |
| OMC | 1 day | 0, 1, 3, 6, 12, 15 | 0, 1, 6, 12 | 0, 1, 3, 6, 12, 15 |
| | 7 days | 0, 1, 3, 6, 12, 15 | 0, 1, 6, 12 | 0, 1, 3, 6, 12, 15 |
| OMC+4 | 1 day | 0, 1, 3, 6, 12, 15 | 0, 1, 6, 12 | 0, 1, 3, 6, 12, 15 |
| | 7 days | 0, 1, 3, 6, 12, 15 | 0, 1, 6, 12 | 0, 1, 3, 6, 12, 15 |

a. Numbers indicate percent additive added by dry soil weight.

Apart from the primary treatment group combinations listed in Table 4, a secondary experimental plan was made to investigate the effect of additive combinations on strength. The additive combinations investigated were of two categories of treatment types: (1) soil sample treated with BCP A + fly ash and (2) soil sample treated with BCPs A + B. Table 5 lists the secondary treatment group combinations evaluated during the study. The strength results of secondary treatment group combinations were compared to those results of the untreated soil samples and 12% of the additives treated soil samples made in primary experimental plan.

**Table 5. Secondary treatment group combinations for strength property tests**

| Moisture content level | Curing period | Additives[a], % | |
|---|---|---|---|
| | | Co-product A + Fly Ash | Co-products A + B |
| OMC-4 | 1 day | 10+2 [b], 6+6, 2+10 | 10+2, 6+6, 2+10 |
| | 7 day | 10+2, 6+6, 2+10 | 10+2, 6+6, 2+10 |
| OMC | 1 day | 10+2, 6+6, 2+10 | 10+2, 6+6, 2+10 |
| | 7 day | 10+2, 6+6, 2+10 | 10+2, 6+6, 2+10 |
| OMC+4 | 1 day | 10+2, 6+6, 2+10 | 10+2, 6+6, 2+10 |
| | 7 day | 10+2, 6+6, 2+10 | 10+2, 6+6, 2+10 |

a. Numbers indicate percent of additive added by dry soil weight.
b.10% co-product A and 2% fly ash.

Atterberg limits and standard Proctor compaction tests as engineering properties tests were also carried out on soil samples prepared with additives at selected percentages in order to examine their influence. The selected percentage of co-products was the one in which the values of compression strength were at maximum.

## Strength Property Testing

The stabilization effect of the soil additive is measured in terms of the increase in load-bearing capacity, as indicated by UCS (Lane et al. 1984; Palmer et al. 1995; Tingle and Santoni 2003). This study also utilized the UCS test as the basis of performance characterization.

ASTM D 2166 (2006) "Standard test method for unconfined compressive strength of cohesive soil" provides general UCS test procedures but doesn't specify the sample geometry. The Portland Cement Association (PCA) recommends three types of sample geometries for compression tests of soil-cement mixture: 4 in. diameter by 4.6 in. height, 2 in. diameter by 2 in. height, and 2.8 in. diameter by 5.6 in. height (PCA 1971). The compaction method for a 2 in. diameter by 2 in. high specimen was developed by researchers at ISU (Chu and Davidson 1960). The compression strengths of 2 in. by 2 in. specimens have been correlated to that of the other geometry specimens for the soil and the soil–fly ash mixture (Chu and Davidson 1960; White et al. 2005). The use of 2 in. by 2 in. specimens can save materials and time. Because of these advantages, the specimens for the UCS test in this study were molded to 2 in. diameter by 2 in. height. The ISU 2 in. by 2 in. method is described in detail by Chu and Davidson (1960).

### *Sample Preparation*

Each sample for UCS testing was prepared following five steps: soil preparation, soil-water-additive mixing, molding, compaction, and curing. The natural soil collected was dried and broken down to particle sizes that could pass a No. 4 (4.75 mm) sieve. Additives were also dried below 60°C to remove the initial water in the co-products and then the water content of co-products was close to 0%.

Once the soil and additive were prepared, the soil was mixed with the water and additives to obtain the desired moisture and additive contents. The materials were mixed together to produce a uniform, homogenous mixture. A sample of the mixture was used to determine the initial moisture content of the soils, according to ASTM D 2216 (2005) "Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass."

A quantity of loose material was measured for each sample that would produce a 2 in. high compacted sample. The ISU 2 in. by 2 in. specimen preparation method specifies that loose materials are compacted in a 2 in. diameter mold with removable collar by dynamic loading. The term "dynamic loading" herein refers to five blows of a 5 lb hammer falling from a height of 12 in. on each end of the single layer of material (Chu and Davidson 1960). However, it was found that this compaction approach did not produce compacted samples with uniform density, which was important for making comparisons. To achieve uniform density of samples, a static compaction approach was employed, which is similar to the approach used in soil specimen preparation for resilient modulus test in accordance with AASHTO T 307 (1999) “Determining the Resilient Modulus of Soils and Aggregate Materials.”

Specially designed mold apparatuses, shown in Figure 9, were fabricated and used to compact loose materials by static compaction. A 1 in. high spacer plug was inserted into the specimen mold with removable collar. Measured amounts of loose materials were placed in the specimen mold and a 4 in. high spacer plug was inserted on the loose materials in the specimen mold. A static load was applied to the 4 in. high spacer plug until the plug rested firmly against the mold end. After compaction was completed, the compacted specimen, as shown in Figure 10, was extracted from the mold using an extrusion ram. The compacted sample was sealed in a plastic wrap and placed in a temperature-controlled room where it was allowed to cure at 25°C and 40% relative humidity to represent field conditions.

**Figure 9. Mold apparatuses for static compaction**

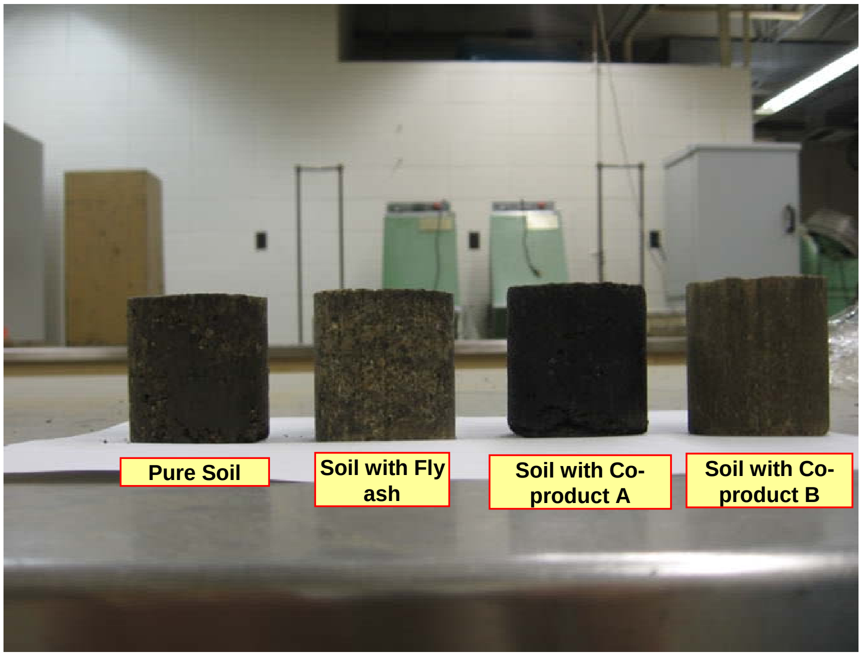


**Figure 10. Prepared samples for UCS test**

*Unconfined Compression Strength (UCS) Test*

The UCS tests were conducted following ASTM D 2166 (2005) after various curing times. A cured sample was positioned in the test instrument. The compression load was applied to each sample at a constant rate of 0.05 in. per minute. The magnitude of the compression load and the corresponding sample deformation were monitored and recorded. Each sample was compressed until a peak load was reached and then decreased or remained constant or until deformation of the sample was past 20% strain before reaching the peak. A sample of the broken material was used to determine the moisture content of the materials according to ASTM D 2216 (2005).

## Engineering Properties Testing

Soil samples prepared with additives at selected percentages were subjected to engineering properties tests to determine their physical properties and compaction characteristics. Engineering properties tests included Atterberg limits (liquid limit [LL] and plastic limit [PL]) according to ASTM D 4318 (2005) “Standard test method for liquid limit, plastic limit and plasticity index of soils” and moisture/density relationship in general accordance with ASTM D 698 (2007) “Method A: Standard test methods for laboratory compaction characteristics of soil using standard effort [12,400 ft-lbf/ft$^3$].”

