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Detection and Assessment of Sulfates in the Pavement Subgrade

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16. Abstract

The ability to accurately detect sulfates in an existing subgrade has been a major challenge for INDOT's pavement maintenance and rehabilitation projects. Although naturally occurring sulfates in soil pose a significant construction challenge, detecting sulfates in an existing subgrade may be problematic and costly, given that the presence of sulfates could result in expensive treatments or even removal of the subgrade. The project evaluated the performance of the ITM-510 test, a colorimetric test method utilized by INDOT to detect sulfates in soils. Soils collected from different Indiana road construction projects and two ASTM reference soils were treated with lime and lime kiln dust and tested for sulfates after being cured for 7 and 28 days. The sulfate content obtained from the ITM-510 test was compared with levels obtained from the ion chromatography (IC) test, an analytical test method used to separate, identify, and quantify ions in solution. Test results indicated that ITM-510 was adequate for identifying the presence of sulfates and for providing accurate values of sulfate concentration at <5,000 ppm sulfate concentrations. For larger concentrations, the errors may be large. It was also found that about 3% sulfate concentration in lime products can raise the sulfate concentration of lime-treated soils by as much as 2,000 ppm.

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EXECUTIVE SUMMARY

Introduction

The subgrade layer is essential for the long-term performance of pavement systems, but problem soils can create challenges in design, construction, and performance. Chemical stabilization, using agents like lime or cement, is a common practice for improving the mechanical properties of problem soils. However, when rehabilitating or maintaining roads where the subgrade has been treated with lime, INDOT can detect sulfates in the existing subgrade, which may result in costly soil removal despite the subgrade showing no signs of distress.

In this study, the test protocol used by INDOT to determine the sulfate concentration of soils was reviewed and compared with other existing sulfate content determination tests. The reliability of the test was also assessed by performing tests on subgrade soils collected from different road construction projects in Indiana and treating them with lime and lime kiln dust used by INDOT for subgrade treatment. The sulfate content was determined by ITM-510, a colorimetric test method utilized by INDOT, and by ion chromatography, an analytical test method used to separate, identify, and quantify ions in solution.

Findings

The following conclusions have been drawn from this research.

1. Lime kiln dust samples used for subgrade treatment may contain sulfates as impurities and are most likely the source of sulfates in the existing treated subgrades.
2. Due to difficulties in accurately distinguishing between the weight loss caused by the decomposition of gypsum and the loss of free water in treated samples, thermogravimetric tests were found to provide unreliable estimates of sulfate content.
3. Ion chromatography (IC) and inductively coupled plasma-optical emission spectroscopy (ICP-OES) can be used as ground-truths to quantify sulfates in soils.
4. The ITM-510 test methodology was adequate for identifying the presence of sulfates and for providing accurate values of sulfate concentration, provided that the sulfate concentration did not exceed 5,000 ppm. For larger sulfate concentrations, the errors may be large.
5. For samples containing high amounts of sulfate (>5,000 ppm), a dilution rate higher than the one used in ITM-510 tests (>1:20) may be necessary to dissolve all available sulfates.
6. Lime samples with a reported sulfate concentration of about 3% were found to raise the sulfate concentration of soils with no initial sulfate content by as much as 2,000 ppm upon treatment. The current INDOT criterion for allowable sulfate in subgrades is 1,000 ppm.

Implementation

Based on the literature reviewed for sulfate test protocols and the results obtained from the laboratory tests, the following implementations are recommended.

1. The ITM-510 test protocol works well and provides an accurate estimate of sulfate concentration in soils. INDOT should continue to use the test to determine the sulfate concentration in subgrade soils.
2. Samples showing high sulfate levels (>5,000 ppm) should be tested at dilutions higher than 1:20 to ensure that all soluble sulfate is dissolved and accurately measured.
3. The current 5% limitation on soluble sulfate content of lime by-products in Article 913 of the INDOT standard specifications should be reduced to 3%.
4. This research did not cover the mechanical performance of lime-treated subgrades testing positive for sulfates. This issue is outside the scope of this project. It is, however, strongly recommended that this problem be investigated, given the large number of roads that have been treated with lime and the potential economic implications of discarding the subgrade soils because of their high sulfate content.

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1. INTRODUCTION

The subgrade layer acts as the foundation for pavement systems and plays a crucial role in their long-term performance. As this layer is composed of in-situ material, when a problem soil is encountered, significant challenges may arise in the design, construction, and performance of the overlying pavement systems. The two most adopted practices to deal with problem soil and thereby enhance the subgrade performance are full or partial soil replacement and chemical stabilization. However, the economic considerations (excavation, material transportation, etc.) associated with soil replacement make it less desirable, as a solution, when compared to chemical treatment/stabilization.

Chemical treatment, a key method to enhance subgrade performance, involves the use of chemical agents such as lime or cement. These agents are known to increase subgrade stiffness while reducing its plasticity and swelling potential. Lime treatment also tends to decrease the maximum dry unit weight of the soil and increase the optimum moisture content (Petry & Lee, 1988). Puppala et al. (1996) demonstrated that lime treatment of silty clay soil from Louisiana resulted in increasing the plastic limit while decreasing both the liquid limit and plasticity index values. Lime treatment also resulted in an increase in the unconfined compressive strength and resilient modulus of the soil. Abu-Farsakh et al. (2014) reported substantial improvement in terms of increased resilient modulus and reduced permanent deformations of soft subgrade soils with cement treatment. Past research on Indiana subgrades has confirmed the improved properties of the subgrade soil with lime (Branam et al., 2016; Sandoval et al., 2019; Tao et al., 2016). A comprehensive field investigation on subgrade soils treated with lime in pavements that had been in service for at least 5 years (Jung & Bobet, 2007; Jung et al., 2009) showed that (1) the lime remained in the soil even after 11 years of service of the roads; (2) the addition of lime decreased the plasticity of the soil and increased its stiffness; and (3) the construction quality observed from the field tests was highly variable. The project found that the fine content of the original soil was reduced by the addition of lime. The lime content in the subgrade was evaluated through thermogravimetric (TGA) analyses and showed that the $CaCO_3$ content, which is related to the content of lime that remained in the subgrade, ranged from 1.2% to 17.5%, with typical values in the range of 5% to 7%.

A problem that INDOT is encountering, when rehabilitating or maintaining roads where the subgrade has been treated with lime, is the detection of sulfates in the existing subgrade. Naturally occurring sulfate in soil poses significant challenges due to its potential to cause expansive reactions with calcium and free hydroxide present in cementitious materials, resulting in the formation of crystalline minerals such as ettringite and thaumasite. The consequences of detecting sulfates are significant as current specifications (*INDOT Standard Specification 2022*, clause 207.02 (INDOT, 2022)) require removal of the soils which is a costly outcome. Moreover, in most cases, this is likely an unnecessary intervention, given that the subgrade soils have been in service for several years and that the pavement has not shown signs of distress. The problem is becoming significant given the number of maintenance and rehabilitation projects that are ongoing or planned. Indiana has been chemically treating the subgrade natural soils with lime, lime kiln dust or cement for many years. This has been, and still is, a successful process that has allowed for a stable work platform to build the pavement, has expedited the construction of pavements and has lowered the cost of construction and maintenance. The chemicals used to treat the subgrade could contain sulfates as a byproduct or as impurities; as a result, the subgrade may test positive for sulfates which, according to specifications, requires the soil to be removed. Also, because of the lime in the soil, the turbidity test used to detect sulfates could give a false positive.

The following are the main objectives of the work.

- Develop a testing protocol or method to improve the detection of sulfates in the pavement subgrade and prevent false positives. To date, the presence of sulfates in lime-treated subgrades that have been in service for several years has been detected using the Indiana ITM-510 test method. The method is based on a turbidity/colorimetric analysis of a soil slurry to measure the water-soluble sulfate ion content in the soil. Thus, an evaluation of the current ITM-510 test method is essential to determine whether the results obtained are accurate and reliable.
- Provide further understanding of the composition of the chemical agents used for subgrade treatment, their chemo-mechanical evolution over time, that may contribute to false positives for sulfates.

These objectives have been accomplished through several tasks, including a literature review of the different testing protocols used for the detection of sulfates, a review of the INDOT standard protocol for soluble sulfate detection, and laboratory testing to assess the accuracy of the INDOT standard. The following sections provide a summary of the work done and the major conclusions drawn.

2. BACKGROUND

2.1 Test Methods for the Detection of Sulfates

The methods available for the detection of sulfates in soils are based on one of the following test principles.

Turbidity: The turbidity method for sulfate detection operates on the principle that sulfate ions (SO_4^{2-}) in a sample react with barium chloride ($BaCl_2$) under acidic conditions to form insoluble barium sulfate ($BaSO_4$) precipitates. This reaction causes the solution to become turbid or cloudy due to the formation of fine suspended particles of barium sulfate. The turbidity of the sample is then measured using a turbidimeter or nephelometer, which quantifies the amount of light scattered by the suspended particles. The degree of turbidity is directly proportional to the concentration of sulfate ions in the sample.

Colorimetry: This method is like the turbidity method. The sulfate ions are precipitated as barium sulfate. These precipitates are then dissolved in a strong acid solution, and an organic dye, such as methylthymol blue or turbidimetric reagent, is added. The resulting solution exhibits a color change whose intensity is directly proportional to the sulfate concentration. This color change is measured using a spectrophotometer at a specific wavelength corresponding to the absorption maximum of the formed complex. Quantification is achieved by comparing the absorbance of the sample with a calibration curve prepared from standard solutions of known sulfate concentrations.

Gravimetry: The soluble sulfate is precipitated as barium sulfate in an acidic medium. The precipitate is filtered, washed and ignited or dried and weighed as $BaSO_4$.

Ion Chromatography (IC): This method is useful in separating, identifying, and quantifying ions in solution. The chromatographic system comprises analytical columns (anion and cation) that separate the ions of interest from the solution. The resin inside the columns is functionalized with either cation or anion exchange groups and acts as the stationary phase. The sample is injected into the chromatographic system and a mobile phase (typically an aqueous solution) is passed through the column. As the mobile phase flows through the resin, ions in the sample interact with the resin. The extent of the interaction depends on the ions' respective charge, size, and affinity for the resin material. Sulfate concentration is measured based on the retention time, which is measured as the time between injection and detection.

Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES): This method is used to determine and quantify the elemental composition of the sample. The sample is introduced into a high-intensity plasma, where the extreme temperatures cause the excitation of electrons in the sample's atoms. As these electrons return to their ground state, they emit light at characteristic wavelengths. By measuring the intensity of this emitted light, the concentration of different elements in the sample can be determined. Calibrating solutions prepared with high purity standards containing known concentrations of the elements of interest are used to create calibration graphs. The concentrations of the elements present in an unknown solution are then determined from these calibration graphs.

A review of testing protocols followed by various state DOTs (Illinois, Ohio, Texas, Colorado, etc.) and other agencies (ASTM, AASHTO, U.S. Army Corps) for the detection of sulfates in soils was conducted. It was observed that many DOTs use a colorimetric/turbidity test like that used by INDOT. Table 2.1 summarizes the differences in sample size, sieve size used to remove larger particles, dilution ratio, mixing duration, and chemicals used by the different agencies. Test methods other than the colorimetric include ion chromatography, used by the California DOT, acid soluble method and conductivity method, used by the U.S. Army Corps, and Gravimetric test methods, used by the Nebraska DOT and AASHTO T 290 standard. INDOT protocol uses a sulfate reagent chemical to precipitate soluble sulfates present in the soil, while few state agencies utilize other chemicals. The quantity and significance of chemicals used in the various standards are summarized in Table 2.2.

The turbidity test generally requires soil samples to be oven- or air-dried and passed through a sieve or series of sieves. Samples are diluted and mixed by mechanical methods (hand mixing or shaking and then allowed to stand for a period before filtration. After filtration, the turbidimeter is calibrated with a vial containing pure filtrate. A reagent is added to another vial of filtrate for the sulfate content analysis. Indiana, Colorado, Oklahoma, Ohio, and Texas DOTs use such turbidity tests.

