

2012

# Moisture susceptibility of subgrade soils stabilized by lignin-based renewable energy coproduct

Sunghwan Kim

*Iowa State University*, [sunghwan@iastate.edu](mailto:sunghwan@iastate.edu)

Kasthurirangan Gopalakrishnan

*Iowa State University*, [rangan@iastate.edu](mailto:rangan@iastate.edu)

Halil Ceylan

*Iowa State University*, [hceylan@iastate.edu](mailto:hceylan@iastate.edu)

Follow this and additional works at: [http://lib.dr.iastate.edu/ccee\\_pubs](http://lib.dr.iastate.edu/ccee_pubs)



Part of the [Civil Engineering Commons](#), and the [Environmental Engineering Commons](#)

The complete bibliographic information for this item can be found at [http://lib.dr.iastate.edu/ccee\\_pubs/53](http://lib.dr.iastate.edu/ccee_pubs/53). For information on how to cite this item, please visit <http://lib.dr.iastate.edu/howtocite.html>.

---

This Article is brought to you for free and open access by the Civil, Construction and Environmental Engineering at Iowa State University Digital Repository. It has been accepted for inclusion in Civil, Construction and Environmental Engineering Publications by an authorized administrator of Iowa State University Digital Repository. For more information, please contact [digirep@iastate.edu](mailto:digirep@iastate.edu).

---

# Moisture susceptibility of subgrade soils stabilized by lignin-based renewable energy coproduct

## Abstract

Sustainable use of biomass as a renewable source of energy can be an alternative solution to the cost of fossil-based energy and global warming. Production of biofuel from plant biomass results not only in bio-based energy, but also in coproducts containing lignin, modified lignin, and lignin derivatives. This paper discusses the moisture susceptibility of subgrade soil stabilized by bio-based energy coproducts containing lignin, with the aim of establishing a new application for bio-based energy coproducts in soil stabilization. An experimental test program was conducted to compare the moisture susceptibility of lignin coproduct-treated soils and traditional fly ash stabilizer-treated soil samples. Additive combinations were also evaluated. There were two types of laboratory tests for moisture susceptibility evaluation: (1) unconfined compression strength (UCS) tests after "dry" and "wet" conditioning, and (2) visual observation of soaked specimens. Results indicate that the biofuel coproducts have excellent resistance to moisture degradation for the Iowa Class 10 soil classified as CL by the Unified Soil Classification System and as A-6(8) by AASHTO. In particular, Coproduct A with higher lignin content is more effective in providing moisture resistance than Coproduct B with lower lignin content as well as conventional additive (Class C fly ash). Moisture resistance comparable to that of Coproduct A could be obtained through the use of additive combinations.

## Keywords

biofuel, coproduct, fly ash, lignin, moisture, pavement, renewable energy, soil stabilization, sustainability

## Disciplines

Civil Engineering | Environmental Engineering

## Comments

This is a manuscript of an article from *Journal of Transportation Engineering* 138 (2012): 1283, doi: [10.1061/\(ASCE\)TE.1943-5436.0000097](https://doi.org/10.1061/(ASCE)TE.1943-5436.0000097)

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “*Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.*” ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

# Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Coproduct

Sunghwan Kim<sup>1</sup>, Kasthurirangan Gopalakrishnan<sup>2</sup>, and Halil Ceylan<sup>3</sup>

**Abstract:** Sustainable use of biomass as a renewable source of energy can be an alternative solution to the cost of fossil-based energy and global warming. Production of biofuel from plant biomass results not only in bio-based energy, but also in coproducts containing lignin, modified lignin, and lignin derivatives. This paper discusses the moisture susceptibility of subgrade soil stabilized by bio-based energy coproducts containing lignin, with the aim of establishing a new application for bio-based energy coproducts in soil stabilization. An experimental test program was conducted to compare the moisture susceptibility of lignin coproduct-treated soils and traditional fly ash stabilizer-treated soil samples. Additive combinations were also evaluated. There were two types of laboratory tests for moisture susceptibility evaluation: (1) unconfined compression strength (UCS) tests after “dry” and “wet” conditioning, and (2) visual observation of soaked specimens. Results indicate that the biofuel coproducts have excellent resistance to moisture degradation for the Iowa Class 10 soil classified as CL by the Unified Soil Classification System and as A-6(8) by AASHTO. In particular, Coproduct A with higher lignin content is more effective in providing moisture resistance than Coproduct B with lower lignin content as well as conventional additive (Class C fly ash). Moisture resistance comparable to that of Coproduct A could be obtained through the use of additive combinations (Coproduct A+ Class C fly ash, Coproduct A+ Coproduct B).

**CE Database subject headings:** Renewable energy; Biomass; Soil stabilization; Sustainable development; Fly ash; Pavements; Moisture; Subgrades.

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “*Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.*” *ASCE Journal of Transportation Engineering*, Vol. 138, No. 11, pp 1283-1290.

## **Introduction**

With the increasing cost of fossil-based energy and the problems of global warming, sustainable use of natural resources has been widely accepted as a viable technology for the solution to these problems. Among various natural resources is biomass, in particular, plant biomass, which is considered as a renewable source to produce alternative energy such as biofuel and ethanol to fossil fuels. Bio-based energy produced from plant biomass has several advantages over conventional fossil fuels. Bio-based energy is renewable, environment friendly, provides energy security, and represents a large economic development in the world (Demirbas and Balat 2006). Its disadvantages are the current high costs and large areas of land involved to produce substantial amounts of energy (Hamelinck et al. 2005).

Plant biomass is a lignocellulosic material consisting of cellulose, hemicellulose, and lignin. Plant biomass includes forest residues (bark, sawdust, shavings, etc.) and agricultural residues (sugar cane, cornhusks, bagasse, wheat straw, etc.). It can be converted into bio-based energy through physical and chemical procedures, some of which have been developed and some that are still under development (Kamm and Kamm 2004). In the bio-based energy conversion procedure, plant biomass also produces many different coproducts that have many unexplored uses (Bothast and Schlicher 2005). The types of coproducts produced depend on the biofuel production and coproduct recovery methods, as well as the source of biomass. Among the many different coproducts is a lignin-containing coproduct that has been considered a waste material or low-value coproduct, with its utilization limited predominantly to fuels in the production of octane boosters, biobased products, and chemical products (Stewart 2008). New applications of biomass-derived lignin need to be developed to provide additional revenue streams to improve the economics of bio-based products and the bioenergy business, resulting in more sustainable use of natural resources.

Lignin, representing the third largest fraction of plant biomass, is a large complex polymer of phenylpropane and methoxy groups, a noncarbohydrate polyphenolic substance that encrusts plant cell walls and cements plant cells together. The various industrial product conversion technologies used with plant biomass can modify natural lignin. The modified lignin can be divided into two principal categories: sulfite lignins (lignosulfonates), derived mainly from the paper industry, and sulfur-free lignins, obtained mainly from biofuel or ethanol production [International Lignin Institute (ILI) 2008]. Most lignin-based industrial products in the forms of binder, dispersant, emulsifier, and sequestrant are derived from sulfite lignins (Lignin Institute 2008).

Various studies have also been conducted on the use of sulfite lignin in civil engineering infrastructure applications. Some studies have demonstrated that sulfite lignin is effective in soil stabilization (Nicholls and Davidson 1958), in dust control on unpaved roads (Sinha et al. 1957), as an extender for asphalt paving mixtures (Sundstrom et al. 1983), and as a water-reducing and set-retarding admixture of concrete (Midness et al. 2002). However, inadequate studies have been conducted to examine the use of biofuel-derived sulfur-free lignins for civil engineering infrastructure applications, whereas the use of sulfur-free lignin has recently gained interest as a result of the diversification of biomass processing schemes (Lora and Glasser 2002).

Recent research at Iowa State University (ISU) has investigated the feasibility of using biofuel coproducts in soil stabilization. A preliminary study demonstrated that biofuel coproducts containing lignin can improve subgrade soil strength (Ceylan et al. 2009, 2010). Following this preliminary study, the present study was conducted to determine if those materials could improve

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “*Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.*” ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

the moisture resistance of existing subgrade materials, thereby arresting the deterioration of pavement systems caused by moisture.