# PERPORMANCE EVALUATION RESULTS AND ANALYSIS

## Strength Property Test Results and Analysis

### *Primary Test Results and Analysis*

The effects of co-product types and contents on UCS were evaluated under different moisture conditions: OMC represented moisture condition providing the maximum dry density of soil and used for quality control of construction, OMC-4% represented the more dry side of soil condition, and OMC+4% represented the more wet side of soil condition. The evaluations were also made under different curing periods. The results are shown graphically in Figures 11 through 13. The UCS values at 0% additive content in these figures indicate untreated soil after one and seven days of curing. The strengths of untreated soils are in all cases lower than the strengths of additive-treated soils. Overall, the strengths under the more dry side of soil condition are higher than the more wet side of soil condition. A high increase in strengths occurs with 12% of co-product A in all cases.

As shown in Figure 11, fly ash representing the traditional soil stabilizer clearly stands out as the most effective additive to enhance the strength of tested soil under the dry condition of soil (OMC-4%). Both co-products (A and B) are also effective in enhancing the strength of soil under the dry condition (OMC-4%). Soil samples treated with co-products obtained more strength with the increased addition of co-products. Especially, the increase in strength of co-product A–treated soil with increased additive content is higher than the strength of co-product B–treated soil under the dry condition of soil. The curing periods influence the strength gain of soil treated by co-product A but not soil treated by co-product B.

Similar to the dry condition of soil (OMC-4%), each of the co-products–treated soil UCS test results in Figure 12 shows strength improvements under the OMC condition of soil, which represents moisture condition for construction. However, the strength improvements of the co-products–treated soil are not higher than those improvements of the fly ash–treated soil. The curing periods influence the strength gain of soil treated by co-product A but not soil treated by co-product B. The 1-day strengths of co-product A–treated soil are lower than the strengths of co-product B–treated soil. However, the 7-day strengths of co-product A–treated soil are higher than those of co-product B–treated soil.

Figure 13 shows that both co-products are still effective in improving the strength of soil under wet conditions of soil (OMC+4%). The strengths of treated soil increase with the increase in co-product concentrations and curing periods. The strengths of soil treated by co-product B are higher than the strengths of soil treated by co-product A. All the results under different moisture conditions indicate that co-product A is more effective in improving strength under dry conditions while co-product B is more effective in improving strength under wet conditions.

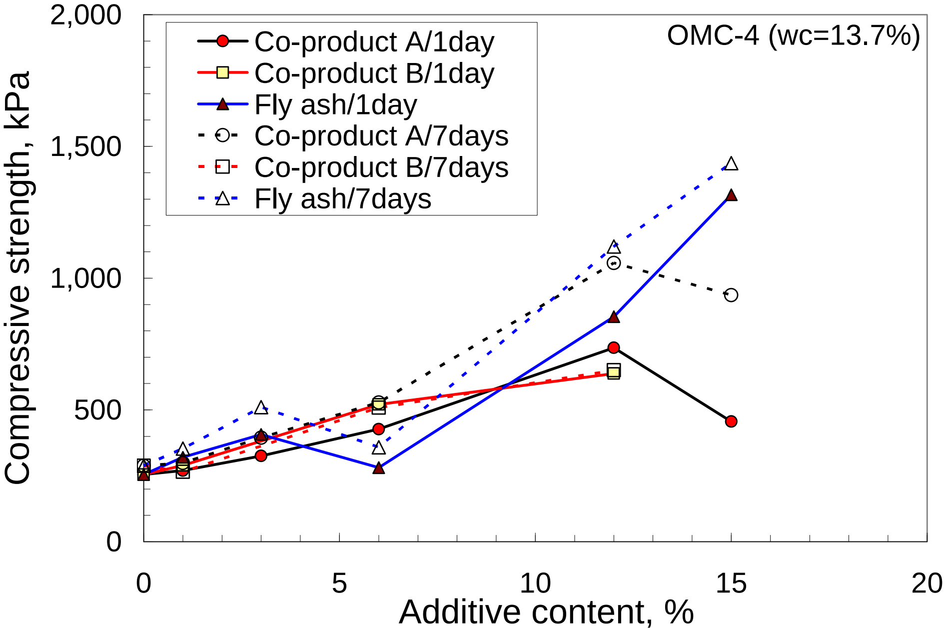


**Figure 11. Variation of UCS under OMC-4% condition**

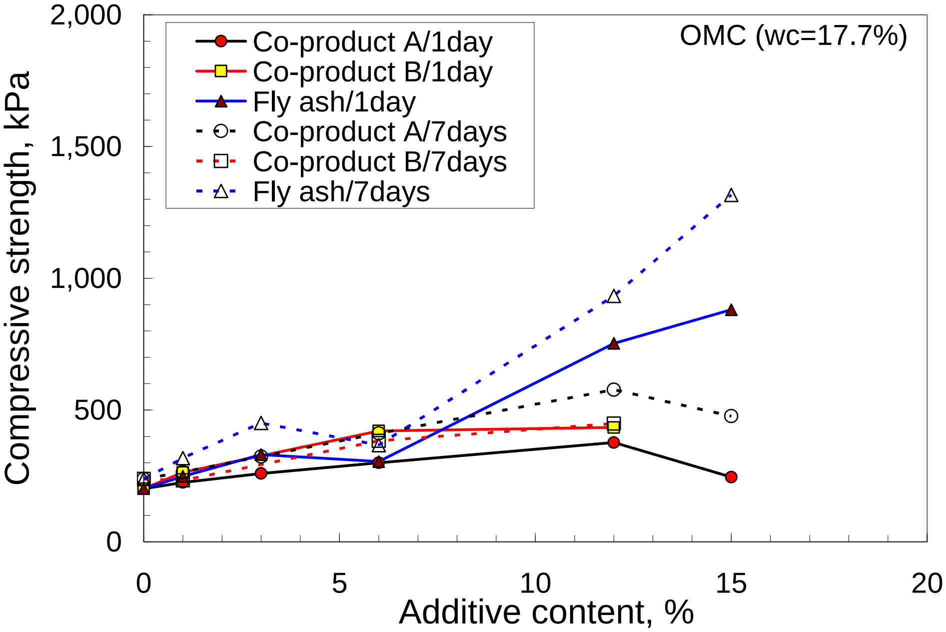


**Figure 12. Variation of UCS under OMC condition**

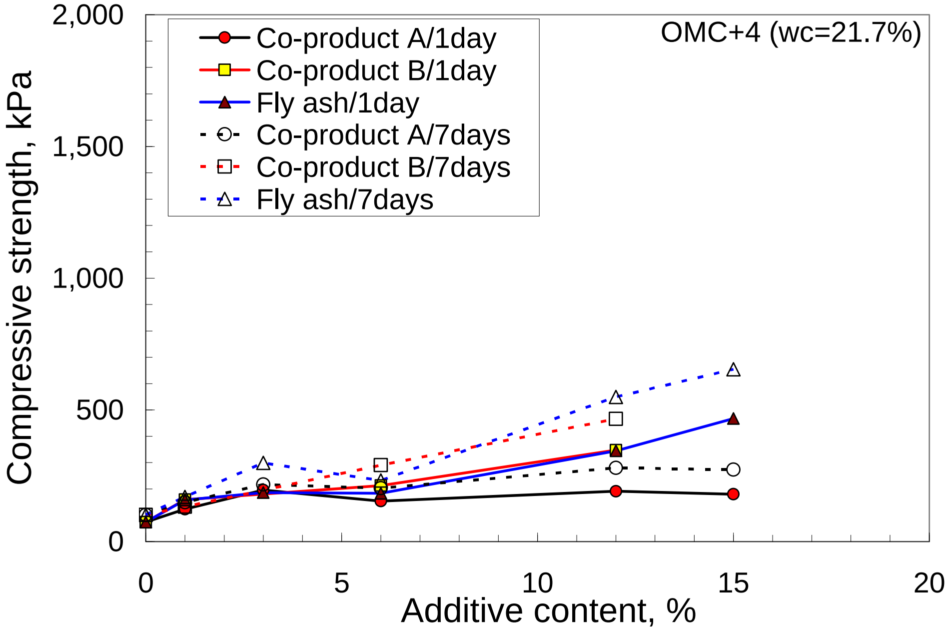


**Figure 13. Variation of UCS under OMC+4% condition**

Paired t-tests were performed to evaluate if the strength improvements of the co-products–treated soils were significant. Two kinds of t-tests, an independent t-test and a paired t-test, can be used to examine the differences between the two groups. In statistical tests, an independent t-test uses the difference of means between two groups, while a paired t-test uses the mean of differences between the observations in one group and the matched observations in the other group. A paired t-test used in this study can consider the correlation between observations, which can be ignored in an independent t-test (SAS Institute, Inc. 2005), i.e., the paired t-test is stricter than an independent t-test. A paired t-test result can be expressed in terms of a p-value, which represents the weight of evidence for rejecting the null hypothesis (Ott and Longnecker 2001). The null hypothesis is the equality of the mean of differences between comparisons. The null hypothesis can be rejected (i.e., the mean of difference between comparisons are significantly different) if the p-value is less than the selected significance level (α). A Type I error (α) of 0.05 was used for all paired t-tests.