One of the major objectives of this project is to identify whether the test standard ITM-510 used by INDOT for the identification of sulfates can yield false positives or false negatives. A literature review was conducted to identify chemicals that could interfere with the results of the test. Table 2.3 lists these chemicals, as well as the reference source. The table also highlights the effect certain chemicals have on the test results; a negative effect implies the chemical acts as an inhibitor and restricts the precipitation of barium sulfate, thereby underpredicting the sulfate concentration, while a positive effect implies the results are overpredicting the actual concentration present in solution. The interferences reported in the HACH document apply to an undiluted 10 ml sample, and these interferences are reported to increase proportionally with sample dilution. In addition to these chemicals, all sources mentioned the removal of any suspended impurities or sample turbidity prior to performing the sulfate tests.

2.2 Lime Production

Lime and its by-products have long been used in the treatment and stabilization of subgrade soils (Bustillo, 2021; Button, 2003; Carpenter et al., 1992; Collins & Emery, 1983; Jung & Bobet, 2007; Solanki et al., 2010). INDOT standard specifications allow for chemical treatment of subgrade soils with lime (hydrated lime and quicklime) or lime by-products (lime kiln dust) to improve soil plasticity, workability, swell potential, and strength. Since the primary objective of this project is to identify the source of sulfates in subgrade soils initially treated with lime, a database of the chemical composition of lime was prepared. Lime, which is produced by the calcination of limestone at high temperatures, is known to include impurities in the form of silica, alumina, iron, sulfur, etc. Although some of the impurities present in the form of sodium, potassium

TABLE 2.1
Laboratory tests used by different agencies to detect sulfates in soils

Agency	Test	Sample Size	Sieve Size	Dilution Ratio	Sample Preparation	Soaking Duration	Chemicals
Indiana DOT (INDOT, 2013)	Colorimeter test (ITM-510)	500 g 20 ± 0.1 g (3 samples)	No. 4 No. 40	1:20 soil/water ratio	1 min hand mixing followed by filtration	Overnight (12 hr)	Sulfate reagent chemicals
Illinois DOT (AASHTO, 2016)	Gravimetric test (modified AASHTO procedure)	100 g	1/4 in. No. 4 No. 10	300 mL	Shaking followed by centrifuging the sample	–	Ammonium hydroxide, NH_4OH. Barium chloride, $BaCl_2{\cdot}2H_2O$; Hydrochloric acid, HCl; Silver nitrate, $AgNO_3$; Sulfuric acid, H_2SO_4
Ohio DOT (ODOT, 2005)	Turbidity test (Supplement 1122)	100 g (±20 g) Each (3 samples)	No. 4	400 mL 1:20 soil/water ratio	1 min hand mixing followed by filtration	16–24 hr	Sulfate reagent chemicals (tablets or powder)
Texas DOT (TxDOT, 2005)	Turbidity test (Texas 145E)	40 g (3 samples)	No. 40	400 mL 1:20 soil/water ratio	1 min hand mixing followed by filtration	Overnight (12 hr)	SulfaVer4 powder pillow (HACH chemical)
California DOT (Cal Trans, 2006)	Ion chromatography test (Test 147)	100 g	–	300 mL	Shake vigorously for 15 min, followed by filtration	–	Sodium bicarbonate, $NaHCO_3$; Sodium carbonate, Na_2CO_3
Colorado DOT (CDOT, 2021)	Turbidity test (CP-L 2103)	25 g	No. 4 No. 40	250 mL of distilled water 10:1 dilution	Shaking followed by filtration	16 hr	Barium chloride ($BaCl_2$) 100:1
Nebraska DOT (NDT, 2024)	Gravimetric test (modified AASHTO method)	100 g	1/4 in. 10 No. 4 No. 10	300 mL	Shaking followed by centrifuging the sample	–	Ammonium hydroxide, NH_4OH; Barium chloride, $BaCl_2{\cdot}2H_2O$; Hydrochloric acid, HCl; Silver nitrate, $AgNO_3$; Sulfuric acid, H_2SO_4
Oklahoma DOT (ODOT, 2005)	Turbidity test (OHD L-49)	300 g–5,000 g	No. 10	1:40 soil/water ratio	By hand, for 15 sec every 5 min, for 15 min; with mechanical shaker for 15 min followed by filtration	16 hr	Sodium sulfate (Na_2SO_4)
ASTM C1580 (ASTM, 2021)	Turbidity	100 g A: 30 g B: 3 g	No. 30	400 ml of distilled water	Stir on a magnetic stirrer for at least 1 hr but no more 1.2 hr followed by filtration	–	Barium chloride–Crystals of barium chloride, $BaCl_2{\cdot}2H_2O$; Sodium hydroxide, NaOH; Hydrochloric acid, HCl
AASHTO T 290-95 (AASHTO, 2020)	Gravimetric and turbidity method	100 g	1/4 in. No. 4 No. 10	300 mL	Shaking followed by centrifuging the sample	–	Ammonium hydroxide, NH_4OH; Barium chloride, $BaCl_2{\cdot}2H_2O$; Hydrochloric acid, HCl; Silver nitrate, $AgNO_3$; Sulfuric acid, H_2SO_4
U.S. Army Corps (WBDG, 1971)	Acid soluble method and conductivity method	1 g	No. 50	50 mL	Mixing for 15 minutes followed by filtration	–	HCl

Note: "–" implies specific information that was not mentioned in the document.

TABLE 2.2
Purposes of different chemicals used in sulfate determination tests

Test	Apparatus	Chemical	Quantity	Purpose
AASHTO T 290 Method B (AASHTO, 2020)	Turbidity: photometer	Barium chloride	–	To precipitate soluble sulfates
		Glycerin solution	1 volume of glycerin in 1 vol of water	To prevent $BaSO_4$ precipitates from settling down due to flocculation
		NaCl solution	240 g NaCl + 1 L water + 20 mL conc HCl	Same
		Sulfate standard solution	0.1479 g of anhydrous Na_2SO_4 + 1 L water	To overcome difficulty if sulfate concentration is low
		Conc HNO_3	A drop	To precipitate finely divided suspended matter
AASHTO T 290 Method A (AASHTO, 2020)	Gravimetric: furnace	NH_4OH	Not listed in procedure	–
		$BaCl_2{\cdot}2H_2O$	100 g in 1 L water	To precipitate soluble sulfates
		$AgNO_3$	10 g in 100 mL	To test washings for presence of chlorides
		H_2SO_4	Few drops	To expel silica as silicon tetrafluoride
		HNO_3	–	To precipitate finely divided suspended matter
		HCl	1 vol in 9 vol of water	To remove silica by dehydration.
		HF	A drop	To expel silica as silicon tetrafluoride
		Methyl orange indicator	0.05 g in 100 mL	As an end point for acidity check
		Picric acid	Standard	Faster precipitation and coarser precipitate
Modified AASHTO procedure-Illinois (AASHTO, 2016)	Gravimetric: furnace	$BaCl_2{\cdot}2H_2O$	5 ml–while heating and left min 2 hr settling period	To precipitate soluble sulfates
		$AgNO_3$		To test washings for presence of chlorides
		HNO_3	1–2 drops	To settle out finely divided suspended matter
		HCl	10 ml	To make solution acidic
		Methyl orange indicator	1–2 drops (0.01 g in 100 ml)	As an end point
ASTM C 1580 (ASTM, 2021)	Photometer	Barium chloride crystals $BaCl_2{\cdot}2H_2O$	0.3 g	To precipitate soluble sulfates
		Conditioning reagent	5 ml {30 ml HCl + 95% ethanol + 300 reagent water + 75 g NaCl + 50 mL glycerol}	To prevent $BaSO_4$ precipitates from settling down due to flocculation. If it is not prevented the turbidity observation keeps going down thereby not allowing the observer to record stable data
		Sulfate standard solution	0.1479 g of anhydrous Na_2SO_4 + 1 L water	To make calibration chart for the photometer
		Sodium hydroxide	4 g in 1 L	To neutralize the pH of the filtrate (7 ± 1) (0.1 N NaOH)
		HCl	8.6 mL conc HCl in 1 L	To neutralize the pH of the filtrate (7 ± 1) (0.1 N HCl)

or sulfur compounds might get removed during the calcination process, heterogeneous impurities in the form of clay particles or sulfur oxides arising from the fuel tend to remain as fine fractions during the production of lime (Oates, 2008, 2010). The presence of oxides and trace elements in lime and its by-products has the potential to create expansive mineral products. When the sulfur trioxide (SO_3) content exceeds 3% or the magnesium oxide (MgO) content goes beyond approximately 3%–5%, the formation of ettringite or brucite may occur (Little & Nair, 2009). Additionally, sulfur impurities can contribute to an increase in the soluble sulfate content of subgrade soils. These soils, which before treatment exhibited negligible or acceptable sulfate concentrations, may display a rise in sulfate levels with the addition of lime. A typical range for the levels of impurities found in limestones is presented in Table 2.4.

2.2.1 Types of Lime and Lime By-Products

The term lime refers specifically to the oxides and hydroxides of calcium and magnesium. For industrially manufactured lime, the limestone rock must contain at

TABLE 2.3
List of chemicals found to interfere with sulfate determination tests

Source	Chemical	Quantity	Effect
ASTM D 516 (ASTM, 2023)	Aluminum polymer	Not specified	Non-uniform precipitation
	Chloride	$>5{,}000$ mg/L	Negative
	Organic matter	Large quantity	Non-uniform precipitation
	Polyphosphates	1 mg/L	Negative
	Phosphonates	Low concentration	Negative
	Silica	>500 mg/L	Positive
Hach document (DOC316.53.01312)	Barium	At all levels	Negative
	Calcium	$>20{,}000$ mg/L as $CaCO_3$	NA
	Chloride	$>40{,}000$ mg/L as Cl^-	NA
	Magnesium	$>10{,}000$ mg/L as $CaCO_3$	NA
	Silica	>500 mg/L as $CaCO_3$	NA

Note: NA indicates that the information not available (the document does not explicitly mention the effect).

TABLE 2.4
Typical ranges of impurities/trace elements in commercial limestones (adapted from Oates, 2008)

	Typical Range		
Impurity or Trace Element	**Low**	**High**	**Units**
Silica (as SiO_2)	0.1	2	%
Alumina (as Al_2O_3)	0.04	1.5	%
Iron (as Fe_2O_3)	0.02	0.6	%
Sulfur (as $CaSO_4$)	0.01	0.5	%
Carbonaceous matter	0.01	0.5	%
Manganese (as MnO_2)	20	1,000	mg/kg
Antimony	0.1	3	mg/kg
Arsenic	0.1	15	mg/kg
Boron	1	20	mg/kg
Cadmium	0.1	1.5	mg/kg
Chromium	3	15	mg/kg
Copper	1	30	mg/kg
Fluoride	S	3,000	mg/kg
Lead	0.5	30	mg/kg
Mercury	0.02	0.1	mg/kg
Molybdenum	0.1	4	mg/kg
Nickel	0.5	15	mg/kg
Selenium	0.02	3	mg/kg
Silver	0.2	4	mg/kg
Tin	0.1	15	mg/kg
Vanadium	1	20	mg/kg
Zinc	3	500	mg/kg

least 50% calcium carbonate. Two primary types of lime products are applied in the chemical stabilization or modification of subgrade soils: quicklime and hydrated lime (or slaked lime). Quicklime is produced by the thermal decomposition of limestone. It consists mainly of calcium oxide, with magnesium oxide as a secondary component. Slaked limes, on the other hand, are produced by reacting quicklime with water to produce either hydrated lime (a dry calcium hydroxide powder), milk of lime, or lime putty. Another commonly used soil stabilizing agent is lime kiln dust (LKD). LKD refers to the fine particles that are emitted from the combustion process of limestone in a lime kiln. It is generated as a by-product and consists mainly of calcium carbonate, calcium hydroxide, and other residual materials. Table 2.5 lists the different types of lime products available.

2.2.2 INDOT Specifications for Subgrade Soils and Lime Products

INDOT has developed specifications for the type of soil that requires stabilization as well as for the type of lime that can be used (Table 2.6 and Table 2.7). The lime used must meet AASHTO M 216 (ASTM C 977) or equivalent for purity requirements while the lime by-products shall be hydrated lime or quicklime by-products manufactured in accordance with ASTM C25. Additionally, lime by-products should contain a minimum of 60% total calcium and magnesium oxides (non-volatile basis), a minimum of 30% available calcium hydroxide plus magnesium oxide calculated as calcium hydroxide, and a soluble sulfate content of less than 5%. The quantities for lime (hydrated lime, quicklime) and lime by-products to be used are based on 5% and 6% of the maximum dry density of the soils, respectively. The optimum lime content, however, is estimated based on ASTM D6276 which follows the Eades and Grim pH test criterion. This test aims to identify the necessary lime quantity to fulfill immediate lime-soil reactions and provide an adequate amount of calcium to maintain a high residual pH, facilitating significant long-term pozzolanic reactions.