### **Lignin as Soil Stabilizer**

Soil stabilization is the process of blending and mixing materials with soil to improve the properties of geotechnical materials (JDAAF 1994). Traditional soil-stabilizing additives or admixtures traditionally used include hydrated lime, Portland cement, and fly ash. Both natural and modified lignin have also been implicated as having a positive role in soil stabilization (Kozan 1955; Nicholls and Davidson 1958; Johnson et al. 2003; Ceylan et al. 2009; Ceylan et al. 2010). Adding lignin to clay soils increases the soil stability by causing dispersion of the clay fraction (Davidson and Handy 1960; Gow et al. 1961). According to Gow et al. (1961), the dispersion of the clay fraction benefits stability of the soil-aggregate mix by: a) plugging voids and consequently improving water-tightness and reducing frost susceptibility, b) eliminating soft spots caused by local concentrations of binder soil, c) filling voids with fines thus increasing density, and d) increasing the effective surface area of the binder fraction which results in greater contribution to strength.

It has been demonstrated that lignin results in better improvements for ground modification compared with nonorganic stabilizers (Palmer et al. 1995). Lignin is also used in combination with other chemicals to achieve soil improvement (Puppala and Hanchanloet 1999). As a soil additive, lignin causes dispersion of the clay fraction of some soils, resulting in an increase in the shear strength of the soil (as a result of particle rearrangement) (Addo et al. 2004). Various studies on lignin as a soil additive have concluded that lignin is primarily a cementing agent (Woods 1960; Ingles and Metcalf 1973; Landon and Williamson 1983).

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 increase resulting from the addition of some commercially available chemical additives. The laboratory methods included unconfined compression (UCS) test (ASTM D2166) and a wet sieve analysis test (ASTM D 422). The testing was performed at three sample-drying conditions, 24-hour air-dried, 24-hour bag cured, and immediate sample testing. The results indicate that each additive tested varies in cohesive strength in the range of 27 – 379 kPa (4 – 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.

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

The Quebec Department of Roads conducted laboratory tests comparing the engineering properties of lignin-treated aggregate with that 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. Also, the increase in strength was found to be nearly linearly proportional

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “*Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.*” ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

to the amount of lignin used. Water absorption tests indicated that water absorbed through capillary action was reduced substantially. Moisture–density relationship tests showed that an increase in the amount of lignin added to the soil increased density and reduced 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,  $\text{CaCl}_2$  and  $\text{MgCl}_2$  at different concentrations. Three different road soil materials with different soil classifications were used. The seven-day air-cured samples exhibited large strength increase 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.

Puppala and Hanchanloet (1999) studied the effect of a new chemical treatment method using a liquid comprising 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 percentage increase in UCS with the SA-44/LS-40 treatment ranged between 30% to more than 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 stability of soils. They also reported that the strength of lignin-treated soil increases rapidly with an increase in the length of air curing duration. Also, lignin is organic material; its presence in the environment can be considered less harmful than that of industrial by-products such as fly-ash, which cause leaching of heavy metals.

U.S. Army Engineer Research and Development Center (ERDC) conducted a laboratory experiment 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). 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 was used as traditional stabilizers to provide a comparison under the same mixing, compaction, and curing conditions. The USC test results of each additive under wet and dry conditions were compared to those of the remaining nontraditional additives, the traditional stabilization results, and a series of control specimens that were not stabilized. These studies found that lignosulfonate provides excellent waterproofing for clay soils and silty-sand. The five percent of lignosulfonate was reported as optimum additive quantity for silty-sand.

In most of previous studies described, natural lignin or sulfite lignin (lignosulfonates) has been utilized. It has been hypothesized that since the ethanol co-product from corn stover fermentation is high in lignin (sulfur-free lignin), which is thought to play a role in stabilizing soil, soil incorporation of the ethanol co-product may help maintain or improve soil structure and stability (Johnson et al. 2004). The utilization of lignin based biofuel co-product 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. Recently, researchers at ISU are investigating the feasibility of this approach. Preliminary results of this research indicate that lignin based biofuel co-products are promising materials to improve strength of the Iowa class 10 soil classified to CL or A-6(8) (Ceylan et al.

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “*Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.*” ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

2009; Ceylan et al. 2010). However, the effect of lignin based biofuel co-products on other geochemical engineering properties of soil such as moisture and freeze/thaw durability should be understood before application of co-product on actual field soil stabilization.

## Materials

### Soil

Natural soils were collected from a new construction site for US-20 in Calhoun county, Iowa, US. The engineering properties of the soil samples are shown in Table 1. The collected soil samples used in this study classified as an A-6(8) soil and CL in accordance with the American Association of State Highway and Transportation Officials (AASHTO) soil classification system and Unified Soil Classification System (USCS), respectively, and as class 10 soil as per Iowa department of transportation (DOT) specification (IA DOT 2008). The class 10 soil is the typical excavated soil including 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 used in construction specified or should be removed.

**Table 1.** Engineering Properties of Soil Investigated

Property	Soil
<i>Classification</i>	
AASHTO (group index)	A-6(8)
USCS group symbol	CL
USCS group name	Sandy lean clay
<i>Grain size distribution</i>	
Gravel (> 4.75 mm),%	7.6
Sand (0.075–4.75 mm),%	40.4
Silt and clay (< 0.075mm),%	51.9
<i>Atterberg limits</i>	
Liquid limit (LL) ,%	39.3
Plasticity limit (PL),%	16.0
Plasticity index (PI),%	23.3
<i>Proctor test</i>	
Optimum moisture content (OMC),%	17.7
Maximum dry unit weight ( $\gamma_{d \max}$ ), kg/m <sup>3</sup> (pcf )	1,691 (105.7)

### Additives

Two types of biofuel co-product containing lignin were used as additives and designated as co-products A and B in this study. The co-product A was obtained from a commercial biomass conversion facility located in Canada. This co-product A 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 wherein plant material (biomass), such as forest residues (bark, sawdust,

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “*Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.*” ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

shavings, etc.) and agricultural residues (sugar cane, cornhusks, bagasse, wheat straw, etc.), are exposed to 400-500 °C in an oxygen free environment (Dynamotive Energy Systems Corporation 2007). Recently, several qualification trial tests of this co-product A for heating the Iowa Capitol Complex were conducted by the State of Iowa Department of Administrative Services-General Services Enterprise (DAS-GSE) in partnership with Dynamotive Energy Systems Corporation and Biogreen Resources (IA DAS 2008). The 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 for use of liquid fuel is not a separate phase because it lowers the viscosity of the fuel.

Coproduct B was obtained from a full-scale, wet-mill, corn-based ethanol plant of Grain Processing Corporation (GPC) in 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.

Ottumwa Class C fly ash was selected as the traditional additive against which to compare biofuel co-products relative performance. The Ottumwa class C fly ash is a coal combustion by-product from Ottumwa Generating Station (OGS) located near Chillicothe, Iowa. This fly ash is commonly used for soil treatment in Iowa.

## **Experimental Program**

The laboratory experimental program was conducted using two types of tests: (1) UCS tests after “dry” and “wet” conditioning, and (2) visual observations of soaked specimens. The stabilization effect of a soil additive is measured in terms of the increase in loadbearing capacity as indicated by UCS (Lane et al. 1984; Palmer et al. 1995; Tingle and Santoni 2003). This study also used UCS testing as the basis for performance characterization. Dry and wet specimens were subjected to UCS tests to evaluate the moisture susceptibility of additive-treated specimens. Specimens were also fully soaked in water and observed to see if they failed because of moisture during specific periods.

### ***Specimens Preparation***

The natural soil collected was dried and broken down into particles that could pass through a No. 4 (4.75 mm) sieve. Additives were also dried to remove the water initially contained in the coproduct. The amounts of water and additive calculated on the basis of the dry weight of the soil were mixed thoroughly to produce a homogenous soil blend. The additives mixed with soil and water were categorized into uncombined additives and combined additives. Uncombined additives were Coproduct A and Coproduct B, biofuel coproducts containing lignin, and fly ash as a traditional soil stabilizer additive. Combined additives were Coproduct A/fly ash Coproduct A/B mixtures.

On the basis of prior experience (Ceylan et al. 2009, 2010), the amounts of additives required were 12% of each uncombined additive, 10% Coproduct A/2% fly ash, and 10% Coproduct A/2% Coproduct B. These amounts of additive provided the strongest soil mixtures. Untreated soil mixtures with no additives, as a control, were also prepared. The target moisture content for all mixtures was optimum moisture content (OMC) of untreated soil determined in accordance with ASTM (2007). Blended soil samples were statically compacted in the cylindrical mold (51 × 51 mm). Compacted specimens were sealed in plastic wrap and then



**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “*Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.*” ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

placed in a temperature-controlled room, where they were allowed to cure at 25°C and 40% relative humidity. Curing periods were 1 and 7 days after sample fabrication for the UCS test.