Each additive treatment combination was compared to the natural soil to determine if the strength differences were significant. As seen in Table 6, the strengths of natural soil are not significantly different from 1% of co-products A and B under OMC-4% and OMC conditions and 15% of co-product A under OMC conditions. All other treatment combinations possessed a significantly higher strength than the natural soil. Each co-product treatment combination was also compared to the fly ash treatment combination. Table 7 presents the paired t-test results for these comparisons. All the paired t-test results indicate that the co-product A and B are effective in improving soil strength, but this improvement is not higher than the one resulting from fly ash.

**Table 6. Paired t-test results for comparisons of natural soil strengths with the additive-treated soil strengths**

| | Additive content | Moisture content | | | | | |
|---|---|---|---|---|---|---|---|
| | | OMC-4 | | OMC | | OMC+4 | |
| Additive type | (%) | p-value | Different? | p-value | Different? | p-value | Different? |
| Fly Ash | 1 | 0.0008 | Yes | 0.0116 | Yes | 0.0007 | Yes |
| Fly Ash | 3 | 0.0057 | Yes | 0.0039 | Yes | 0.0075 | Yes |
| Fly Ash | 6 | 0.0172 | Yes | 0.0043 | Yes | 0.0002 | Yes |
| Fly Ash | 12 | 0.0017 | Yes | 0.0013 | Yes | 0.0046 | Yes |
| Fly Ash | 15 | <.0001 | Yes | 0.0027 | Yes | 0.0012 | Yes |
| Co-product A | 1 | 0.2373 | No | 0.1433 | No | 0.0007 | Yes |
| Co-product A | 3 | 0.0075 | Yes | 0.0186 | Yes | 0.0002 | Yes |
| Co-product A | 6 | 0.0023 | Yes | 0.0064 | Yes | 0.0031 | Yes |
| Co-product A | 12 | 0.0026 | Yes | 0.0103 | Yes | 0.0037 | Yes |
| Co-product A | 15 | 0.0244 | Yes | 0.0518 | No | 0.0034 | Yes |
| Co-product B | 1 | 0.406 | No | 0.1308 | No | 0.0283 | Yes |
| Co-product B | 6 | 0.0018 | Yes | 0.0018 | Yes | 0.0015 | Yes |
| Co-product B | 12 | 0.003 | Yes | 0.0003 | Yes | 0.0013 | Yes |

**Table 7. Paired t-test results for comparisons of the fly ash–treated soil strengths with the BCP-treated soil strengths**

| | Additive content | Moisture content | | | | | |
|---|---|---|---|---|---|---|---|
| | | OMC-4 | | OMC | | OMC+4 | |
| Additive type | (%) | p-value | Different? | p-value | Different? | p-value | Different? |
| Co-product A | 1 | 0.0443 | Yes | 0.1207 | No | 0.0088 | Yes |
| Co-product A | 3 | 0.0055 | Yes | 0.0252 | Yes | 0.1844 | No |
| Co-product A | 6 | 0.0019 | Yes | 0.1436 | No | 0.0239 | Yes |
| Co-product A | 12 | 0.0683 | No | 0.0111 | Yes | 0.0076 | Yes |
| Co-product A | 15 | 0.0042 | Yes | 0.0008 | Yes | 0.0012 | Yes |
| Co-product B | 1 | 0.0535 | No | 0.1707 | No | 0.1420 | No |
| Co-product B | 6 | 0.0072 | Yes | 0.0625 | No | 0.0015 | Yes |
| Co-product B | 12 | 0.0278 | Yes | 0.0042 | Yes | 0.1660 | No |

### *Secondary Test Results and Analysis*

The effects of various additive combinations on strength were evaluated under different moisture conditions. The results are shown graphically in Figures 14 through 16. “A,” “B,” and “FA” in these figures correspond to co-product A, co-product B, and fly ash, respectively. Overall, the strengths of untreated soils are in all cases lower than the strengths of combined additive-treated soils. Paired t-tests were also performed to evaluate if the strengths of different additive combinations were statically different than the fly ash combinations. Table 8 presents these paired t-test results.

As shown in Figure 14, among combined additive combinations are the 10% co-product A + 2% fly ash combination and the 10% co-product A + 2% co-product B combination that present higher strengths for OMC-4%. Even though the average values of strength for these combinations are less than fly ash, 0.1208 and 0.0749 of p-values in Table 6 indicate that these differences are not significant. The strength results for OMC in Figure 15 show that the 6% co-product A + 6% fly ash combination possesses higher strength among combined additive combinations. The strength of this combination is higher than that of co-product A or B. However, the strength of this combination is lower than that of fly ash. As shown in Figure 16 and Table 8, the combined additive concentrations of co-product A and fly ash possess strengths comparable to fly ash for OMC+4%. The strength of the 10% co-product A + 2% co-product B combination is also not significantly different than those combinations with fly ash.