2.2.3 Lime Database

A database of lime and lime-by-products was compiled as part of the project (Arulrajah et al., 2017; Ban et al., 2022; Beeghly & Schrock, 2010; Burnham et al., 1992; Button, 2003; Cheah et al., 2023; Collins & Emery, 1983; Daita et al., 2005; Dernegård et al., 2017; Drapanauskaite et al., 2021; Dvorkin & Zhitkovsky, 2023; Hach Co., 2003; Kadhim et al., 2020; Kakrasul et al., 2017; Kakrasul et al., 2018; Kavak & Akyarli, 2007; Khater, 2011; Kurudirek et al., 2010; Kadhim et al., 2020; Latif et al., 2015; Mohammadinia et al., 2020;

TABLE 2.5
Different types of lime products (adapted from Dowling et al., 2015)

Name	Formula	Kiln Temperature	Synonyms
Quicklime	CaO	900°C–1,200°C	Calcium oxide, lime and burnt lime
Dolime	CaO MgO	900°C–1,200°C	Dolomitic lime, dolime and calcium magnesium oxide
Sintered dolime	CaO MgO	1,800°C	Dead burned dolime
Slaked lime	$Ca(OH)_2$	N/A	Calcium hydroxide, calcium hydrate, caustic lime and hydrated lime
Hydrated dolime	$CaMg(OH)_4$	N/A	Calcium magnesium tetrahydroxide

TABLE 2.6
INDOT specifications for soils requiring chemical treatment

Soil Property	Test Method	Requirement
Maximum dry density	AASHTO T 99	≥90 pcf
Organic material	AASHTO T 267	≤6%
Sulfate content	ITM-510	≤1,000 ppm

TABLE 2.7
INDOT specifications for the lime used in chemical treatment

Lime	Test Method	Composition	Requirement
Hydrated lime	AASHTO M 216	–	–
Quicklime	AASHTO M 216	–	–
Lime by-product	ASTM C25	CaO + MgO (non-volatile)	≥60%
		$Ca(OH)_2$ + MgO (as equivalent $Ca(OH)_2$)	≥30%
		Soluble Sulfate	≤5%
	ASTM C110	No. 4 (4.75 mm)	≤5%
		No. 30 (600 μm)	≤15%
		No. 100 (150 μm)	≤30%

Nyström et al., 2019; Solanki, 2010; Tran et al., 2004; Xu et al., 2018; Yahya et al., 2004). The database comprises two primary sources of information: journal papers and safety data sheets. The total number of lime compounds obtained, along with the percentage of samples with a reported sulfate concentration (reported either as %$CaSO_4$, %SO_3, %S, or %SO_4), are presented in Figure 2.1. The concentrations reported in the figure were based on a wide variety of tests (ion chromatography, inductively coupled plasma, x-ray fluorescence, etc.). They were converted into equivalent sulfate concentrations (%SO_4), to facilitate comparisons. Figure 2.2 shows the range of sulfate concentrations typically observed in lime samples. It also highlights the 5% upper limit allowed per INDOT specifications (Section 913 Indiana Department of Transportation specification standards) for limes (5%) used for subgrade treatment.

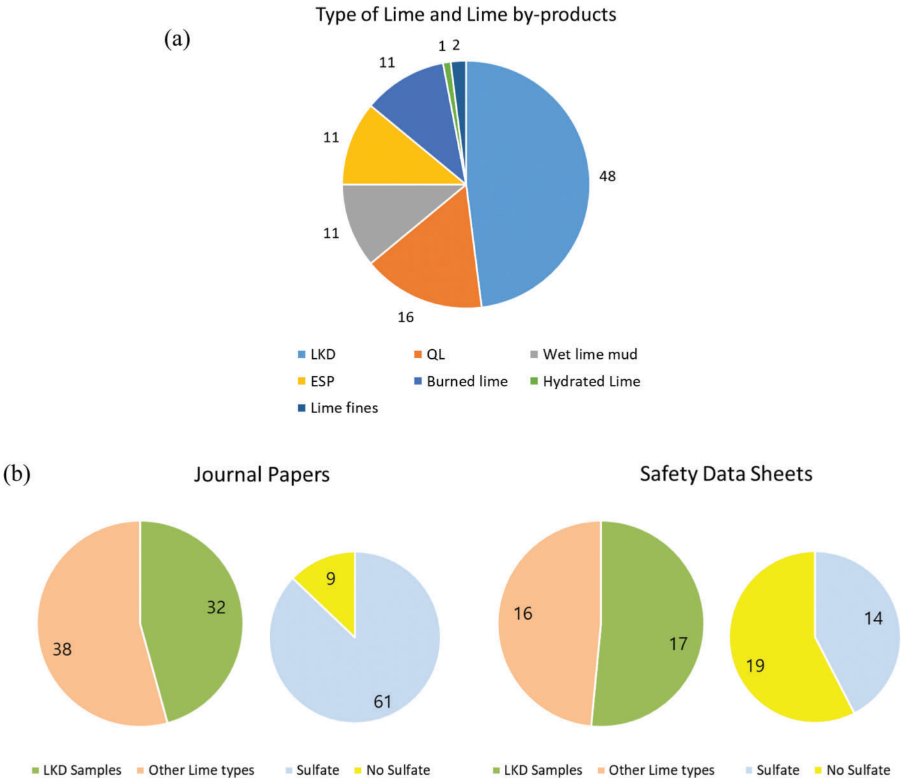

Figure 2.1 Lime classification: (a) different types of lime and lime by-products found in the literature, and (b) lime data obtained from journal papers and safety data sheets.

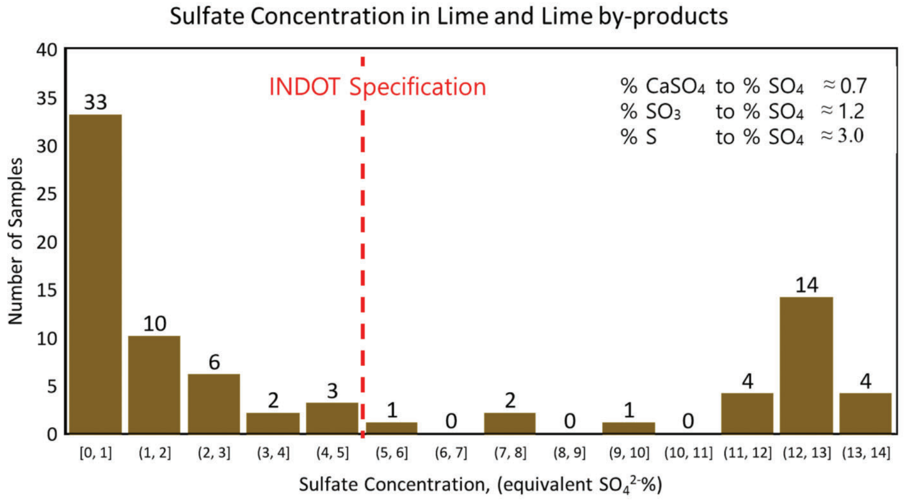

Figure 2.2 Sulfate concentration from collected data.

3. LABORATORY TESTING

3.1 Lime and Soil Samples

Chemical compounds (lime, lime kiln dust) were obtained from manufacturers/vendors frequently contracted by INDOT for subgrade stabilization. Fourteen chemical samples were obtained from different plant locations of Carmeuse. Mineralogical data of these chemicals provided by the manufacturer are presented in Table 3.1. The water-soluble sulfate content given by the manufacturer for lime kiln dust samples was based on results from ASTM D516 standard, which is a turbidity test. Out of the fourteen samples received, two lime kiln dust samples (an LKD sample from the Buffington plant, referred to as LKD_Bu, and the other from the Cheney K2 plant, referred to as LKD_CK2) and two quicklime samples (high calcium quicklime sample from Beachville plant referred to as HC85_BV and quicklime from Black River, referred to as QL_BR) were selected to prepare soil-lime mix samples.

TABLE 3.1
Carmeuse chemical specification data for lime products

Sample No.	Plant	Product	Percent of CaO	Percent of MgO	Water Soluble Sulfate Ion Content (%)	Free Lime as CaO	Free Lime as $Ca(OH)_2$
1	Beachville	Hi Cal 85 Quicklime	95.89	1.42	NA	NA	NA
2	Beachville	Cal 85 Quicklime	95.9	1.45	NA	NA	NA
3	Black River	Quicklime	92.06	3.16	NA	NA	NA
4	Black River	LKD	71.59	4.01	1.32	34.63	45.72
5	Longview	LKD	61.18	12.4	1.266	25.08	33.13
6	Longview	Hi Cal QL	96.67	2.62	NA	NA	NA
7	Cheney K2	LKD	69.62	3.1	2.712	30.43	40.2
8	Buffington	LKD	79.02	2.16	1.101	33.67	44.48
9	Buffington	Hi Cal Fines	91.74	2.56	NA	NA	NA
10	Luttrell	Quicklime	96.8	1.18	NA	NA	NA
11	River Rogue	LKD	77.64	2.07	1.095	29.7	39.23
12	River Rogue	Lime Fines	97.93	1.86	NA	NA	NA
13	Maple Grove	LKD	50.3	34.47	1.887	11.08	14.64
14	Maysville	LKD	72.07	7.21	0.6158	17.75	23.45

TABLE 3.2
Designation and sampling location of soil samples

Soil	Sample Designation	Location
Soil 1	I-70	I-70, Indianapolis
Soil 2	SR-46	SR-46, Clay County
Soil 3	S-BRITE	S-BRITE, Tippecanoe County
Soil 4	SR-37	SR-37, Martinsville
Soil 5	I-65	I-65, Tippecanoe County
Soil 6	HCM_SC	Hawthorn Coal Mine, Sullivan County
Soil 7	ASTM CH	Vicksburg Buckshot Clay
Soil 8	ASTM CL	Annapolis Clay

Five sites were selected for collecting subgrade soil samples. The location of the sites is displayed in Figure 3.1, and site details are provided in Table 3.2. Four of the sites selected (Sites 1, 2, 4, and 5) are INDOT road construction projects. Site 3 is located at the Steel Bridge Research Inspection Training and Engineering (S-BRITE) Center and is herein, a Purdue research facility in West Lafayette, IN. It is herein referred to using the center's acronym. In addition to the subgrade soil samples collected from INDOT construction sites, three more soil samples were selected: Hawthorn Coal Mine spoil samples, Vicksburg Buckshot clay, and Annapolis clay. Hawthorn coal mine samples were collected from Sullivan County in Indiana as part of a previous JTRP project (Jung et al., 2009). They are referred to as HCM_SC in this report. The other two soils are ASTM reference clays. Four samples were collected from the I-70 site in Indianapolis. The first sample was collected from an older section of the site, where the previously treated subgrade was exposed; this is referred to as Sample 1 old, treated. No prior information about the kind of treatment for this sample could be obtained. The second sample was obtained from a section wherein the subgrade was freshly treated with cement, referred to as Sample 2, cement-treated. The remaining two samples were obtained from exposed untreated subgrade sections.

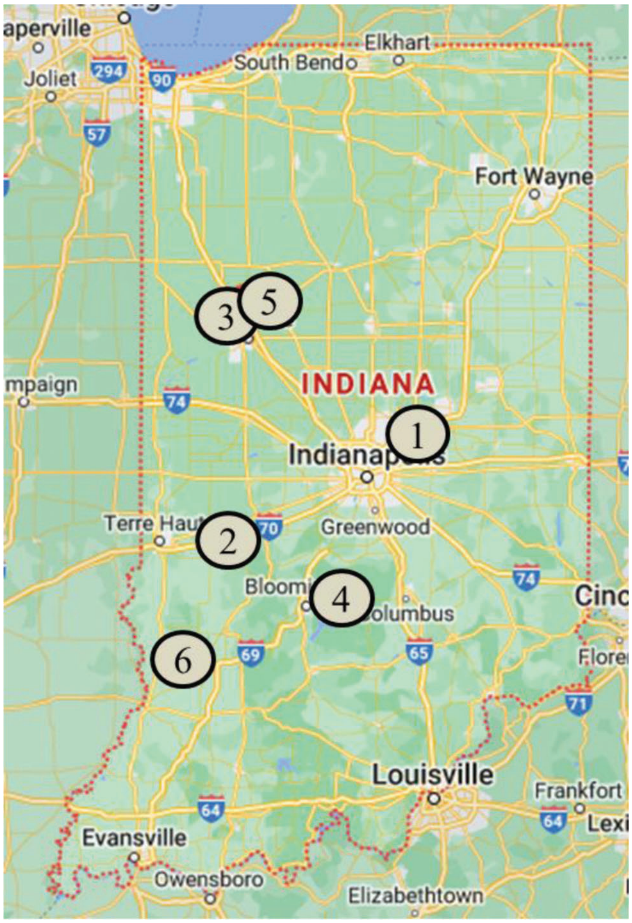

Figure 3.1 Sites selected for collection of subgrade soil samples.