### ***Unconfined Compression Strength (UCS) Test***

Compacted specimens of each mixture were subjected to dry and wet preconditioning procedures for UCS tests once the designated curing period was complete. Specimens in the dry precondition were tested without water saturation, whereas specimens in the wet precondition were tested after specified water saturation procedures. The U.S. Army ERDC used dry and wet preconditioning procedures for UCS tests to evaluate the stabilization of clay soils and silty sand with nontraditional chemical or liquid stabilizers (Santoni et al. 2002, 2005; Tingle and Santoni 2003). The ERDC reviewed moisture susceptibility tests of several stabilized materials and found 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 moisture susceptibility of stabilized materials. The wet test procedure used in this research was similar to the one developed by ERDC.

The wet test procedure in this research included full saturation and half-saturation of the specimen. Full saturation requires complete immersion of the specimen on its side in a water bath for 1 h. Half-saturation was also conducted because some of specimens were broken in full saturation. Note that full saturation of specimens was not performed in the ERDC study for the same reason. One side of the specimen was soaked in water for 5 min (see Fig. 1).



**Fig. 1.** Example of fly ash treated specimen under half saturation procedure

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “*Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.*” ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

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 to ASTM D2166 (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 2 lists the experimental treatment group combinations evaluated for UCS test during this research.

**Table 2.** Experimental Treatment Group Combinations for UCS Test

Conditioning	Curing period	Additives <sup>a</sup> ,%				
		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 <sup>b</sup>	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

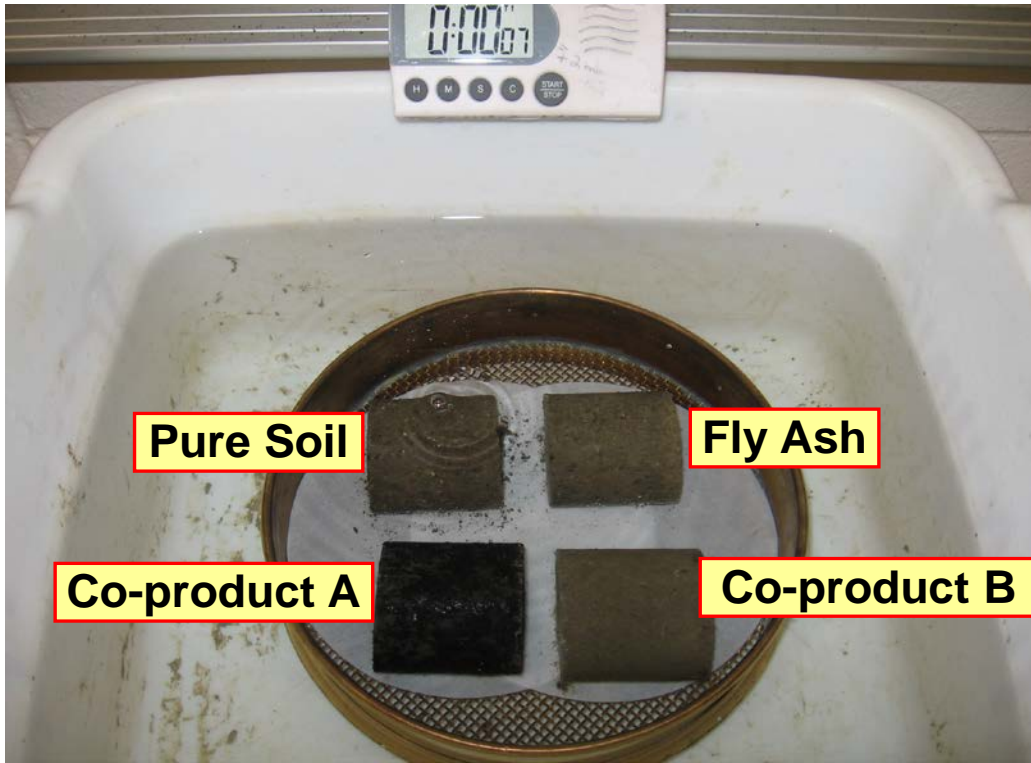
a. Numbers indicate% of additive added by dry soil weight.

b. 10% co-product A and 2% fly ash.

### ***Soaking Test***

As separate UCS tests, compacted specimens of each mixture after 1 day of curing were subjected to so-called soaking tests. The objective of these tests was to evaluate the long-term moisture susceptibility of specimens treated or not treated with additives and to determine when specimens disintegrated because of water. Specimens were fully soaked in water as seen in Fig. 2. Two sets of specimens were prepared for these tests. Test Set 1 included untreated soil (pure soil), 12% fly ash-treated soil, 12% Coproduct A-treated soil, and 12% Coproduct B-treated soil. Test Set 2 included 10% Coproduct A/2% fly ash- and 10% Coproduct A/2% Coproduct B-treated soil. Specimens were observed for failure for 7 days after soaking.

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.” ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.



(a)



(b)

**Fig. 2.** Soaking tests: (a) test set 1; (b) test set 2

## Test Results and Discussions

### UCS Test Results

The results of UCS tests under dry and wet (half- and full saturation) preconditions are shown graphically in Figs. 3–5. After 1 day of curing, specimens are subjected to dry, half-saturation and full-saturation conditions for UCS tests as shown in Figs. 3 and 4. After 7 days of curing, specimens are subjected to dry and full-saturation conditions for UCS tests as shown in Fig. 5. In these figures, A, B, and FA represent Coproduct A, Coproduct B, and fly ash. The 0 value in Figs. 4 and 5 indicate that a specimen disintegrated when exposed to water. The untreated soil specimen was used as the control specimen. Overall, additive-treated soils are in all cases stronger than untreated soils under dry and wet conditions. The fly ash-treated soil test results show the most improvement in UCS under dry conditions. However, fly ash-treated soil specimens disintegrated in the wet precondition (see Fig. 4) or were reduced in strength after the wet precondition compared with coproduct-treated soil specimens (see Figs. 3 and 5). Duration of curing has less influence on the strength gain of soil specimens treated with Coproduct B than on other soil specimens.

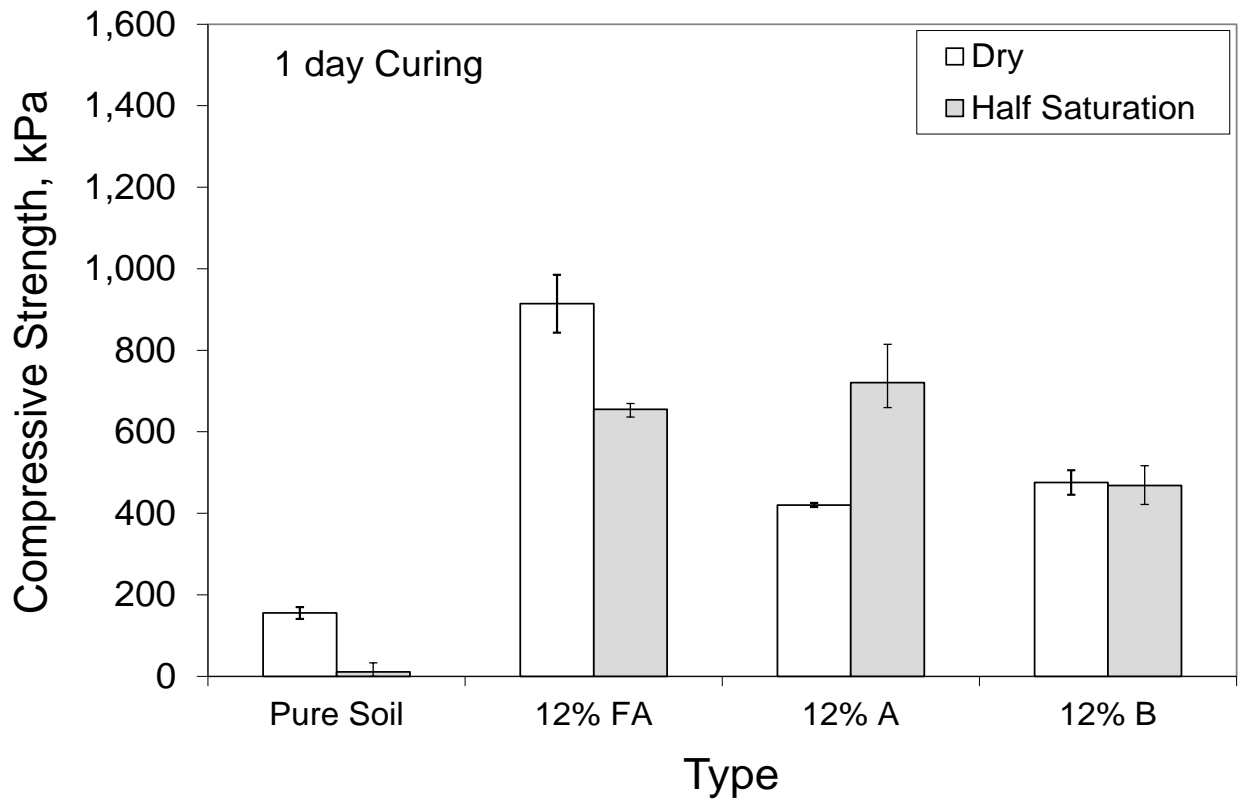


Fig. 3. UCS tests results for dry and half saturated specimens after 1 day curing