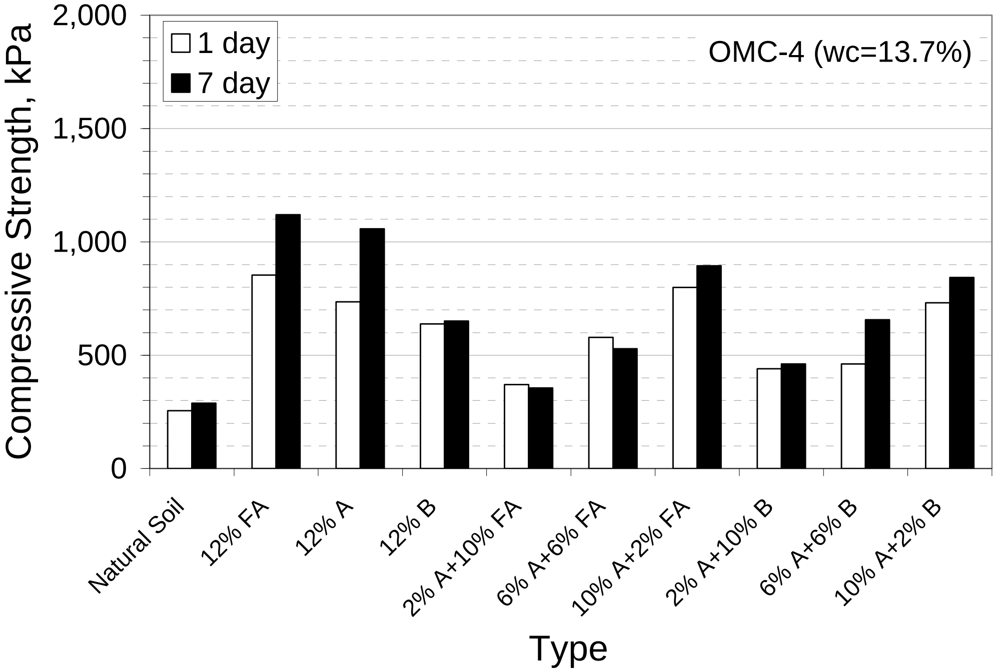


**Figure 14. UCS results for secondary treatment group under OMC-4% condition**

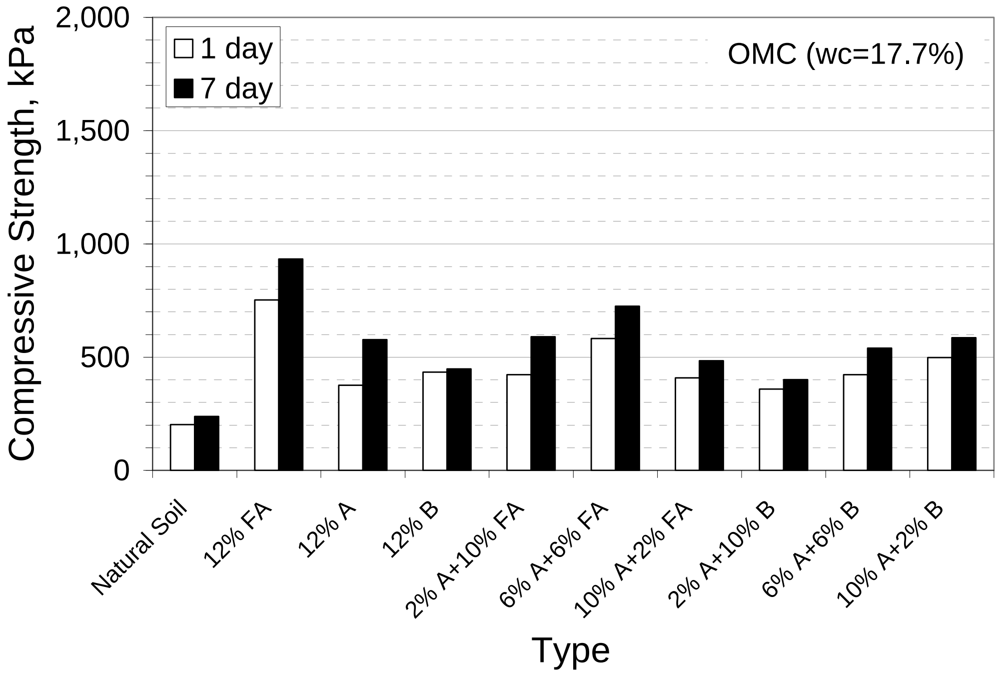


**Figure 15. UCS results for secondary treatment group for OMC condition**

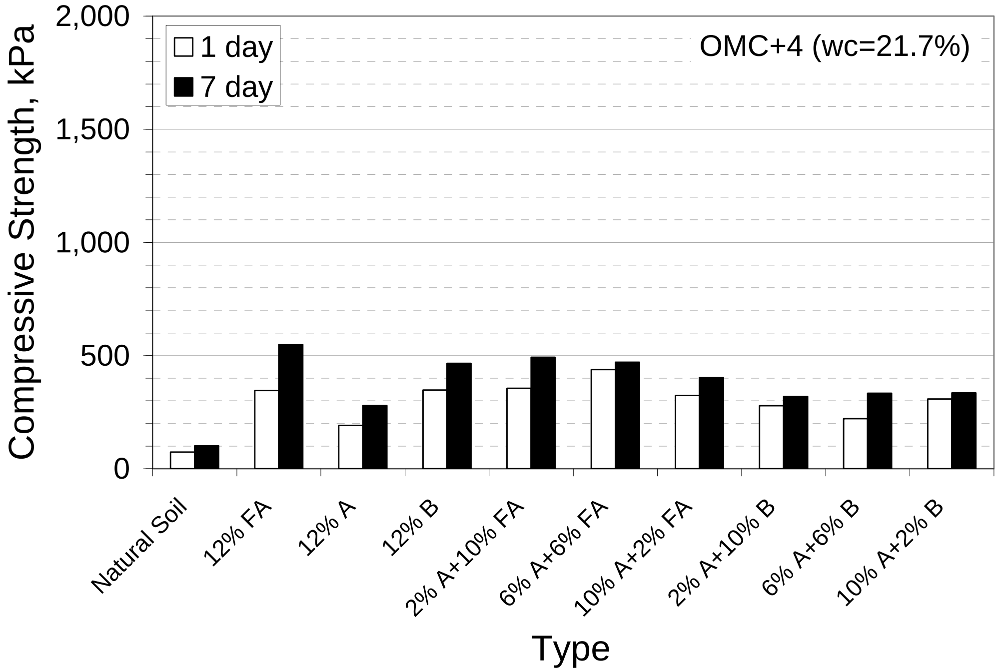


**Figure 16. UCS results for secondary treatment group for OMC+4% condition**

**Table 8. Paired t-test results for comparisons of fly ash–treated soil strengths with the combined additives treated soil strengths**

| | | Moisture content | | | | | |
|---|---|---|---|---|---|---|---|
| | Additive | OMC-4 | | OMC | | OMC+4 | |
| Additive type | content (%) | p-value | Different? | p-value | Different? | p-value | Different? |
| Co-product A and Fly Ash | 2 + 10 | 0.0047 | Yes | 0.0077 | Yes | 0.2898 | No |
| Co-product A and Fly Ash | 6 + 6 | 0.0129 | Yes | 0.0235 | Yes | 0.4651 | No |
| Co-product A and Fly Ash | 10 + 2 | 0.1208 | No | 0.0033 | Yes | 0.0955 | No |
| Co-products A and B | 2 + 10 | 0.0039 | Yes | 0.002 | Yes | 0.0468 | Yes |
| Co-products A and B | 6 + 6 | 0.0027 | Yes | 0.0045 | Yes | 0.0176 | Yes |
| Co-products A and B | 10 + 2 | 0.0749 | No | 0.0056 | Yes | 0.0693 | No |

## Engineering Properties Test Results

Figures 17 and 18 present the effect of additives on consistency limits and compaction properties of soil, respectively. Twelve percent of co-product content was selected in this evaluation because a high increase in UCS occurred with 12% of co-products in this study. The co-product A, similar to fly ash, reduced the plasticity of soils as a result of an increase in the plastic limit value, but the co-product B increased the plasticity of soils as a result of an increase in the liquid limit and plastic limit values.

Co-product A decreases the OMC with 1,664 kg/m$^3$ of the maximum dry unit weight when compared to the maximum dry unit weight of untreated soil. However, co-product B decreases the maximum dry unit weight with 16.5% of OMC when compared with the maximum dry unit weight of untreated soil. The decrease in maximum dry unit weight with co-product B is attributed to the lower specific gravity of co-product B than that of soil. These results indicate that co-product A is a more promising additive, considering the reduction in the plastic property and the decrease in OMC with increasing maximum dry unit weight as indicators of improvement for soil stabilization purposes.