The soil samples collected at the sites were subjected to laboratory tests. Atterberg limits and particle size distribution were determined following the AASHTO T-89/T-90 (AASHTO, 2011) and AASHTO T-88 standards, respectively. Representative samples were washed over the #10 sieve, and sieve analysis was performed, while a subsample of the passing portion was used for hydrometer analysis. In addition to classification tests (AASHTO, 2012), pH and loss on ignition tests were also performed on all samples as per ASTM 4972 and ASTM D2974, respectively (ASTM, 2020, 2024). Table 3.3 summarizes the results of these tests.

3.2 Testing Program for Soil-Lime Mix Samples

Compacted specimens of soil and lime mixes were prepared and cured for 7 and 28 days. The percentage of lime used for treatment was decided based on the optimum lime content obtained per ASTM D6276 (ASTM, 2019). When this procedure yielded an optimum dosage of <5%, an "overdosed" lime concentration equaling 5% was used. The following procedure was used to prepare the compacted soil specimens. Air-dried soil was first broken down to pass #4 sieve and then mixed with lime, with the lime quantity being in accordance with the dosage percentage relative to the soil's weight. Water was then added to the soil to reach the OMC, and the resulting mixture was hand mixed and then compacted in a Harvard miniature mold. The compacted specimens were wrapped in cling film and stored in Ziploc bags and placed inside a water cooler for curing at room temperature. At the end of the curing period, the samples were airdried and crushed in a mortar and pestle. The pulverized soil-lime mix was passed through a #40 sieve and subsequently used to prepare a soil slurry. The slurry was shaken for a minute and allowed to soak overnight before being filtered using 0.45-micron filter paper. The setup used for filtration is presented in Figure 3.2. The filtrate was used to perform tests for sulfate concentration measurements, while the soil-lime mix was further passed through a #200 sieve for mineralogical tests.

3.2.1 Mineralogical Tests

The mineralogical composition of some of the treated specimens was analyzed using X-ray diffraction (XRD) and thermogravimetric analysis (TGA). The pulverized soil-lime mix was passed through #200 sieve for these analyses. XRD analyses were performed using

TABLE 3.3
Index properties, classification, pH and LOI results of the subgrade soils

	Site 1 I-70			
	Sample 1: Old, Treated	**Sample 2: Cement-treated**	**Sample 3: Untreated**	**Sample 4: Untreated**
Fines %	63	51	58	52
LL %	32	NP	39	30
PL %	15	NP	19	15
AASHTO Classification	A-6	A-4	A-6	A-6
LOI (%)	2.0	1.9	2.9	1.7
pH with Test Water	8.1	11.1	8.4	8.3
(Temp (°C))	(22.9)	(22.5)	(22.9)	(22.7)
pH with $CaCl_2$	6.5	9.9	6.5	6.6
(Temp (°C))	(22.5)	(22.5)	(22.4)	(22.4)
	Site 2 - SR-46	Site 3 - S-BRITE	Site 4 - SR-37	Site 5 - I-65
Fines %	50	49	23	46
LL %	26	28	23	24
PL %	15	17	12	15
AASHTO Classification	A-6	A-6	A-2-4	A-4
LOI (%)	2.2	2.0	1.1	2.6
pH with Test Water	8.8	7.6	8.5	8.6
(Temp (°C))	(22.8)	(23.5)	(22.9)	(23.1)
pH with $CaCl_2$	6.5	5.7	6.3	6.4
(Temp (°C))	(23.6)	(23.4)	(23.3)	(23.1)
	Site 6 - HCM_SC	ASTM CH	ASTM CL	–
Fines %	86	–	–	–
LL %	38	61	34	–
PL %	20	19	15	–
AASHTO Classification	A-6	A-7	A-6	–
LOI (%)	–	2.2	2.3	–
pH with Test Water	5.9	8.1	3.1	–
(Temp (°C))	(24.8)	(23.2)	(22.7)	
pH with $CaCl_2$	4.4	6.1	2.4	–
(Temp (°C))	(24.5)	(23.2)	(23.4)	

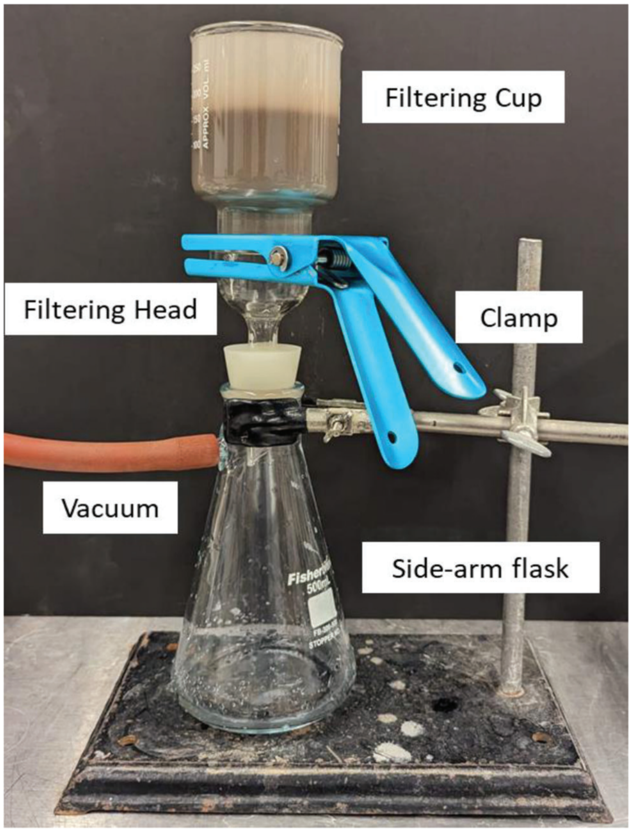

Figure 3.2 Set-up used for filtration.

a SIEMENS D500 X-ray diffractometer while the TGA tests were performed using TGA-2050, thermogravimetric analyzer manufactured by TA Instruments. Approximately 20 mg of soil were placed in the furnace of the analyzer and then heated in a nitrogen gas at a rate of 10°C/min from 20°C to 1,000°C.

3.2.2 Tests for Sulfate Content Determination

Two tests were utilized to obtain the sulfate concentration: ITM-510 and ion chromatography (IC). ITM-510 is an indirect turbidity test that measures sulfate concentration by precipitating the soluble sulfates as barium sulfate. It works on the principle of scattering light. A HACH Multiparameter Colorimeter with a detection range varying from 0 to 70 ppm was used for the ITM-510 tests (Figure 3.3). The INDOT facility in Indianapolis utilizes a similar device.

Figure 3.3 HACH Multiparameter Colorimeter used for ITM-510 tests.

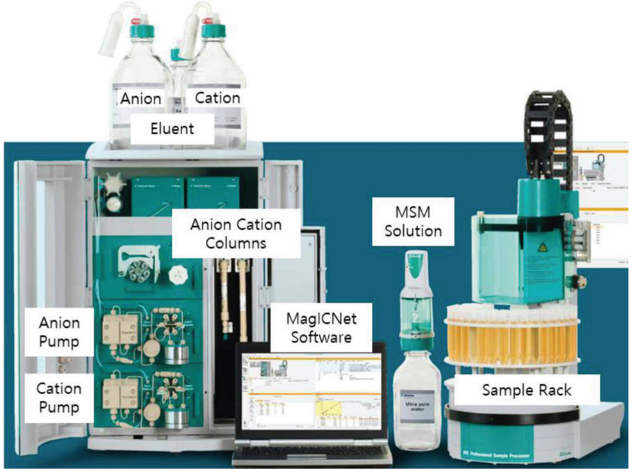

Figure 3.4 Metrohm Ion Chromatograph used for IC tests (image adapted from Metrohm, n.d.).

The ion chromatography tests were performed on a Metrohm Ion Chromatograph and the results were analyzed with the help of MagIC net software (Figure 3.4). The filtrate extracted from the soil water slurry was injected into the chromatographic system, while a mobile phase (anion eluent comprised of 3.2 mM of sodium carbonate and 1.0 mM of sodium bicarbonate) was passed through the anion column. As the mobile phase flows through the resin in the column, the sulfate ions in the sample interact with it. The retention time of sulfate ions was calibrated against standard sulfate solutions, and the sulfate ion concentration in the filtrate was measured by recording the electrical conductivity of the passing elute.

Ion chromatography tests were performed to assess the accuracy of sulfate concentrations determined by the ITM-510 standard. IC is considered more accurate for identifying the composition of ions in a solution. Both devices have limitations regarding the maximum amount of sulfate that can be detected; thus, filtrates with high sulfate were further diluted to fall within the range of each test equipment.

Table 3.4 summarizes the experimental program followed for sulfate content determination. Unless otherwise noted the ITM-510 tests are based on a 1:20 dilution, in accordance with the testing standard.

TABLE 3.4
Overview of tests performed for sulfate content determination

	TGA	XRD	ITM-510	IC
Soils				
I-70 Sample 1	×	–	×	–
I-70 Sample 2	×	–	×	–
S-BRITE	×	×	×	×
HCM_SC[c1]	×	–	×	×
I-65	×	–	×	×
SR-37	×	–	×	×
ASTM CH	×	–	×	×
ASTM CL[c1]	×	–	×	×
Chemical Additives				
LKD_Bu[c2]	×	–	×	×
LKD_CK2[c3]	–	–	×	×
QL_BR[c4]	×	–	×	×
HC85_BV	–	–	×	×
Gypsum	×	×	–	–
Soil-Lime Mixes				
I-70 S1 + LKD_Bu[a]	×	–	×	–
I-70 S2 + LKD_Bu[a]	×	–	×	–
S-BRITE + LKD_Bu[a]	×	–	×	–
HCM_SC + LKD_Bu[b1]	×	–	×	–
ASTM CH + LKD_Bu[b2, d]	×	–	×	–
ASTM CL + LKD_Bu[b2, d]	×	–	×	–
I-65 + QL_BR[b2]	×	–	×	–
SR-37 + QL_BR[b2]	×	–	×	–
ASTM CH + QL_BR[b2, d]	×	–	×	–
ASTM CL + QL_BR[b2, d]	×	–	×	–
ASTM CH + LKD_Bu[b1, c1]	–	–	×	×
ASTM CH + QL_BR[b1]	–	–	×	×
ASTM CH + LKD_CK2[b1]	–	–	×	×
ASTM CL + LKD_Bu[b1, c1]	–	–	×	×
ASTM CL + QL_BR[b1, c1]	–	–	×	×
ASTM CL + LKD_CK2[b1, c1]	–	–	×	×
S-BRITE + LKD_CK2[b1, c1]	–	–	×	×
S-BRITE + HC85_BV[b1]	–	–	×	×
I-65 + LKD_CK2[b1, c1]	–	–	×	×
I-65 + HC85_BV[b1]	–	–	×	×
HCM_SC + LKD_Bu[b1, c1]	–	–	×	×
HCM_SC + LKD_CK2[b1, c1]	–	–	×	×
Soil-Gypsum Mixes				
S-BRITE + gypsum[b3]	×	×	×	–
ASTM CH + gypsum[b3, d]	×	–	×	–
ASTM CL + gypsum[b3, d]	×	–	×	–

Note: x represents tests performed.
[a] Tests performed at optimum lime dosage.
[b1] Tests performed on overdosed samples, dosage = 5%.
[b2] Tests performed on overdosed samples, dosage = 10%.
[b3] Tests performed with gypsum, dosage = 15%.
[c1] Dilution factor: 1:20 and 1:40
[c2] Dilution factor: 1:3, 1:5, 1:10, 1:20, 1:40, and 1:80
[c3] Dilution factor: 1:3, 1:10, 1:20, 1:40, and 1:80
[c4] Dilution factor: 1:3 and 1:20
[d] Test performed on samples cured for a 7-day period only.