Reference to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) "Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product." ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

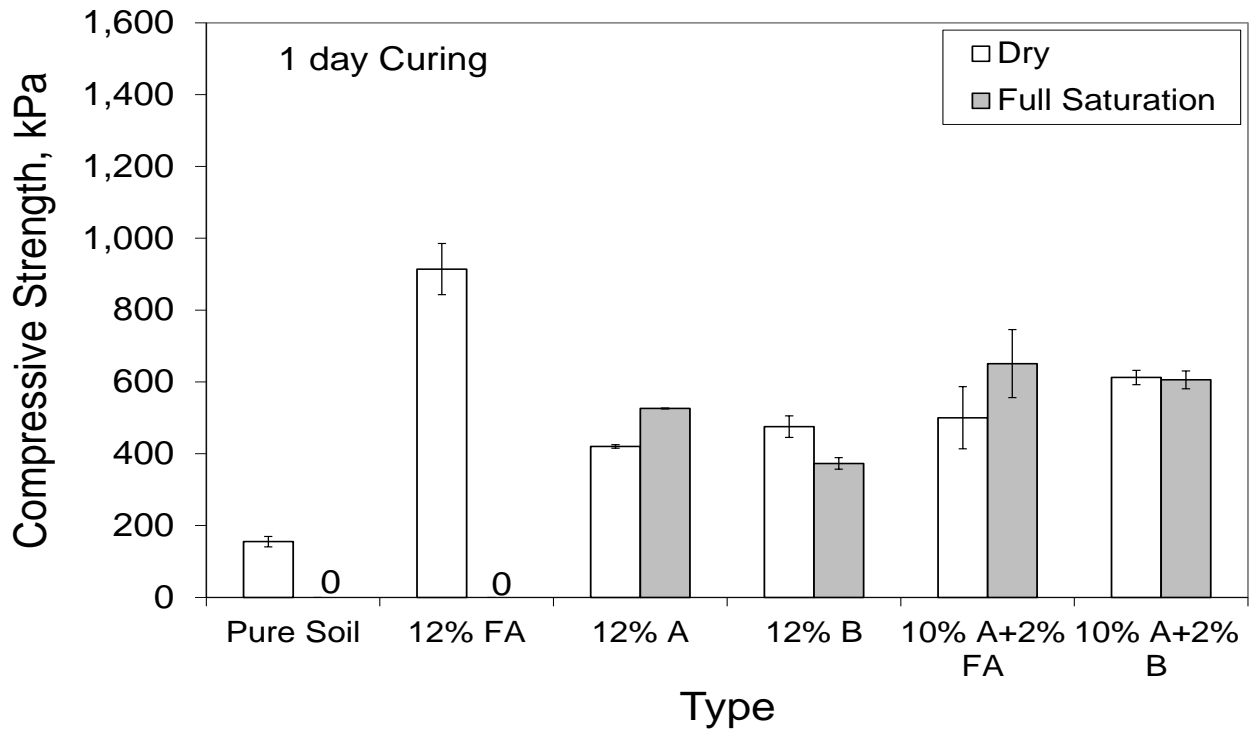


Fig. 4. UCS tests results for dry and full saturated specimens after 1 day curing

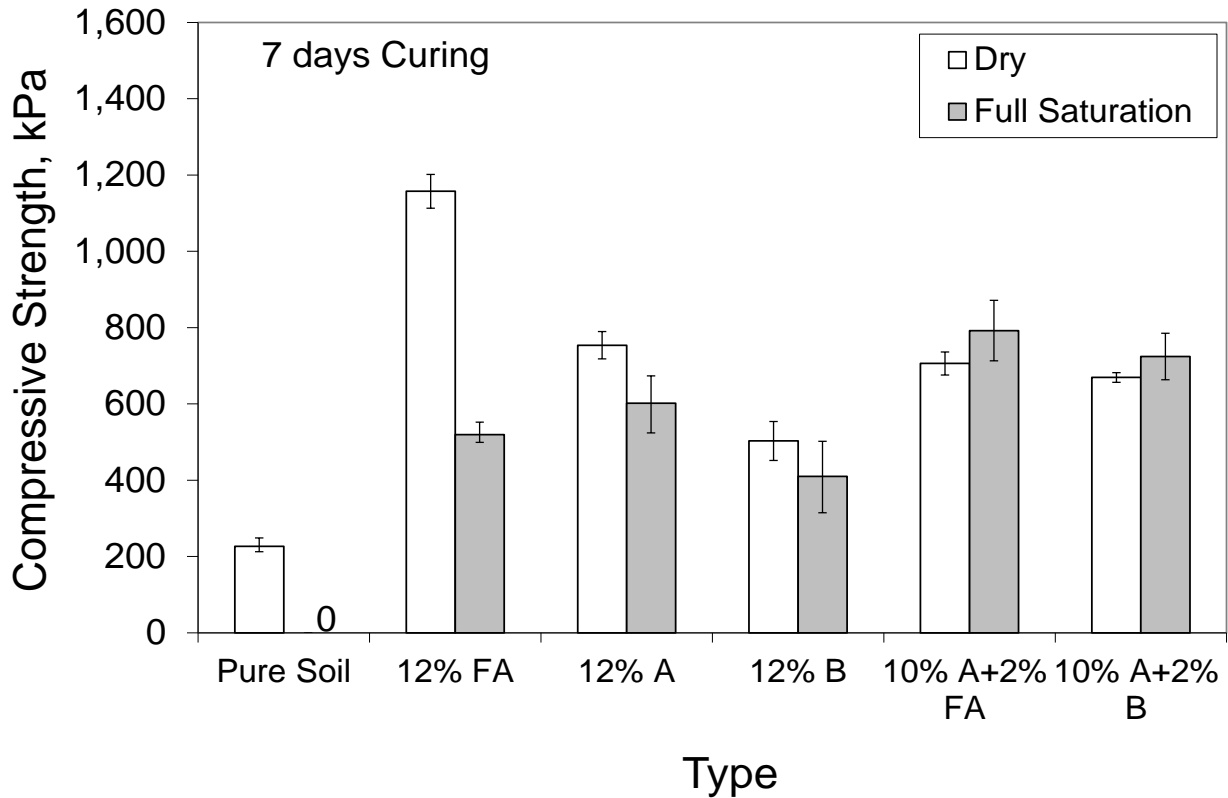


Fig. 5. UCS tests results for dry and full saturated specimens after 7 days curing

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “*Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.*” ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

Quantitative assessments of the degree to which additives improve strength and moisture resistance were made as following equations

$$SI, \% = \frac{SAD - SCD}{SCD} \times 100 \quad (1)$$

where, SI = percent strength improvement under dry condition, SAD = average strength of additive treated soil specimen under dry condition, and SCD = average strength of control (pure soil) specimen under dry condition. Note that strength improvement under wet condition was not calculated since control specimens under wet condition have small strength value (see Fig.3) or disintegrated (see Figs. 4 and 5).

$$MR, \% = \frac{SW - SD}{SD} \times 100 \quad (2)$$

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

The values of SI are summarized in Table 3. The SI values of fly ash treated soils after one and seven days curing are more than 400% while the others ranged from about 100 to 300%. These results indicated that all of additives used in this study could significantly improve the UCS of the pure soil.