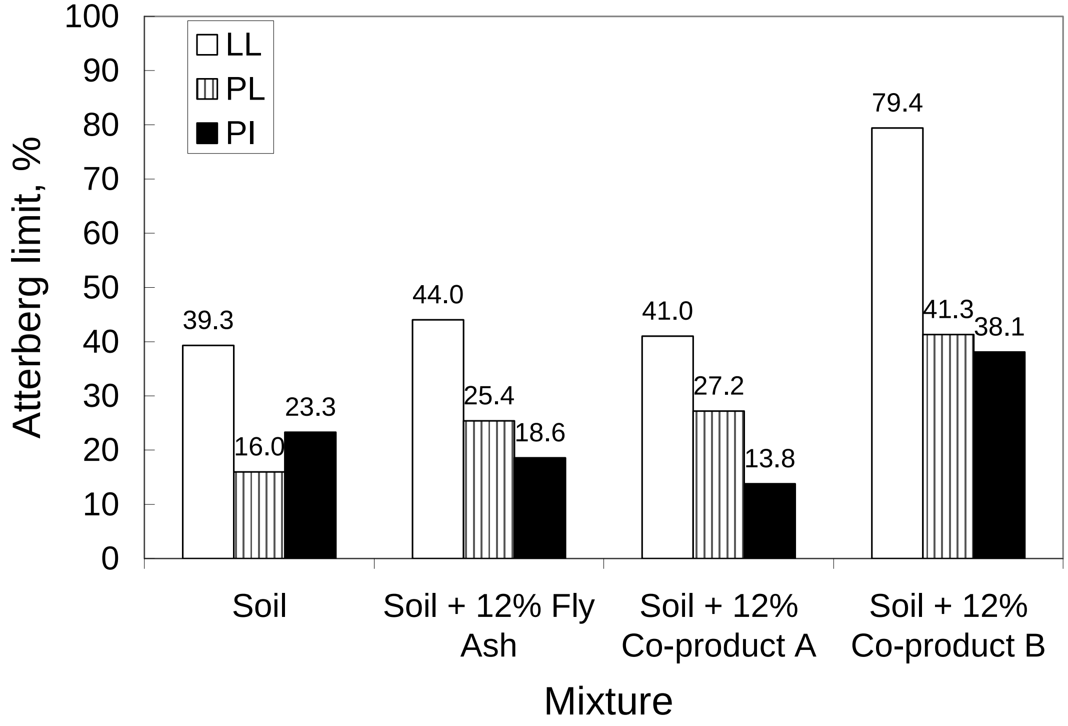


**Figure 17. Effect of additives on consistency limits of soil**

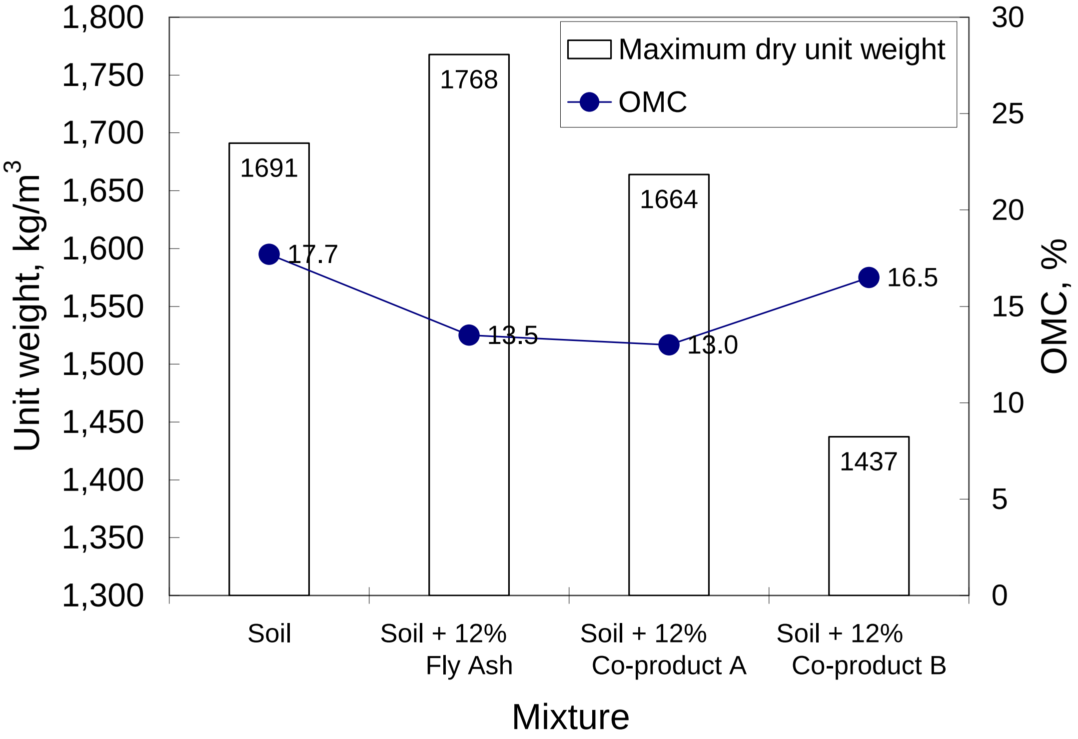


**Figure 18. Effect of additives on compaction properties of soil**

## MOISTURE SUSCEPTIBILITY EVALUATION

### Experimental Program

Following performance evaluations, the effect of lignin-based BCPs on the moisture susceptibility of subgrade soil after compaction was investigated. The laboratory experimental program was conducted using two types of tests: the UCS tests after "dry" and "wet" conditioning procedures and the visual observations of soaked specimens (so-called soaking tests). The UCS test was applied to dry and wet specimens to evaluate moisture susceptibility of additive-treated specimens. Specimens were also fully soaked in water over a period of time to examine if specimens would fail due to moisture damage and, if so, at what time periods.

The required amounts of additives for sample preparation were selected as 12% of each uncombined additive (co-product A, co-product B, and fly ash), 10% of co-product A and 2% of fly ash, and 10% of co-product A and 2% of co-product B. These amounts of additive provided the highest strengths of treated soil mixtures in previous performance characterization. The control untreated soil mixtures were also made without the addition of any additives. The target moisture content for all mixtures was OMC of untreated soil.

### Unconfined Compression Strength (UCS) Test and Results

The compacted specimens of each mixture were subjected to "dry" and "wet" pre-conditioning procedures for UCS tests once the designated curing period was complete. "Dry" pre-conditioned specimens were tested without water saturation, while "wet" pre-conditioned specimens were tested using specified water saturation procedures. U.S. Army Engineer Research and Development Center (ERDC) utilized "dry" and "wet" pre-conditioning procedures for UCS tests to evaluate the stabilization of clay soils and silty-sand with nontraditional chemical or liquid stabilizers (Santoni et al. 2002; Tingle and Santoni 2003; Santoni et al. 2005). ERDC reviewed several stabilized materials' moisture susceptibility tests and suggested that available tests were deemed to be either not representative of field conditions, too complicated for large numbers of repetitions, or too harsh to permit effective specimen evaluation. Thus, ERDC developed a simplistic "wet" test procedure to evaluate the stabilized material's moisture susceptibility. The "wet" test procedure utilized in this research was similar to the one developed by ERDC.

The "wet" test procedures in this research include a full saturation and a half saturation of the specimen. A full saturation of the specimen requires completely immersing the specimen on its side in a water bath for a period of one hour. A half saturation of the specimen was also conducted because some of specimens were broken in full saturation. Note that the full saturation of the specimen was not selected in ERDC's study because of same reason. Half a side of the specimen was soaked in water for a period of five minutes (see Figure 19).

A specimen subjected to full saturation or half saturation was then removed from the water and allowed to drain for five minutes. The specimen was then subjected to UCS testing in accordance with ASTM D 2166 (2006). The "wet" procedure permitted not only a physical evaluation of structural strength loss due to moisture but also a visual observation of the susceptibility to moisture (Santoni et al. 2002; Tingle and Santoni 2003; Santoni et al. 2005). Table 9 lists the experimental treatment group combinations evaluated for the UCS test during this research.

**Figure 19. Example of fly ash treated specimen under half-saturation procedure**

**Table 9. Experimental treatment group combinations for UCS test**

| Conditioning | Curing period | Additives[a], % | | | | |
|---|---|---|---|---|---|---|
| | | Co-product A | Co-product B | Fly Ash | Co-product A + Fly Ash | Co-products A + B |
| Dry | 1 day | 0, 12 | 0, 12 | 0, 12 | 0, 10+2[b] | 0, 10+2 |
| | 7 days | 0, 12 | 0, 12 | 0, 12 | 0, 10+2 | 0, 10+2 |
| Wet – full saturation | 1 day | 0, 12 | 0, 12 | 0, 12 | 0, 10+2 | 0, 10+2 |
| | 7 days | 0, 12 | 0, 12 | 0, 12 | 0, 10+2 | 0, 10+2 |
| Wet - half saturation | 1 day | 0, 12 | 0, 12 | 0, 12 | 0, 10+2 | 0, 10+2 |

a. Numbers indicate percent of additive added by dry soil weight.
b.10% co-product A and 2% fly ash.

The results of UCS tests under "dry" and "wet" (half and full saturation) pre-conditions are shown graphically in Figures 20 through 22. After one day curing, the specimens were subjected to dry and half- and full-saturation conditions for the UCS tests, as shown in Figures 20 and 21. After seven days of curing, the specimens were subjected to dry and full-saturation conditions for UCS tests, as shown in Figure 22. "A," "B," and "FA" in these figures are co-product A, co-product B, and fly ash. The "0" value in Figures 21 and 22 indicates that a specimen disintegrated when exposed to water. The untreated soil specimen was used as a control specimen. Overall, the strengths of additive-treated soils were in all cases higher than untreated soils under the "dry" and "wet" conditions. The fly ash–treated soil test results show the most improvement of UCS under the “dry” conditions. However, the fly ash–treated soil specimens disintegrated during “wet” pre-conditioning (see Figure 21) or provided more strength reduction after “wet” pre-conditioning compared to the co-products–treated soil specimens (see Figures 20 and 22). The curing periods have less influence on strength gain of soil specimens treated by co-product B than the strength gain of the other soil specimens.