4. TEST RESULTS

A comprehensive testing program was completed to assess the performance of the ITM-510 test used by INDOT for the determination of soluble sulfates in soils. Lime was identified as a potential source of sulfates in subgrades already in existence; thus, select soils collected from different Indiana road construction projects were treated with lime and lime kiln dust. These treated soils were then subjected to sulfate content determination using ITM-510 and ion chromatography (IC) tests.

4.1 Screening of Materials with ITM-510

Table 4.1 summarizes the eight soil samples used for this phase of the investigation, all of which were mixed with either the lime kiln dust obtained from Carmeuse, Buffington plant (LKD_Bu – sample 8 in Table 3.1) or quicklime sample obtained from Carmeuse Black River plant (QL_BR – sample 3 in Table 3.1) or both. The LKD_Bu sample was reported to have an initial sulfate concentration of 1.1% (Carmeuse data specification report), while the QL_BR sample had no sulfate present in it. The initial sulfate concentration of the untreated soils, as well as the sulfate concentration after 7 and 28 days of curing, were determined using ITM-510. In addition to treatment with lime at optimum dosage, a few samples were overdosed to account for uneven lime application in the field. Additional samples were treated with gypsum to prepare a pseudo-sulfate-rich soil. These results are presented in Table 4.1. The tabulated sulfate concentrations are based on tests performed using 1:20 dilution consistent with the ITM-510 method.

The ITM-510 test was able to detect the presence and absence of sulfates in all the soils tested. It was observed that the addition of lime to soils with no initial sulfate resulted in an increased sulfate concentration post treatment. Sulfate concentration measurements made on identical soil-lime mix specimens tested after a 7-day curing period highlighted that the results were consistent and assured repeatability of the tests, as can be seen in Table 4.1. It is important to note that all samples exhibited a consistent increase in sulfate concentration at 28 days compared to 7 days. Another important observation was the effect of lime on soils with an initial sulfate content, addition of lime was found to lower the sulfate concentration. These results formed the basis for the second phase of the testing program, which focused on understanding the effect of dilution rate on sulfate content measurements and the extent to which lime could raise the sulfate concentration of soils with no initial sulfate concentrations. Prior to beginning the second phase, the soil samples were mixed thoroughly and sieved through #4 sieves. The passing portion was then carefully stored in individual Ziploc bags. This was done to homogenize the soils and ensure repeatability. To further validate the initial findings, additional lime samples were incorporated into the second phase.

TABLE 4.1
Results for ITM-510 tests performed on treated samples

	Treatment			Sulfate Concentration Post Treatment, ppm		
Soil	**Chemical**	**Dosage %**	**Sulfate Concentration of Untreated Soil, ppm**	**7-Day Sample 1**	**7-Day Sample 2**	**28-Day Sample**
I-70 S1	LKD_Bu	3	9	253	484	733
I-70 S2	LKD_Bu	3	1,110	796	904	1,247
S-BRITE	LKD_Bu	3	0	100	167	478
HCM_SC	LKD_Bu	5	4,840	2,271	1,782	4,640
ASTM CH	LKD_Bu	10	0	0	0	–
ASTM CL	LKD_Bu	10	1,380	15,737	–	–
S-BRITE	Gypsum	15	0	5,756	6,655	9,536
ASTM CH	Gypsum	15	0	12,570	12,540	–
ASTM CL	Gypsum	15	1,380	14,400	16,818	–
I-65	QL_BR	10	0	0	0	13
SR-37	QL_BR	10	0	0	0	0
ASTM CH	QL_BR	10	0	0	0	–
ASTM CL	QL_BR	10	1,380	198	144	–

TABLE 4.2
Initial sulfate concentration of soil and lime based on 1:20 dilution ratio

		Sulfate Concentration (ppm)	
		ITM-510	**IC**
Soil	ASTM CH	0[2]	44[3]
	ASTM CL[1]	3,254[3]	3,503[3]
	S-BRITE	0	27
	I-65	0	248
	HCM_SC	5,087[2]	7,339[2]
Lime	LKD_Bu	18,240[4]	18,055[4]
	QL_BR	0	197
	LKD_CK2	18,696[3]	23,677[3]
	HC85_BV	0	86

[1] The earlier reported value for ASTM CL was found to be lower, the results presented here are after thorough mixing to ensure homogenization. The average value reported does not include the results from phase 1.

[2] Average of two samples.

[3] Average of three samples.

[4] Average of four samples.

The selected soil and lime samples for the second phase are presented in Table 4.2.

4.2 Evaluation of TGA and XRD Tests for Sulfate Content Determination

A preliminary analysis of the performance of the ITM test method was conducted utilizing mineralogical tests such as XRD and TGA. The free calcium introduced during lime treatment reacts with the soluble sulfate present in the soil to form calcium sulfate (gypsum). Gypsum is also a common mineral present in naturally occurring sulfate-rich soils. The decomposition of gypsum is very distinctly observed in a TGA test. Gypsum ($CaSO_4.2H_2O$) loses its hydration water in two phases, first between 60°C–150°C by converting gypsum into bassanite ($CaSO_4.0.5H_2O$). The second loss occurs between 105°C–240°C when bassanite converts into anhydrous calcium sulfate (Ghazi Wakili et al., 2007). TGA tests on sulfate-rich soil show the following four distinct weight loss markers: loss of water adsorbed by clays at ~100°C, decomposition of gypsum at ~150°C, decomposition of ammonium sulfate at ~240°C and dehydroxylation of kaolinite at ~500°C (Jung & Santagata, 2014). These weight-loss markers were used to estimate the sulfate in select soils both before and after treatment with lime.

Figures 4.1 and 4.2 shows XRD and TGA plots for the S-BRITE soil treated with gypsum. The peaks of gypsum were clearly identifiable in the XRD spectra of the treated samples. The weight loss and derivative of weight loss with temperature plots obtained from the TGA test show a clear weight loss occurring in the gypsum as well as in the treated samples at around 150°C. The test results conducted on soil-gypsum mixes revealed that although the ITM test identified the presence of sulfates, the sulfate concentration estimated from the TGA was higher than the quantity of sulfate introduced in the soil. This is since the mass loss from observed in the TGA tests between 100°C and 150°C reflects the loss of both the hydration water of gypsum (or of its partially dehydrated forms) as well as of the water added during compaction. As a result, the TGA test was considered unreliable, and this testing methodology was not pursued further. Additional TGA tests performed on soil-lime mixes are presented in Appendix A.

4.3 Effect of Salt Solubility on Sulfate Concentration

Naturally occurring sulfate soils are composed of different minerals. The solubility of a sulfate salt in water governs the sulfate concentration in the soil. Two sulfate salts with different water solubilities, gypsum and sodium sulfate, were selected to quantify the concentration of soluble sulfate ions. At 20°C, ~2 g of gypsum and ~49 g of sodium sulfate, respectively, would dissolve completely in 1,000 ml of water. The ITM test requires 20 g of soil mixed in 400 ml of water.

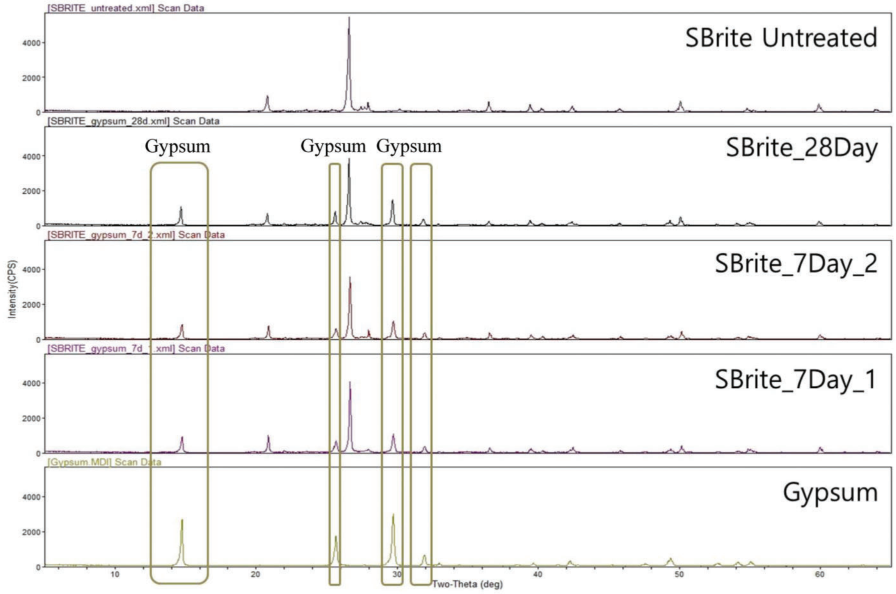

Figure 4.1 XRD plots for S-BRITE and gypsum.

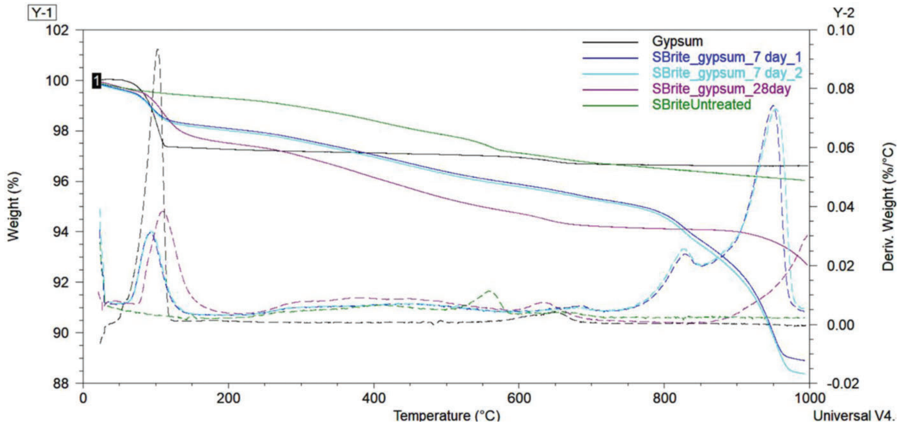

Figure 4.2 TGA plots for S-BRITE and gypsum.

Considering the impurities in lime as one of the two salts mentioned, the following calculations demonstrate the percentage of sulfate that can be dissolved in water during the test.

A lime sample containing a 3% sulfate impurity means that a 20 g sample of lime used for the test has 0.6 g of sulfate. Based on the molecular weight of sulfate and salts, the 0.6 g of sulfate is either due to 1.075 g of gypsum or 0.8875 g of sodium sulfate. The solubilities of these salts and the amount of water used for the test show that only ~0.84 g of gypsum can dissolve completely in 400 ml of water. This demonstrates that impurities present as gypsum may require more water in the test to determine the sulfate concentration accurately. However, the amount of water used may be sufficient for more soluble salts.

In a similar manner, to understand the sulfate content of tests performed on soil-lime mix samples, consider a lime with 3% sulfate.

Lime used = 5% by weight of soil.

Average sample dry weight = 100 g.

Sample for ITM test = 20 g.

Approximate lime for ITM test = 1 g.

Impurities in lime = 0.03 g (0.05375 g gypsum or 0.044375 g sodium sulfate).

Based on the amount of lime in the sample, the test procedure has enough water to completely dissolve the sulfate present as either gypsum or sodium sulfate. In other words, the above calculations demonstrate that a dilution factor of 1:20 is sufficient to dissolve all the sulfate ions present in soil-lime mixes. However, for pure lime samples used in the ITM test, only about 80% of the sulfate (as gypsum impurity) would be soluble with a 1:20 dilution. Therefore, lime samples with high sulfate content/impurities may require a higher dilution rate to yield an accurate estimate of their sulfate concentration.

4.4 Comparison of Sulfate Content Determined by ITM-510 and Ion Chromatography Tests

The accuracy of the sulfate content determined from the ITM-510 test, was verified through additional soil-lime mix specimens, where the sulfate concentration was determined using both ITM-510 and ion chromatography. Five soils and four lime samples with varying initial sulfate concentrations were used to prepare compacted samples (Table 4.2). It should be noted that the lime manufacturer reported sulfate concentration for the LKD samples using a different dilution ratio (1:3, based on personal communication) and a different test method (ASTM D516). As the optimum lime dosage for the select soil and lime mixes was found to be less than 5%, all the treated samples were overdosed with 5% lime by weight of soil. Tests on the soil-lime mixes were performed using 1:20 and 1:40 dilution ratios. Dilution ratios between 1:3 and 1:80 was used for the tests on the lime samples. This was done to investigate the effect of dilution ratio on the accuracy of the ITM tests.