**Table 3.** Strength Improvement (SI) of Additive Treated Soils Comparing to Control (Pure Soil)

Curing period	Strength improvement (SI),%				
	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 4 outlines 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 MR value indicated that specimen disintegrated in wet per-condition before UCS test. Fly ash-treated soil specimens met the effective moisture resistance criterion (< 50% of MR value) only under half saturation condition. In addition, fly ash treated specimen with one day curing was disintegrated during full saturation condition. Coproducts-treated soil specimens met the effective moisture resistance criterion under all the tested conditions. Especially, the strength of co-products A treated specimen with one day curing increase under wet condition (half and full saturation). Combined additives not only met the effective moisture resistance criterion but also improved strength of soil. These results indicated that biofuel co-products containing sulfur-free lignin provided significant resistance to moisture degradation for clay soil. This is similar to ERDC study finding that lignosulfonate provided excellent moisture resistance for clay soils (Tingle and Santoni 2003).

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.” ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

**Table 4.** Moisture Resistance (MR) of Control (Pure Soil) and Additive Treated Soils

Wet condition	Curing period	Moisture resistance (MR),%					
		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 <sup>c</sup>	2	N/A <sup>d</sup>	N/A
Full saturation	1 day	100	100	-25	22	-30	1
	7 days	100	55	20	19	-12	-8

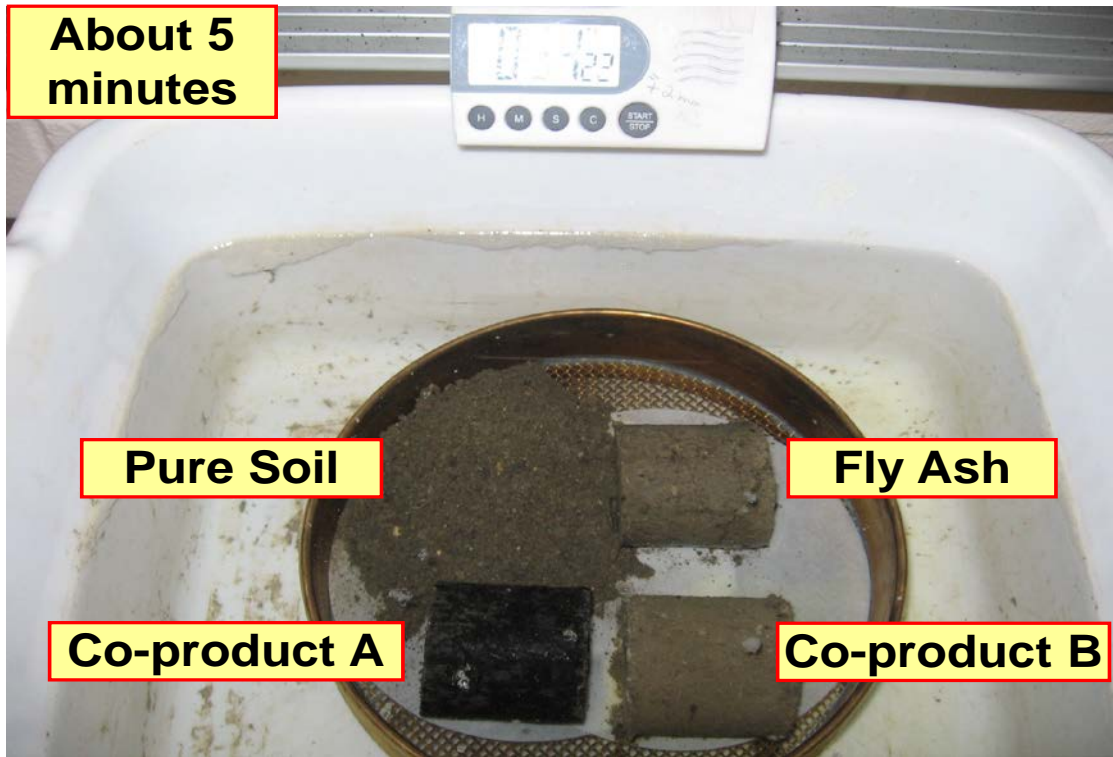
c. Strength increase.

d. Not available.

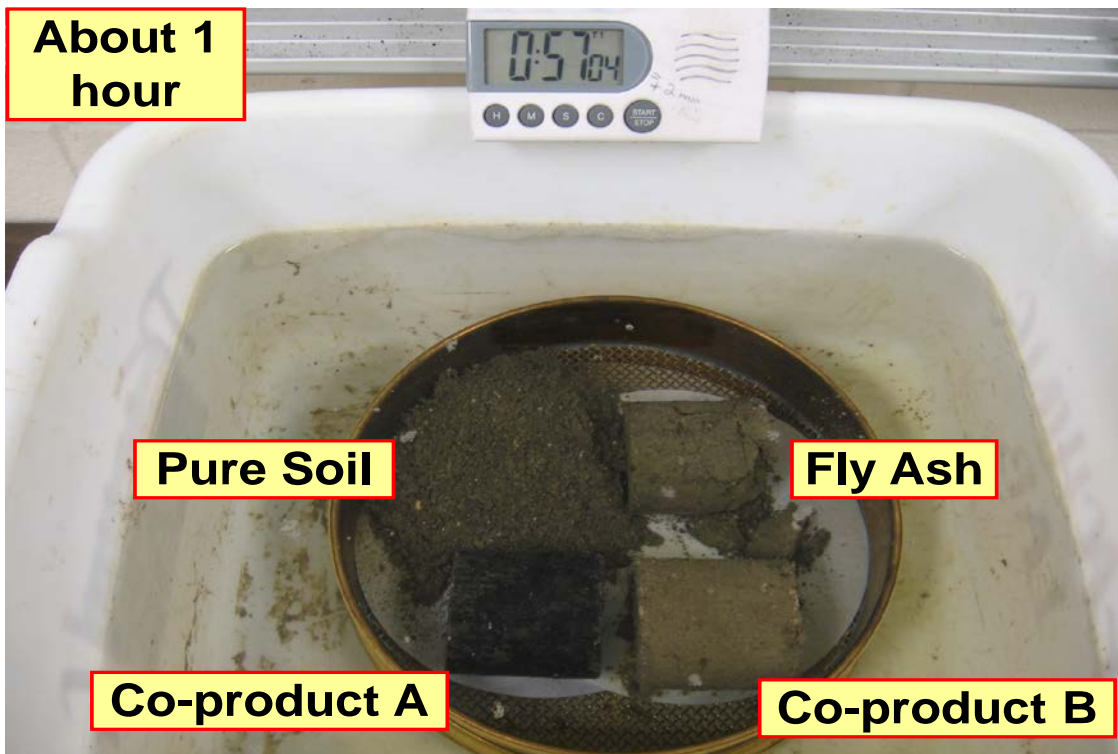
### ***Soaking Test Results***

Fig. 6 illustrates 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). Fig. 6 (a) show that pure soil specimen completely disintegrated at about 5 minutes after specimen was placed in the water. Fig. 6 (b) presented that fly ash treated soil specimen began to disintegrate at about one hour after soaking. As seen Fig. 6 (c) and (d), the co-product B treated soil specimen began to disintegrate at about 4 hours after soaking and completely disintegrated at about one day after soaking. However, the deterioration of the specimens after seven days of soaking did not occur in soil specimens treated with the co-product A, co-product A + fly ash and co-products A + B combinations (see Fig. 6 (e) and (f)). The specimens that didn't deteriorate at the conclusion of the soaking tests were then subjected to UCS testing. The UCS of these specimens, as shown in Fig. 7, are 122 kPa for co-product A, 128 kPa for co-products A + B, and 152 kPa for co-product A + fly ash combinations. These soaking test results demonstrated that the biofuel co-product A treated soil specimens did not deteriorate even after long-term moisture exposure as well as had some degree of strength after an extended period of soaking. These results indicate that biofuel co-product A can provide excellent waterproofing for clay soil.

Reference to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) "Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product." ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.