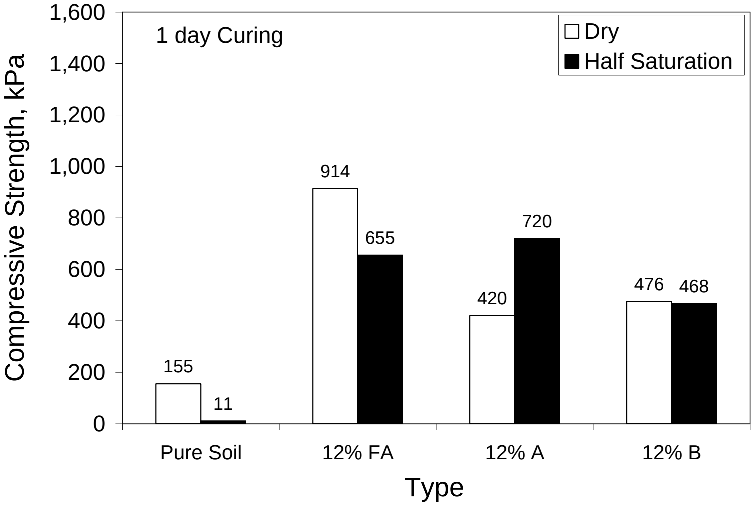


**Figure 20. UCS test results for dry and half-saturated specimens after one day curing**

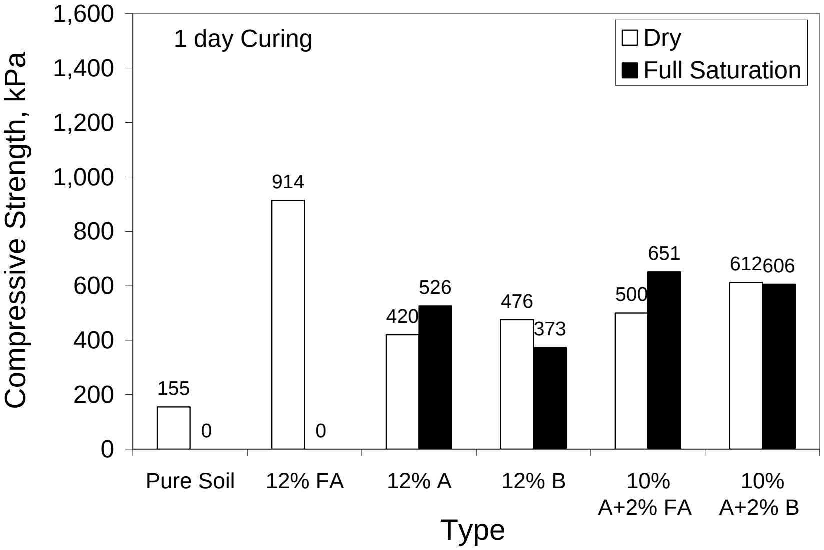


**Figure 21. UCS test results for dry and fully saturated specimens after one day curing**

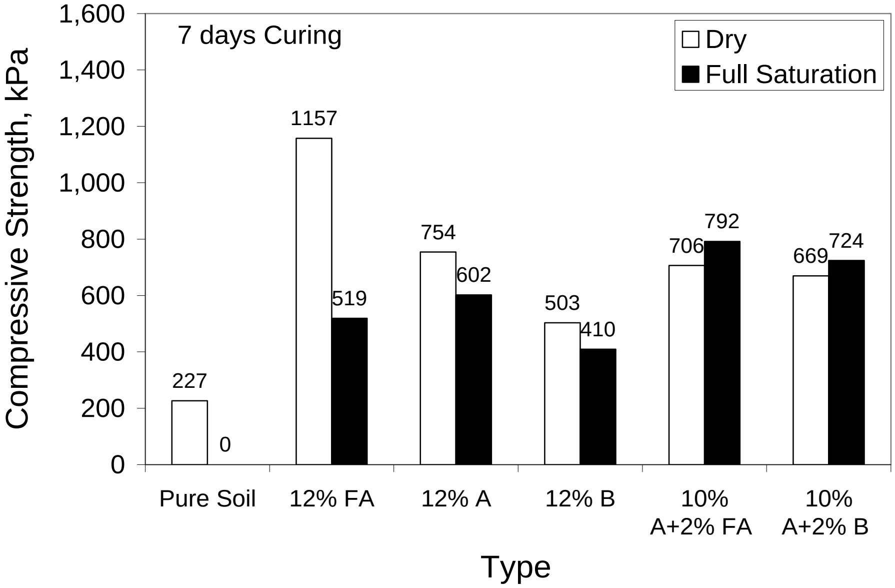


**Figure 22. UCS test results for dry and fully saturated specimens after seven days curing**

Quantitative assessments of the degree to which additives improved strength and moisture resistance were made using the following equations:

$$SI,\% = \frac{SAD - SCD}{SCD} \times 100\,, \tag{1}$$

where *SI* is a percent strength improvement under "dry" conditions, *SAD* is the average strength of additive-treated soil specimen under "dry" conditions, and *SCD* is the average strength of the control (pure soil) specimen under "dry" conditions.

$$MR,\% = \frac{SW - SD}{SD} \times 100\,, \tag{2}$$

where *MR* is percent moisture resistance, *SW* is average strength of specimen under "dry" conditions, and *SD* is the average strength of specimen under "wet" conditions. For this experiment, significant strength improvement was defined as more than 100 percent of the SI value and effective moisture resistance was defined as less than 50 percent of the MR value.

The SI values are summarized in Table 10. The SI values of fly ash–treated soils after one and seven days curing were more than 400%, while the others ranged from about 100% to 300%. These results indicate that all the additives used in this study could significantly improve the UCS of the pure soil.

**Table 10. SI of additive-treated soils compared to control (pure soil)**

| | Strength improvement (SI), % | | | | |
|---|---|---|---|---|---|
| Curing period | Fly Ash | Co-product A | Co-product B | Co-product A + Fly Ash | Co-products A + B |
| 1 day | 489 | 171 | 206 | 222 | 294 |
| 7 days | 411 | 233 | 122 | 212 | 195 |

Table 11 presents the moisture resistance of control (pure soil) and additive-treated soils. The strengths of the control specimens were reduced by 93% and 100% when tested under half and full saturation. The 100% of the MR value indicated that the specimen disintegrated in "wet" pre-conditioning before the UCS test. Fly ash–treated soil specimens met the effective moisture resistance criterion (less than 50% of the MR value) only under the half-saturation condition. In addition, fly ash–treated specimens with one day of curing disintegrated during full-saturation conditioning. Co-products–treated soil specimens met the effective moisture resistance criterion under all of the tested conditions. In particular, the strength of co-product A–treated specimens with one day of curing increased under "wet" conditioning (half and full saturation). Combined additives not only met the effective moisture resistance criterion but also improved the strength of the soil. These results indicate that BCPs containing sulfur-free lignin provide significant resistance to moisture degradation for clay soil. This finding is similar to ERDC's finding that lignosulfonate provides excellent moisture resistance for clay soils (Tingle and Santoni 2003).

**Table 11. MR of control (pure soil) and additive-treated soils**

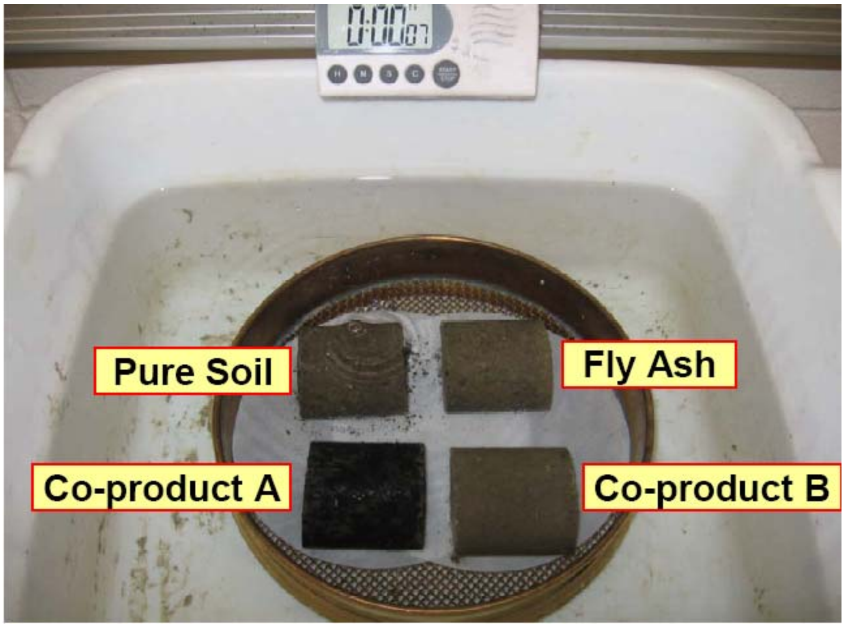
| | | Moisture resistance (MR), % | | | | | |
|---|---|---|---|---|---|---|---|
| Wet condition | Curing period | Control (pure soil) | Fly Ash | Co-product A | Co-product B | Co-product A + Fly Ash | Co-products A + B |
| Half saturation | 1 day | 93 | 28 | -71 [a] | 2 | N/A [b] | N/A |
| Full saturation | 1 day | 100 | 100 | -25 | 22 | -30 | 1 |
| | 7 days | 100 | 55 | 20 | 19 | -12 | -8 |

a. strength increase.
b. not available.