4.4.1 Effect of Dilution Ratio on Lime Samples

Figure 4.3 and Figure 4.4 show the results of sulfate concentrations determined as per the two tests for two different LKD samples (LKD_Bu and LKD_CK2). The tests were performed by keeping the amount of lime constant while increasing the amount of water added to reach the different dilution ratios. The figures show that increasing the amount of water results in an increased sulfate concentration. The horizontal solid line depicted on these figures identifies the concentration reported by the manufacturer. The results show that, for lime samples, the

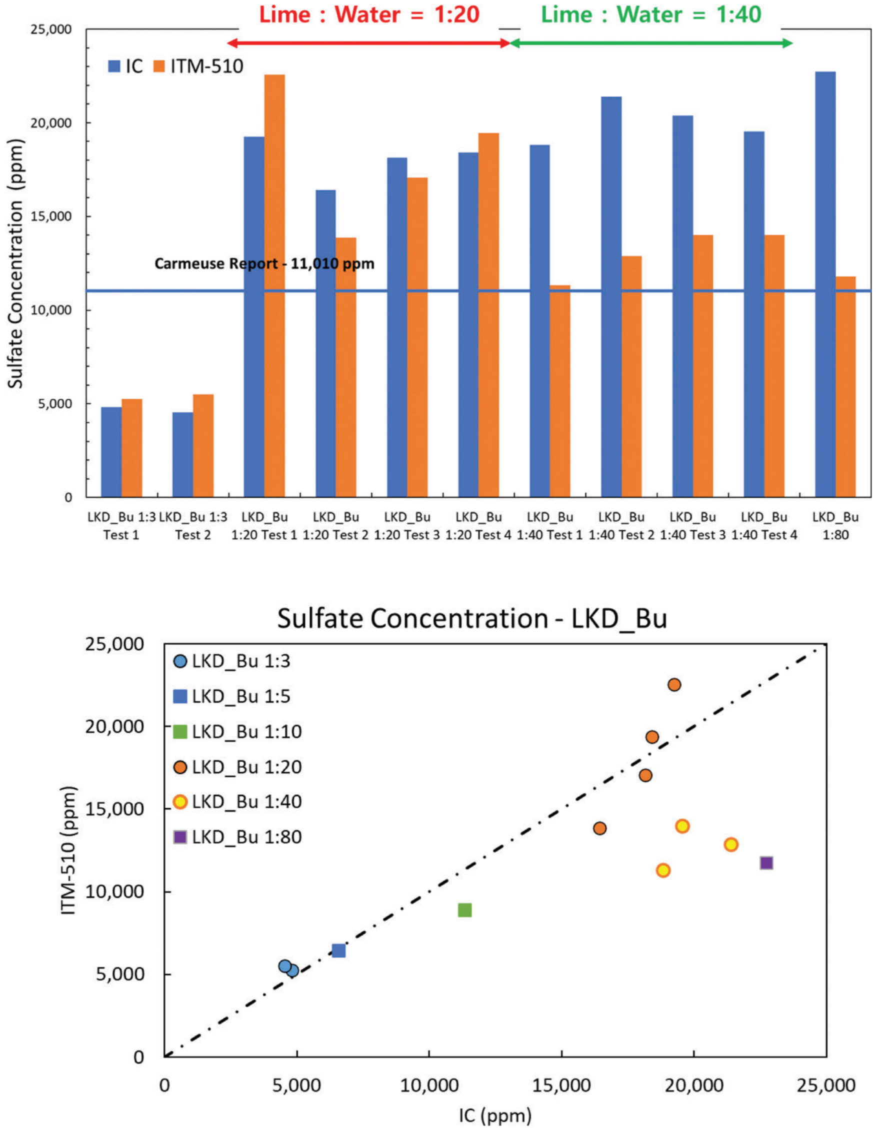

Figure 4.3 Effect of dilution ratio on lime kiln dust from Buffington.

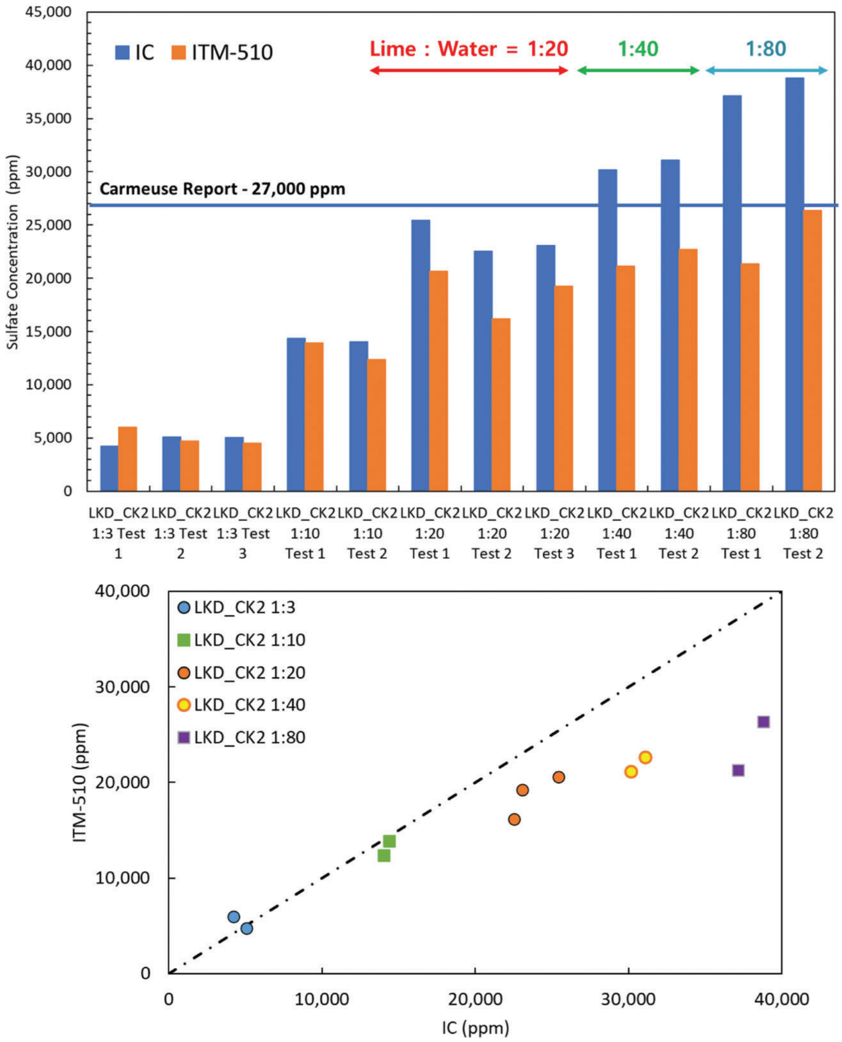

Figure 4.4 Effect of dilution ratio on lime kiln dust from CheneyK2.

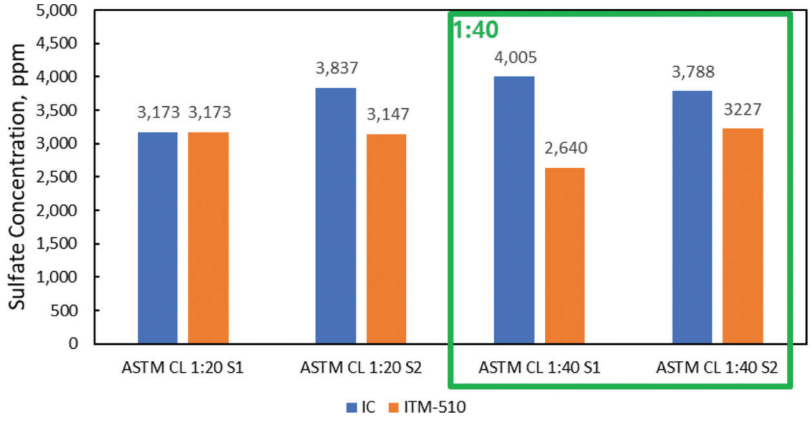

Figure 4.5 Comparison of ITM-510 and IC test results for ASTM_CL.

sulfate content depends on the dilution ratio. Further, the ITM test tends to underpredict the lime content. The tests compare well at low sulfate concentrations and low dilution; however, large discrepancies are observed at high dilution (discussed later).

4.4.2 Effect of Dilution Ratio on Soil-Lime Mix Samples

Figures 4.5 and 4.6 show the results of tests performed at 1:20 and 1:40 dilution rate on soils ASTM CL (average sulfate concentration based on IC: 1:20 dilution = 3,505; 1:40 dilution = 3,897) and

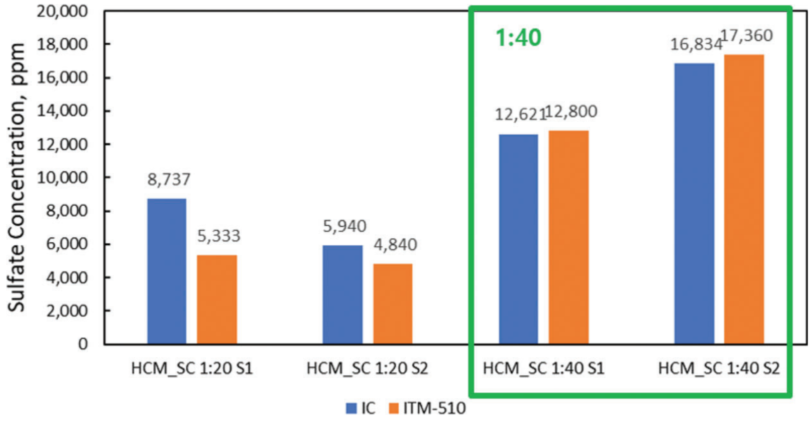

Figure 4.6 Comparison of ITM-510 and IC test results for HCM_SC.

HCM_SC (average sulfate concentration based on IC: 1:20 dilution = 7,339; 1:40 dilution = 14,728). The results indicate that, for a soil with low sulfate concentration (<5,000 ppm), such as ASTM CL, the 1:20 dilution ratio is sufficient to extract all sulfate. However, for higher sulfate concentrations, a higher dilution rate may be needed to dissolve all the sulfate ions present. The figures also show that the ITM results compare well with the IC results, although they generally tend to yield results somewhat lower than IC. It is also important to note here that the results for ASTM CL presented are higher than previously reported (Table 4.1). These results were obtained after thorough mixing to ensure material homogeneity.

Figure 4.7 compares the test results from the tests on the ASTM CL soil treated with three different limes (LKD_Bu, LKD_CK2, and QL_BR), two of which contain a significant % of sulfate (see Table 4.2). The figure shows that lime treatment does not significantly increase the sulfate present in the soil, but in some cases (ASTM + QL_BR and results of 1:20 dilution for ASTM CL+LKD_CK2) leads to a reduction in the measured sulfate content. This could be attributed to the formation of sulfate compounds with reduced solubility. The current project did not focus on the changes in soil mineralogy due to lime addition, and thus, this hypothesis cannot be verified. There is also little difference in the measured sulfate with an increase in the curing period from 7 to 28 days, which contrasts with the results observed in the preliminary testing phase. As observed above, while providing slightly lower sulfate concentrations, the ITM results are generally consistent with those of the IC tests.

Figure 4.8 presents the results for tests on HCM_SC soil upon treatment with two different lime kiln dust samples (LKD_Bu and LKD_CK2). The sulfate concentration was found to be slightly higher for the treated samples when compared with the sulfate concentration of the untreated soil. The HCM_SC soil was also found to exhibit an increased sulfate concentration with a higher dilution ratio (Figure 4.6). Although the results are consistent across the two LKDs used, a more in depth understanding of the geochemistry involved between soil and lime reactions is necessary to derive any major conclusions.

Figure 4.9 discusses the tests performed on soils with no initial sulfate concentration (S-BRITE, ASTM CH and I-65), after treatment with lime containing <3% sulfate concentration (LKD_CK2 and LKD_Bu, as per Carmeuse record). The results indicate that lime is a source for sulfates in the soils. It is important to note that INDOT specification allows for 5% sulfate in lime kiln dust used. This means that a soil treated with such lime could easily surpass the current sulfate threshold of 1,000 ppm (Figure 4.9). Again, ITM results were found to be slightly lower than IC results.