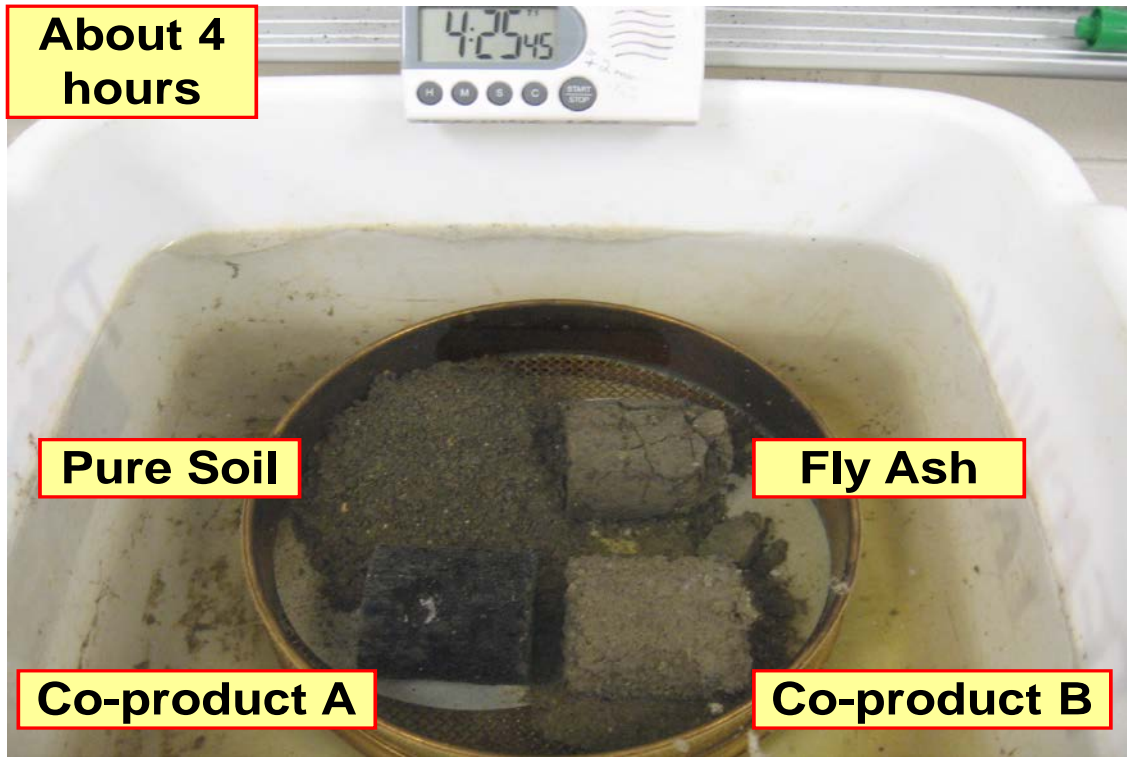
(a)



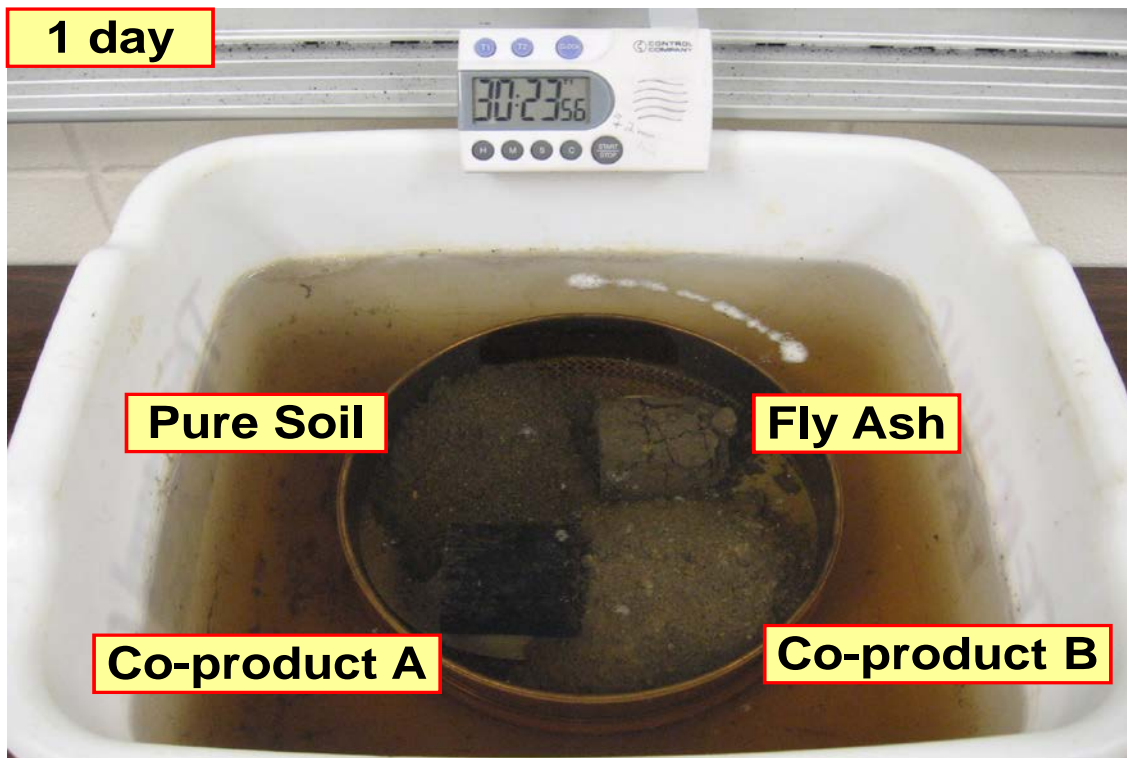
(b)



Reference to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) "Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product." ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

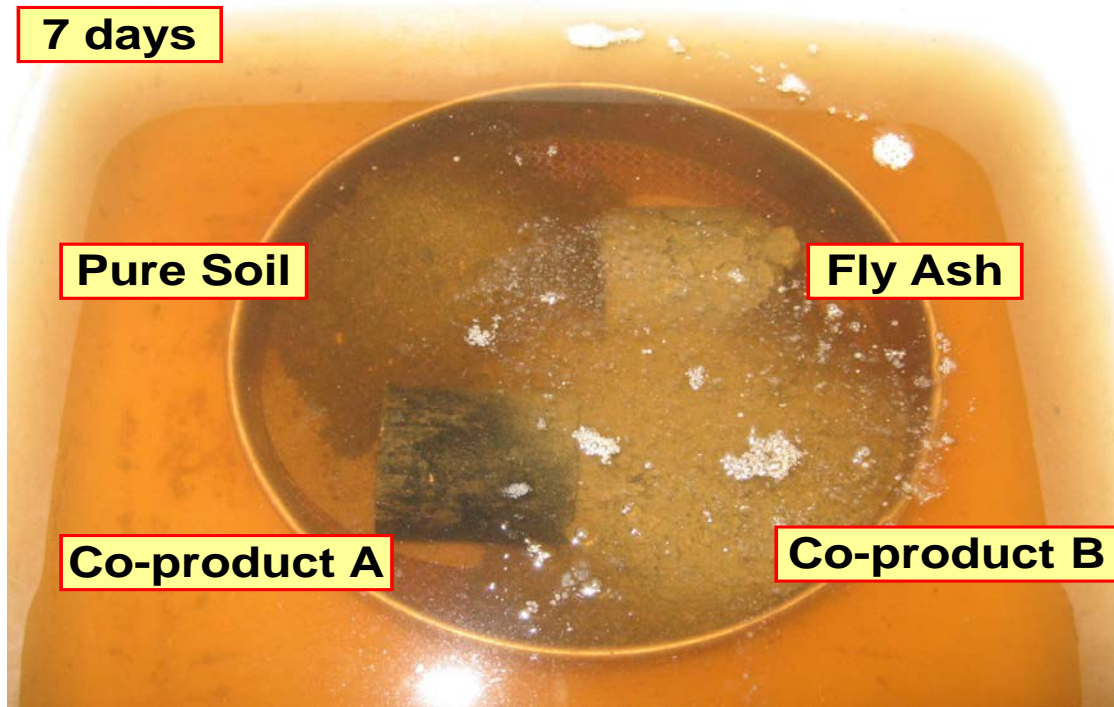


(c)