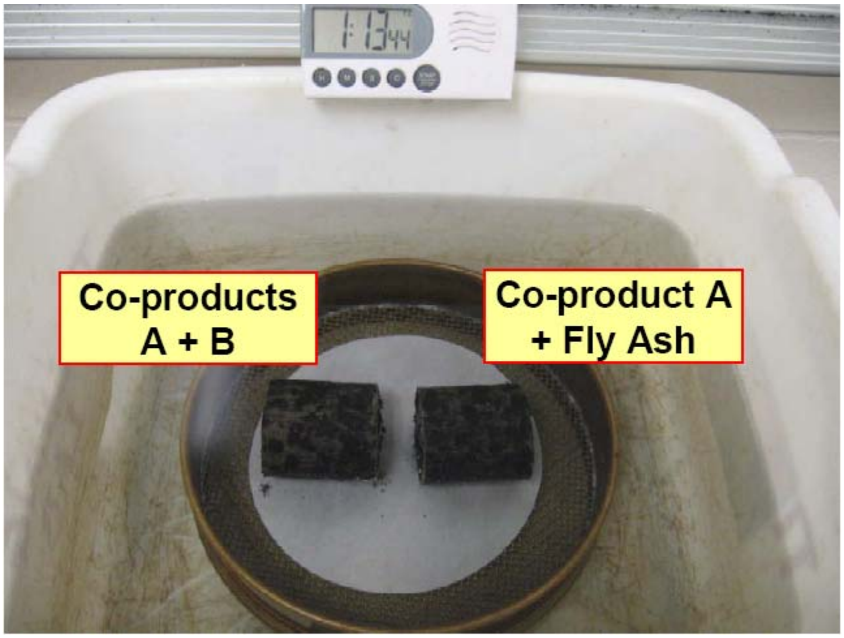
## Soaking Test and Results

Apart from the UCS tests, the compacted specimens of each mixture after the one-day curing period were subjected to so-called soaking tests. The objective of these tests was to evaluate the long-term moisture susceptibility of specimens treated with and without additives and to determine when specimens disintegrated due to water. Specimens were fully soaked in water, as seen in Figure 23. Two test sets of specimens were prepared for these tests. Test set 1 included untreated soil (pure soil), 12% of fly ash–treated soil, 12% of co-product A–treated soil, and 12% of co-product B–treated soil. Test set 2 included 10% of co-product A with 2% of fly ash and

10% of co-product A with 2% of co-product B. The failures of specimens were observed seven days after soaking.



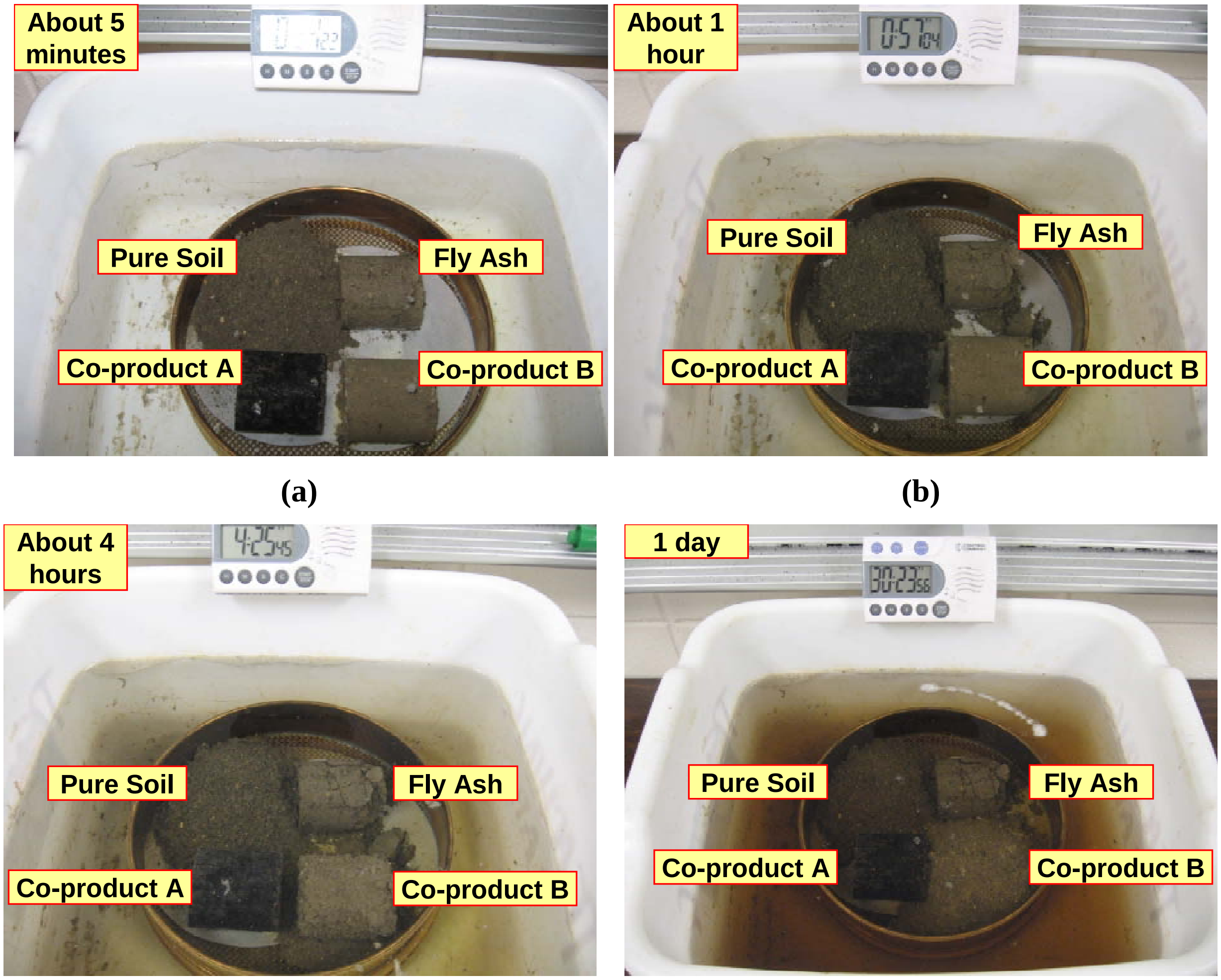
**(a)**



**(b)**

**Figure 1. Soaking tests: (a) test set 1 and (b) test set 2**

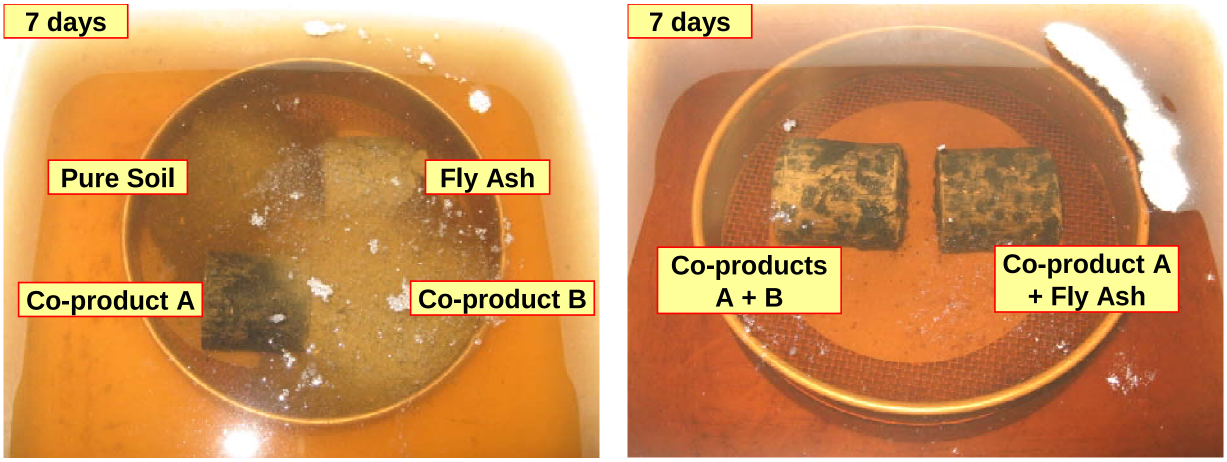
Figure 24 presents soaking test results for test set 1 (pure soil, fly ash, co-product A, and co-product B) and test set 2 (co-product A and fly ash, co-product A and co-product B). Figure 24(a) shows that the pure soil specimen completely disintegrated about five minutes after the specimen was placed in the water. Figure 24(b) displays how the fly ash–treated soil specimen begins to disintegrate about one hour after soaking. As seen in Figures 24(c) and (d), co-product B–treated soil specimens began to disintegrate approximately four hours after soaking and completely disintegrated one day after soaking. However, the deterioration of the specimens after seven days of soaking did not occur in soil specimens treated with co-product A, co-product A and fly ash, and co-products A and B (see Figures 24[e] and [f]). The specimens that didn't deteriorate after the soaking tests were then subjected to UCS testing. The UCS of these specimens shown in Figure 25 are 122 kPa for co-product A, 128 kPa for co-products A and B, and 152 kPa for co-product A and fly ash. These soaking test results demonstrate that the BCP A–treated soil specimens don't deteriorate, even after long-term moisture exposure, and have some degree of strength after prolonged moisture exposure. These results indicate that BCP A can provide excellent waterproofing for clay soils.