Figure 4.10 shows a comparison between the IC and ITM-510 test results. Regression results for the two set of tests (dilution ratio 1:20 in red and 1:40 in green) indicate a general agreement between the IC and ITM-510 test results. The figure shows that ITM provides a good estimate for sulfates when the concentration is below 5,000 ppm. For samples with higher concentrations, the ITM results, in general, underestimate the amount of sulfates present. The discrepancies between the two tests were larger for pure lime samples (Figures 4.3 and 4.4), but not as pronounced in soils treated with lime. The results presented in this plot, along with additional tests performed on soils and lime mixes, both with no initial sulfate concentration are presented in Appendix B.

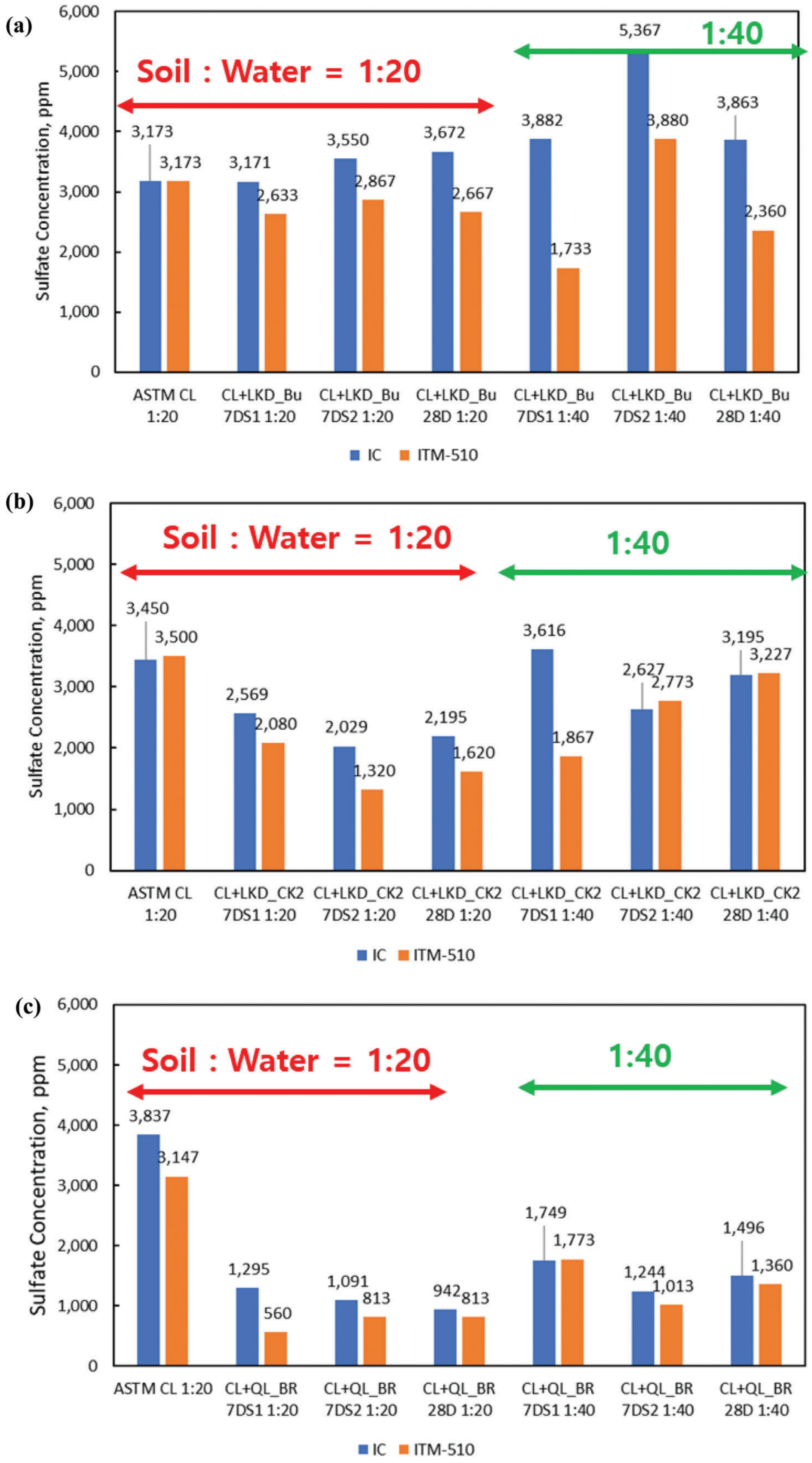

Figure 4.7 Sulfate test results for ASTM CL soil: (a) treated with LKD_Bu; (b) treated with LKD_CK2; (c) treated with QL_BR (7DS = samples cured for 7 days; 28D = samples cured for 28 days).

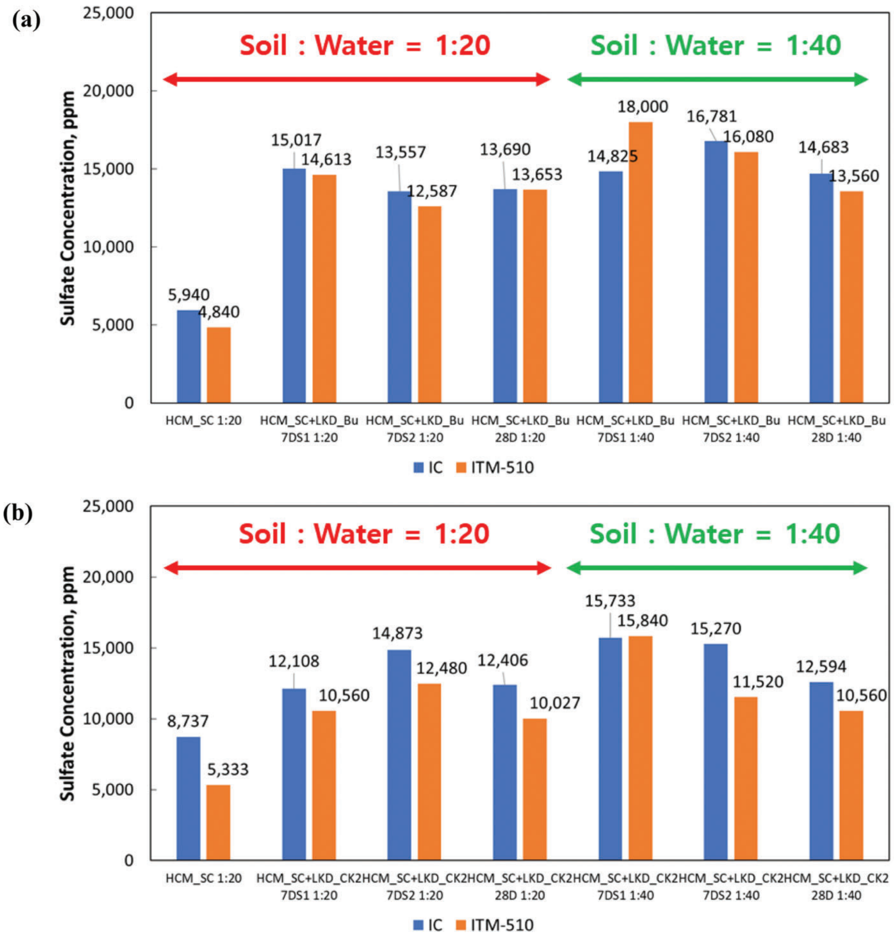

Figure 4.8 Sulfate test results for HCM_SC soil: (a) treated with LKD_Bu; (b) treated with LKD_CK2 (7DS = samples cured for 7 days; 28D = samples cured for 28 days).

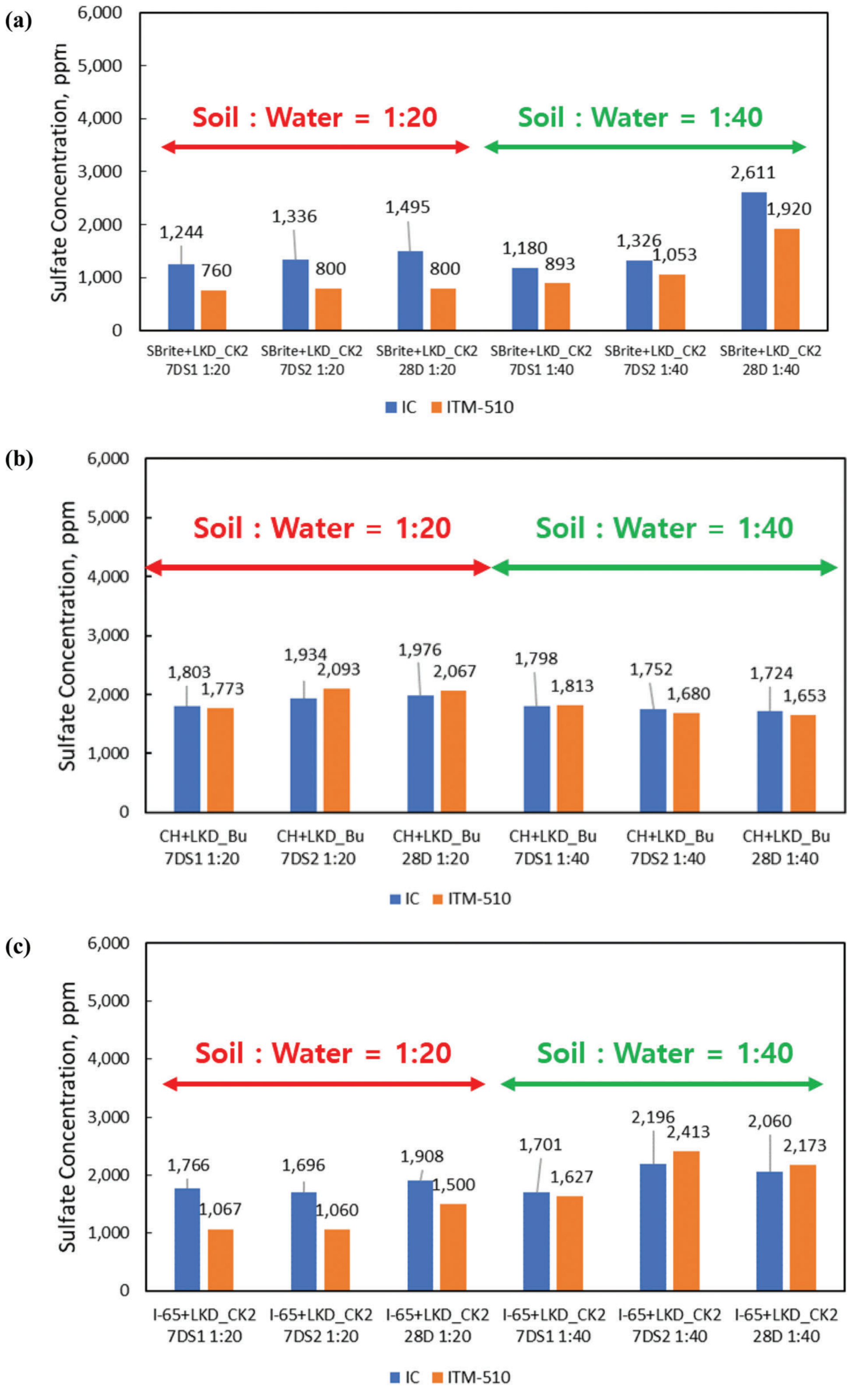

Figure 4.9 Sulfate concentration for treated soils with no initial sulfate concentration: (a) S-BRITE treated with LKD_CK2; (b) ASTM CH soil with LKD_Bu; (c) I-65 soil with LKD_CK2 (7DS = samples cured for 7 days; 28D = samples cured for 28 days).