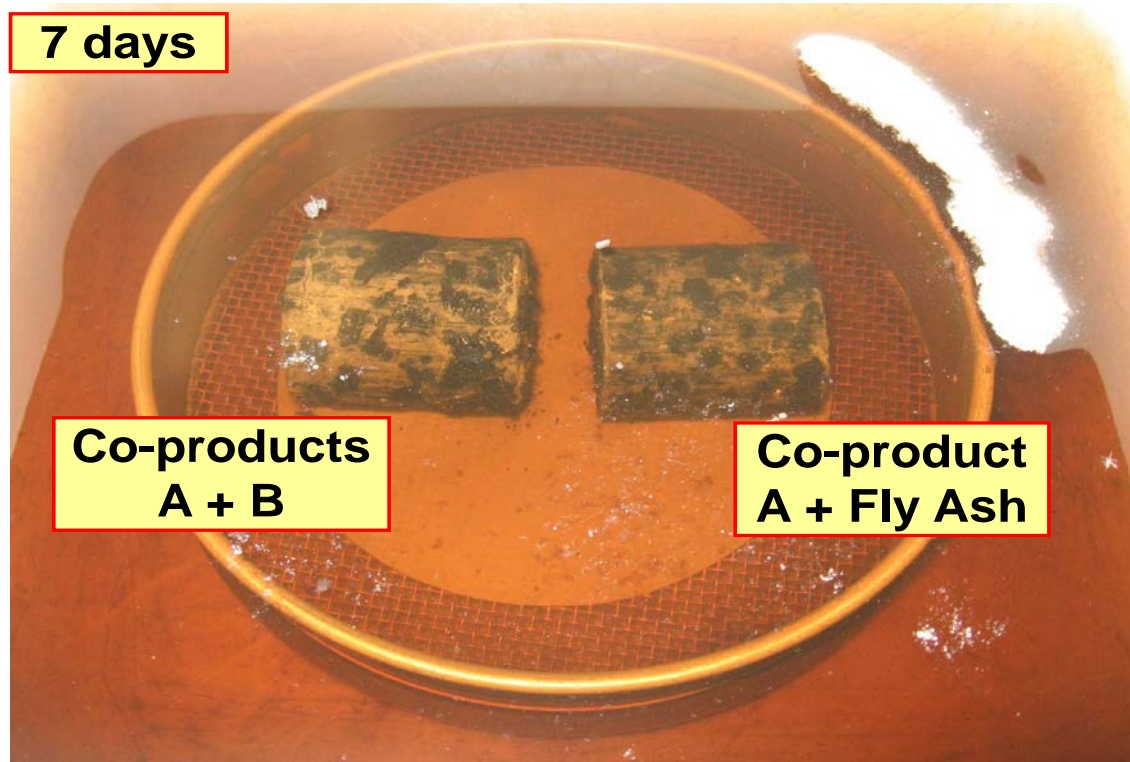


(d)

Reference to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) "Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product." ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.



(e)



(f)

**Fig. 6.** Soaking test results for specimens: (a) 5 min for Test Set 1; (b) 1 h for Test Set 1; (c) 4 h for Test Set 1; (d) 1 day for Test Set 1; (e) 7 days for Test Set 1; (f) 7 days for Test Set

Reference to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) "Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product." ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

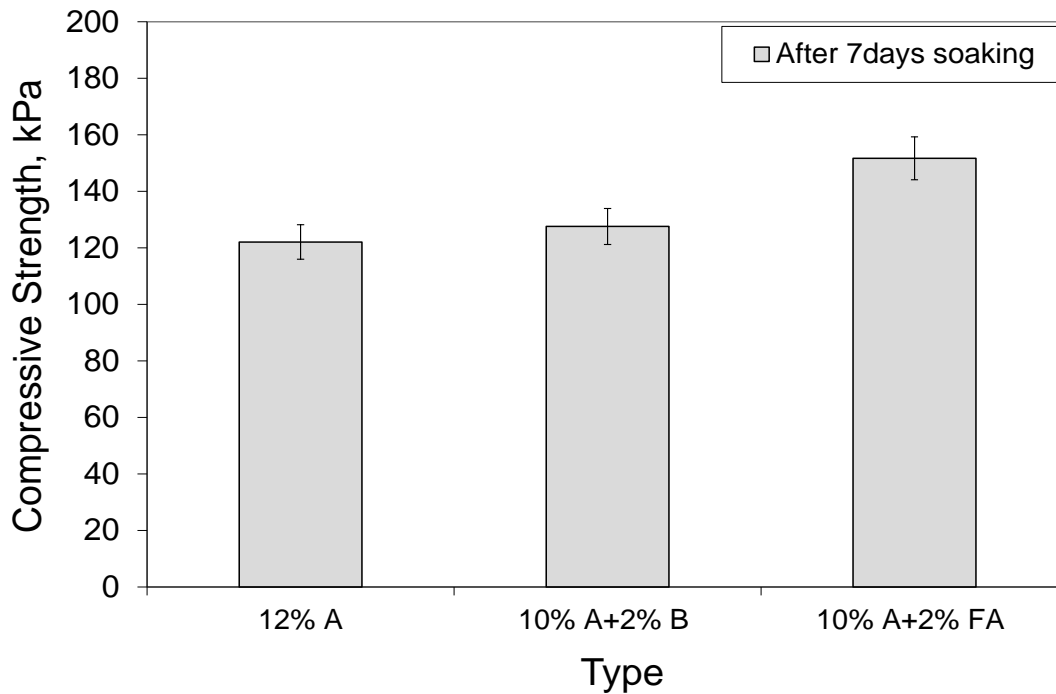


Fig. 7. UCS tests results for specimens without deterioration after soaking test

### Summary and Conclusions

This study investigated the moisture susceptibility of subgrade soils stabilized by a bio-based energy co-product containing lignin. A Laboratory experimental test program was conducted to compare the moisture susceptibility properties of two types of biofuel co-product treated soil samples with untreated and conventional fly ash treated soil samples. Two types of biofuel co-products investigated are: (1) a liquid type co-product with higher lignin content (co-product A), and (2) a powder type co-product with lower lignin content (co-product B). Additive combinations (co-product A + fly ash, co-products A + B) were also evaluated. The experimental program consisted of two types of tests, which were the unconfined compression strength (UCS) tests after "dry" and "wet" conditioning procedure and the visual observations of soaked specimens (so-called soaking tests). The UCS test was conducted on dry and wet specimens to evaluate strength loss of additive treated specimens due to moisture. Each specimen was also fully soaked in water to examine if specimens would fail in the presence of moisture. Based on the experimental studies, the following conclusions are drawn on the effect of bio-based energy co-product containing lignin on moisture susceptibility of subgrade soils:

- Biofuel co-products containing sulfur-free lignin provide excellent resistance to moisture degradation for the Iowa class 10 soil classified as USCS CL and AASHTO A-6(8).
- 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.
- Additive combinations of 10% co-product A + 2 % fly ash and 10% co-product A + 2% co-product B provide moisture resistance comparable to having only co-product A.
-

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “*Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.*” *ASCE Journal of Transportation Engineering*, Vol. 138, No. 11, pp 1283-1290.

Utilization of biofuel coproduct as a stabilization material for soil appears to be technically viable for clay subgrade stabilization. Because much more biofuel coproduct is disposed of rather than used, making more productive use of biofuel coproducts would have considerable benefits for sustainable development. Biofuel coproducts used in this experiment demonstrated excellent potential for improving moisture resistance of low-quality materials for use in low-volume roads. These products could be used to improve the moisture resistance of existing subgrade materials, thereby arresting the deterioration of pavement systems. 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 by-product in coal-fired power plants. However, this change could also result in more production and lower cost of biofuel coproduct compared with traditional soil stabilizers. The lignin in biofuel coproduct could also be beneficially used without adversely impacting the environment, whereas fly ash has the potential for leaching of heavy metals. Recommendations for future work include studying the freeze/thaw durability, resilient modulus characterization, and long-term performance of these biofuel coproducts in the context of a new application of bio-based energy coproduct in soil stabilization.

### **Acknowledgements**

The authors gratefully acknowledge the Iowa Highway Research Board (IHRB), Grow Iowa Values Fund (GIVF) seed grant program, and Iowa State University (ISU) for supporting this study. The authors are also grateful to Dynamotive Energy Systems (Canada) and Grain Processing Corporation (Iowa) for supplying the experimental bio-oil and ethanol by-product investigated in this study. The authors would like to thank Shiyun Wang in his assistance for conducting some aspects of the laboratory tests. The contents of this paper reflect the views of the authors who are responsible for the facts and accuracy of the data presented within. The contents do not necessarily reflect the official views and policies of the IHRB and ISU. This paper does not constitute a standard, specification, or regulation.