(a) (b)

(c) (d)

**Figure 24. Soaking test results for specimens: (a) five minutes for set 1, (b) one hour for set 1, (c) four hours for set 1, (d) one day for set 1**



(e) (f)

**Figure 24 (Continued). Soaking test results for specimens: (e) seven days for set 1, and (f) seven days for set 2**

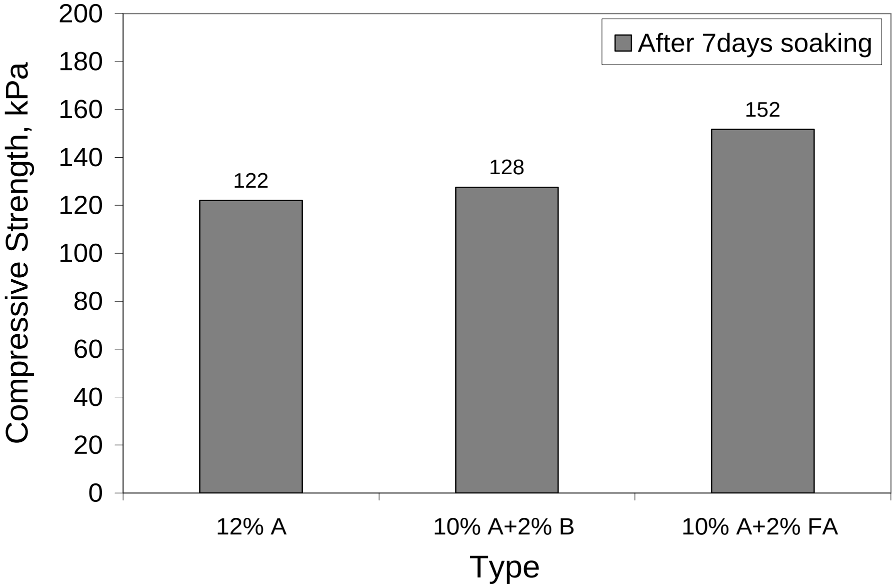


**Figure 25. UCS test results for specimens that did not deteriorate by the end of soaking test**

# CONCLUSIONS

## Project Summary

Soil stabilization is a common practice for improving road soil engineering properties in the construction of both paved and unpaved roads. This study investigated the utilization of BCPs containing lignin in soil stabilization as a new application area for BCPs. Laboratory tests were conducted to evaluate the performance and the moisture susceptibility of two types of BCP-treated soil samples compared to untreated and traditional stabilizer-treated (fly ash) soil samples. The two types of BCPs investigated were (1) a liquid type BCP with higher lignin content (co-product A) and (2) a powder type BCP with lower lignin content (co-product B). Various additive combinations (co-product A + fly ash, co-products A + B) were also evaluated as alternatives to the use of stand-alone co-products.

The experimental tests for performance evaluation included the UCS test as a strength property test and the Atterberg limits and standard Proctor compaction tests as engineering properties tests. Additive contents, moisture contents, and curing periods were incorporated as variables into the strength property test factorial. The experimental program for moisture susceptibility evaluation consisted of two types of tests, which were the UCS tests after “dry” and “wet” conditioning procedures and the visual observations of soaked specimens (so-called soaking test). The UCS test was applied to dry and wet specimens to evaluate strength loss of additive-treated specimens due to moisture. Each specimen was also fully soaked in water and was examined to see whether specimens could fail due to moisture during specific periods.

## Research Findings

Based on the experimental studies, the following findings can be succinctly summarized in the context of utilizing BCPs in soil stabilization:

- The investigated BCPs are promising materials to improve the strength of the Iowa Class 10 soil classified to CL or A-6(8).
- BCPs containing sulfur-free lignin provide excellent resistance to moisture degradation for the Iowa Class 10 soil classified to CL or A-6(8).
- Co-product A is more effective in improving strength under dry conditions, while co-product B is more effective in improving strength under wet conditions.
- The UCS of co-product–treated soil samples increases with the increase in content of co-products. A high increase in UCS occurred with 12% of co-product A in all cases.
- Co-product A with higher lignin content is more effective in providing moisture resistance than co-product B with lower lignin content and fly ash.
- The additive combinations of 10% of co-product A + 2% of fly ash and 10% co-product A + 2% co-product B under dry (OMC-4%) side provided strength comparable to that of the fly ash treatment.
- The additive combinations of co-product A + fly ash under OMC+4% possess strengths

similar to that of fly ash treatment.
- Curing periods have more influence on strength gain of soil treated by co-product A than by co-product B.
- Additive combinations of 10% of co-product A + 2% of fly ash and 10% of co-product A + 2% of co-product B provided moisture resistance comparable to co-product A.

## Recommendations

The addition of lignin-containing co-products to natural soil can benefit the overall soil engineering properties in the construction of both paved and unpaved roads. In general, co-products with higher lignin content (co-product A) are more effective in providing strength improvement and moisture resistance. Co-product A produced from the fast pyrolysis procedure is sometimes called a pyrolysis liquid. The water contents of pyrolysis liquid vary over a wide range (15% to 30%), depending on the type of lignocellulosic biomass as feedstock and the processing conditions. Even though the low viscosity of co-products due to water might easily spread on natural soil and more homogenously mix with natural soil, it is obvious that water in co-products can inhibit soil binding. It is recommended to dry the co-products to remove the initial water in the co-products before employing them in field applications as soil stabilizers. By heating, the viscosity of the dried co-product can easily be reduced to make it workable and thus achieve a continuously homogenous blend of soil and co-product for successful field applications. The other alternative is to dry natural soil before adding the co-products without drying and then to make the total moisture content (existing in soil plus added with co-product) of stabilized soil mixture at the appropriate level.

## Final Remarks and Future Work

Utilization of BCPs as a stabilization material for soil appears to be one of many viable answers to the profitability of the bio-based products and the bioenergy business. Because there is much more BCP that is disposed of rather than utilized, making more productive use of BCPs would have considerable benefits for sustainable development. BCPs used in this experiment demonstrated excellent potential for stabilizing low-quality materials for use in low- and high-volume roads. These products could be used to stabilize existing subgrade materials to provide a stable working platform and to improve strength of undesirable soil materials for use as the load-bearing layer within the pavement system. From an economic perspective, the change to renewable energy from fossil-based energy could result in less production and higher cost of fly ash, which is a byproduct in coal-fired power plants. However, this change could also result in more production and lower cost of BCPs in comparison to traditional soil stabilizers. While fly ash has the potential to leach heavy metals that could contaminate the soil (FHWA 2005), the BCPs could be beneficially used without adversely impacting the environment because the feedstock of biofuels and co-products is natural biomass and is therefore considered biodegradable.

Future research is needed to evaluate the freeze-thaw durability and as well as resilient modulus characterization of these BCP-treated soils. Because the natural soil exposed to climate changes will generally experience alteration of engineering properties, the impact of freeze-thaw cycles on co-product-treated soils should be investigated. Resilient modulus is an essential property for structural design/analysis of pavements. By characterizing the resilient modulus, co-product-treated soils could be used in mechanistic-based pavement design applications. In addition, the long-term performance of these BCPs should be evaluated under actual field conditions and traffic loadings. It is proposed that a second phase of this research be carried out with the objective of evaluating the freeze-thaw durability and long-term performance of BCP-treated soils under actual field conditions. Considering that only one soil type was investigated in this study, any future work should also investigate the use of lignin-based BCPs for a variety of soils that are being used as pavement subgrade and base layers in highway infrastructure systems.

Considering the wide range of pavement-related applications in which modified lignins have already been used, such as concrete admixtures, dust suppressants, and potentially pavement base layer treatment agents and joint and crack sealants, the utilization of BCPs containing lignin in these applications should be investigated in the future. These innovative uses of BCP in pavement-related applications could not only provide additional revenue streams to improve the economics of biorefineries but could also serve to establish green road infrastructures.

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