### **References**

- Addo, J. Q., Sanders, T. G., and Chenard, M. (2004). *Road dust suppression: effect on maintenance stability, safety and the environment phases 1-3*, Mountain-Plains Consortium (MPC) Report No. 04-156, Fargo, ND.
- ASTM D698. (2007). “Standard test method for laboratory compaction characteristics of soils using standard effort (12,400 ft-lbf/ft<sup>3</sup> (600 kN-m/m<sup>3</sup>)).” *Annual Book of ASTM standards*, ASTM International, West Conshohocken, Pennsylvania.
- ASTM D2166. (2006) “Standard test method for unconfined compressive strength of cohesive soil.” *Annual book of ASTM standards*, ASTM International, West Conshohocken, Pennsylvania.
- Bothast, R. J. and Schlicher, M. A. (2005). “Biotechnological processes for conversion of corn into ethanol.” *Applied Microbiology and Biotechnology*, 67 (1), 19-25.
- Ceylan, H., Gopalakrishnan, K., and Kim, S. (2009). “Use of bio-oil for pavement subgrade soil stabilization.” *Proceedings of 2009 Mid-Continent Transportation Research Symposium* (CD-ROM), InTrans, Iowa State University, Ames, IA.

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “*Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.*” *ASCE Journal of Transportation Engineering*, Vol. 138, No. 11, pp 1283-1290.

- Ceylan, H., Gopalakrishnan, K., and Kim, S. (2010). “Soil Stabilizer Derived from Sustainable Energy Co-Product.” *Proceedings of 89th Transportation Research Board Annual Meeting (DVD)*, National Research Council, National Academy of Science, Washington, DC.
- Davidson, D. T., and Handy, R. L. (1960). “Section 21: soil stabilization.” *Highway engineering handbook*, K. B. Wood, ed., McGraw-Hill.
- Demirbas, M. F., and Balat, M. (2006). “Recent advances on the production and utilization trends of bio-fuels: a global perspective.” *Energy Conservation and Management*, 47, 2371-2381.
- Demirel, T., and Davidson, D. T. (1960). “Stabilization of a calcareous loess with calcium lignosulfonate and aluminum sulfate.” *Soil stabilization with chemicals.*, Joint Publication – Bulletin 193 of the Iowa Engineering Experiment Station and Bulletin 22 of the Iowa Highway Research Board, Davidson, D. T. and Associates (Eds.), Ames, IA, 206-221.
- Dynamotive Energy Systems Corporation. (2007). *Dynamotive biooil information book*, Dynamotive Energy Systems Corporation, Vancouver, Canada.
- Gow, A. J., Davidson, D. T., and Sheeler, J. B. (1961). “Relative effects of chlorides, lignosulfonates and molasses on properties of a soil-aggregate mix.” *Highway Research Board Bulletin*, 282.
- Grain Processing Corporation (GPC). (2009). Grain Processing Corporation, Muscatine, Iowa, <<http://www.grainprocessing.com/>> (March 5, 2009).
- Hamelinck, C. N., Suurs, R. A. A., and Faaij, A. P. C. (2005). “International bioenergy transport costs and energy balance.” *Biomass and Bioenergy*, 29, 114–134.
- Hoover, J. M., Davidson, D. T., Plunkett, J. J., and Monoriti, E. J. (1959). “Soil-organic cationic chemical-lignin stabilization.” *Highway Research Board Bulletin*, 241, 1-13.
- Hurtubise, J. E. (1953). “Soil stabilization with lignosol.” *Canadian Chemical Process*, 37, 58-61.
- ILI. (2008). “About Lignin”, the International Lignin Institute, <<http://www.ili-lignin.com/aboutus.php>> (Aug. 1, 2009).
- Ingles, O. G., and Metcalf, J. B. (1973). *Soil stabilization: principles and practices*, John Wiley and Sons, New York.
- Iowa Department of Administrative Services (IA DAS). (2008). *State set to test BioOil®*, Iowa Department of Administrative Services, Des Moines, Iowa, 2008.
- Iowa Department of Transportation (IA DOT). (2008). “Roadway and borrow excavation.” *Standard specifications*, Section 2102, Ames, Iowa.
- Johnson, J. M., Carpenter-boggs, L., and Lindstrom, M. J. (2003). “Humic acid and aggregate stability in amended soils.” *Proceedings of the Natural Organic Matter in Soils and Water North Central Region Symposium*, 21.
- Johnson, J. M. F., Reicosky, D., Sharratt, B., Lindstrom, M., Voorhees, W., and Carpenter-Boggs, L. (2004). “Characterization of soil amended with the by-product of corn stover fermentation.” *Soil Sci. Soc. Am. J.*, 68, 139–147.
- Joint Departments of the Army and Air Force (JDAAF). (1994). *Soil stabilization for pavements*, TM 5-822-14/AFMAN 32-8010, Washington, DC.
- Kamm, B., and Kamm, M. (2004) “Principles of biorefineries.” *Applied Microbiology and Biotechnology*, 64 (2), 137-145.
- Kozan, G. R. (1955). *Summary review of lignin and chrome-lignin processes for soil stabilization*, Miscellaneous paper, U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS.

**Reference** to this paper should be made as follows: Kim, S., Gopalakrishnan, K., and Ceylan, H. (2012) “Moisture Susceptibility of Subgrade Soils Stabilized by Lignin-Based Renewable Energy Co-product.” ASCE Journal of Transportation Engineering, Vol. 138, No. 11, pp 1283-1290.

- Landon, B., and Williamson, R. K. (1983). “Dust-abatement materials: evaluation and selection.” *Transportation Research Record: Journal of the Transportation Research Board*, 898, National Research Council, Washington, DC., 250-257.
- Lane, D. D., Baxter, T. E., Cuscino, T., and Coward, C. Jr. (1984). “Use of laboratory methods to quantify dust suppressants effectiveness.” *Transactions of the Society of Mining Engineers of the American Institute of Mining, Metallurgical, and Petroleum Engineers*, 274, Society for Mining, Metallurgy, and Exploration, Littleton, Colo., 2001–2004.
- Lignin Institute (2008). Lignin Institute, <<http://www.lignin.org/01augdialogue.html>> (March 1, 2009).
- Lora, J. H., and Glasser, W. G. (2002). “Recent industrial applications of lignin: a Sustainable alternative to nonrenewable Materials.” *Journal of Polymers and the Environment*, 10 (1-2), 39-48.
- Mindess, S., Yong, J. F., and Darwin, D. (2002). *Concrete*, Prentice Hall, NJ.
- Nicholls, R. L., and Davidson, D. T. (1958). “Polyacids and lignin used with large organic cations for soil stabilization.” *Highway Research Board Proceedings*, 37, 517-537.
- Palmer, J. T., Edgar, T. V., and Boresi, A. P. (1995). *Strength and density modification of unpaved road soils due to chemical additives*, Mountain-Plains Consortium (MPC) Report No. 95–39, Fargo, ND.
- Puppala, A. J., and Hanchanloet, S. (1999). “Evaluation of a chemical treatment method (sulphuric acid and lignin mixture) on strength and resilient properties of cohesive soils.” *Proceedings of 78th Transportation Research Board Annual Meeting* (CD ROM), National Research Council, National Academy of Science, Washington, DC.
- Santoni, R. L., Tingle, J. S., and Webster, S. L. (2002). “Stabilization of silty sand with nontraditional additives.” *Transportation Research Record: Journal of the Transportation Research Board*, 1787, National Research Council, Washington, DC., 61-70.
- Santoni, R. L., Tingle, J. S., and Nieves, M. (2005). “Accelerated strength improvement of silty sand with nontraditional additives.” *Transportation Research Record: Journal of the Transportation Research Board*, 1936, National Research Council, Washington, DC., 34-42.
- Sinha, S. P., Davidson, D. T., and Hoover, J. M. (1957). “Lignins as stabilizing agents for northeastern Iowa loess.” *Iowa Academy of Science Proceedings*, 64, 314-347.
- Stewart, D. (2008). “Lignin as a base material for materials applications: chemistry, application and economics.” *Industrial Crops and Products*, 27 (2), 202-207.
- Sundstrom, D. W., Herbert E. K., and Daubenspeck, T. H. (1983). “Use of byproduct lignins as extenders in asphalt.” *Ind. Eng. Chem. Prod.*, 22(3), 496-500.
- Tingle, J. S., and Santoni, R. L. (2003). “Stabilization of clay soils with nontraditional additives.” *Transportation Research Record: Journal of the Transportation Research Board*, 1819, National Research Council, Washington, D.C., 72-84.
- Woods, K. B. (1960). *Highway engineering handbook*, 1<sup>st</sup> Edition, McGraw-Hill.