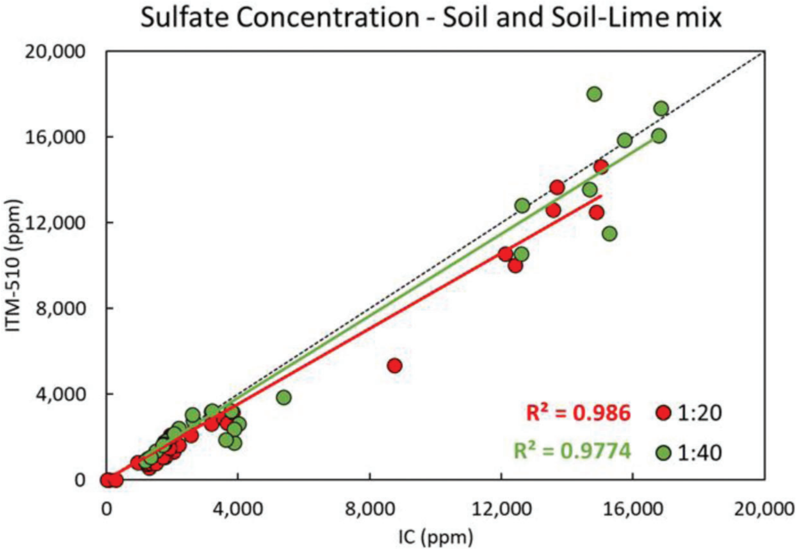

Figure 4.10 Comparison of ITM-510 and IC test results for soil-lime mixes.

5. SUMMARY, CONCLUSIONS, AND RECOMMENDATIONS FOR IMPLEMENTATION

5.1 Summary

The subgrade layer is essential for the long-term performance of pavement systems, but problem soils can create challenges in design, construction, and performance. Chemical stabilization, using agents like lime or cement, is a common practice adopted to improve the mechanical properties of such soils. A problem that INDOT is encountering when rehabilitating or maintaining roads, where the subgrade has been treated with lime, is the detection of sulfates in the existing subgrade. This may result in costly soil removal despite the subgrade showing no signs of distress.

In the current research work, the test protocol used by INDOT to determine the sulfate concentration of soils was reviewed and compared with other existing sulfate content determination tests. In addition, the reliability of the test was assessed by performing tests on subgrade soils collected from different road construction projects and treating them with lime and additives used by INDOT for subgrade treatment. The sulfate content was determined by ITM-510, a colorimetric test method utilized by INDOT, and ion chromatography, an analytical test method used to separate, identify, and quantify ions in solution.

5.2 Conclusions

The following conclusions have been drawn from the research performed.

1. Lime kiln dust samples used for subgrade treatment may contain sulfates as impurities and are most likely the source of sulfates in the existing treated subgrades.
2. Due to difficulties in accurately distinguishing between the weight loss caused by the decomposition of gypsum and the loss of free water in treated samples, thermogravimetric tests are found to provide unreliable estimates of sulfate content.
3. Ion chromatography (IC) and inductively coupled plasma-optical emission spectroscopy (ICP-OES) can be used as ground-truths to quantify sulfates in soils.
4. The ITM-510 test methodology has been found to be adequate for identifying the presence of sulfates and for providing accurate values of sulfate concentration, provided that the sulfate concentration does not exceed 5,000 ppm. For larger sulfate concentrations, the errors may be large.
5. For samples containing high amounts of sulfate (>5,000 ppm), a dilution rate higher than the one used in ITM-510 tests (>1:20) may be necessary to dissolve all available sulfates.
6. Lime samples with a reported sulfate concentration of about 3% are found to raise the sulfate concentration of soils with no initial sulfate content by as much as 2,000 ppm upon treatment. The current INDOT criterion for allowable sulfate in subgrades is 1,000 ppm.

5.3 Recommendations for Implementation

Based on the literature reviewed for sulfate test protocols and the results obtained from the laboratory tests, the following recommendations for implementation are made.

1. The ITM-510 test protocol works well and provides an accurate estimate of sulfate concentration in soils. INDOT should continue to use the test to determine the sulfate concentration in subgrade soils.
2. Samples showing high sulfate levels (>5,000 ppm) should be tested at dilutions higher than 1:20 to ensure that all soluble sulfate is dissolved and accurately measured.
3. The current limitation on soluble sulfate content of lime by-products in Article 913 of the INDOT standard specifications, set at 5%, should be reduced to 3%.
4. This research did not cover the mechanical performance of lime-treated subgrades testing positive for sulfates. This issue is outside the scope of the project. It is,

however, strongly recommended to investigate this problem, given the large number of roads that have been treated with lime and the potential economic implications of discarding the subgrade soils because of their high sulfate content.

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APPENDICES

Appendix A. Thermogravimetric Test Results

Appendix B. ITM-510 and IC Test Results

APPENDIX A. THERMOGRAVIMETRIC TEST RESULTS

The following table lists the various soils, lime products, gypsum, and treated soil samples subjected to thermogravimetric (TGA) analysis. The associated plots are attached after this table.

Table A.1 Samples subjected to thermogravimetric analysis

S. No.	Sample
1	TGA for I-70 Sample 1 (untreated)
2	TGA for I-70 Sample 1 treated with LKD_Bu (curing 7 day) Test 1
3	TGA for I-70 Sample 1 treated with LKD_Bu (curing 7 day) Test 2
4	TGA for I-70 Sample 1 treated with LKD_Bu (curing 28 day)
5	TGA for I-70 Sample 2 (untreated)
6	TGA for I-70 Sample 2 treated with LKD_Bu (curing 7 day) Test 1
7	TGA for I-70 Sample 2 treated with LKD_Bu (curing 7 day) Test 2
8	TGA for I-70 Sample 2 treated with LKD_Bu (curing 28 day)
9	TGA for lime kiln dust from Buffington plant (LKD_Bu)
10	TGA for quicklime from Black River (QL_BR)
11	TGA for SBRITE (untreated)
12	TGA for SBRITE soil treated with gypsum (curing 7 day) Test 1
13	TGA for SBRITE soil treated with gypsum (curing 7 day) Test 2
14	TGA for gypsum kept at room temperature
15	TGA for gypsum kept at 75°C
16	TGA for gypsum kept at 110°C
17	TGA for ASTM CH soil (untreated)
18	TGA for ASTM CH soil treated with gypsum (curing 7 day) Test 1
19	TGA for ASTM CH soil treated with gypsum (curing 7 day) Test 2
20	TGA for ASTM CH soil treated with QL_BR (curing 7 day) Test 1
21	TGA for ASTM CH soil treated with LKD_Bu (curing 7 day) Test 1
22	TGA for ASTM CL soil (untreated)
23	TGA for ASTM CL soil treated with gypsum (curing 7 day) Test 1
24	TGA for ASTM CL soil treated with gypsum (curing 7 day) Test 2
25	TGA for ASTM CL soil treated with QL_BR (curing 7 day) Test 1
26	TGA for ASTM CL soil treated with LKD_Bu (curing 7 day) Test 1
27	TGA for HCM_SC (untreated)
28	TGA for HCM_SC treated with LKD_Bu (curing 7 day) Test 1
29	TGA for HCM_SC treated with LKD_Bu (curing 7 day) Test 2
30	TGA for HCM_SC treated with LKD_Bu (curing 28 day)
31	TGA for SR-37 (untreated)
32	TGA for SR-37 treated with QL_BR (curing 7 day) Test 1
33	TGA for SR-37 treated with QL_BR (curing 7 day) Test 2
34	TGA for SR-37 treated with QL_BR (curing 28 day)
35	TGA for SBRITE treated with LKD_Bu (curing 7 day) Test 1
36	TGA for SBRITE treated with LKD_Bu (curing 7 day) Test 2
37	TGA for SBRITE treated with LKD_Bu (curing 28 day)
38	TGA for I-65 (untreated)
39	TGA for I-65 treated with QL_BR (curing 7 day) Test 1
40	TGA for I-65 treated with QL_BR (curing 7 day) Test 2

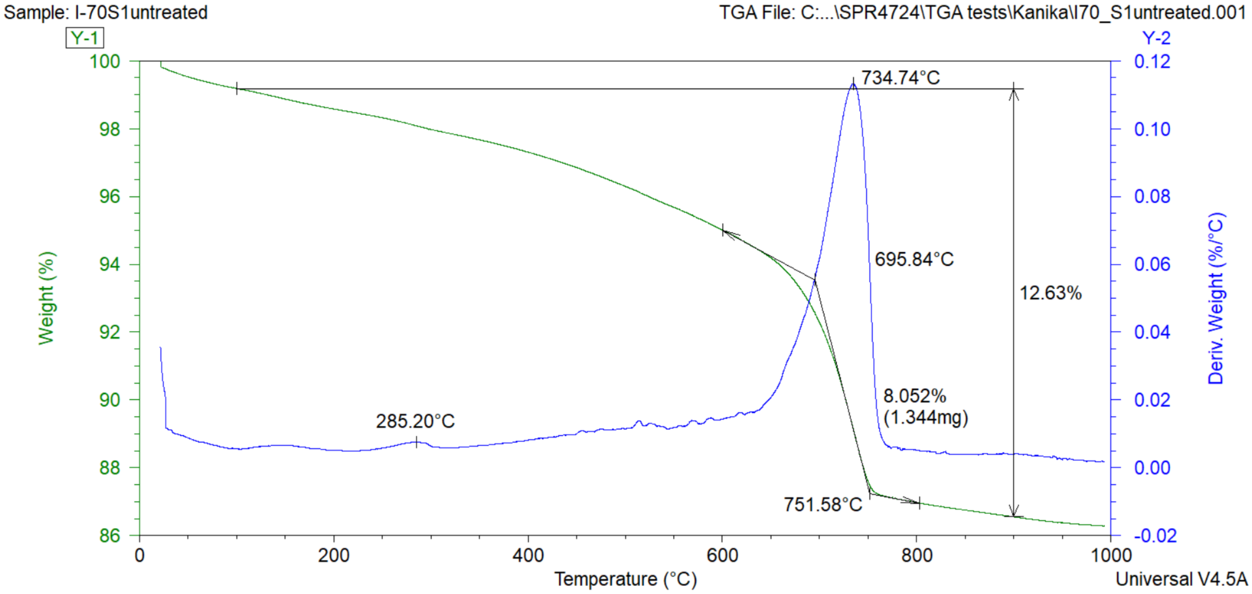

Figure A.1 TGA for I-70 Sample 1 (untreated).

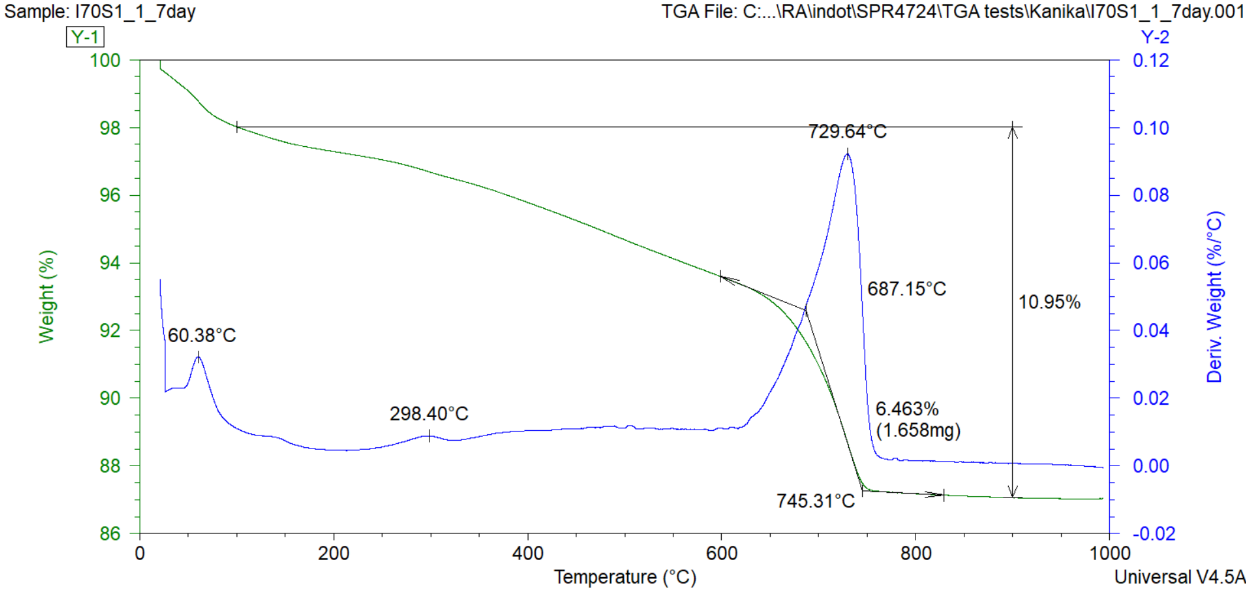

Figure A.2 TGA for I-70 Sample 1 treated with LKD_Bu (curing 7 days) Test 1.

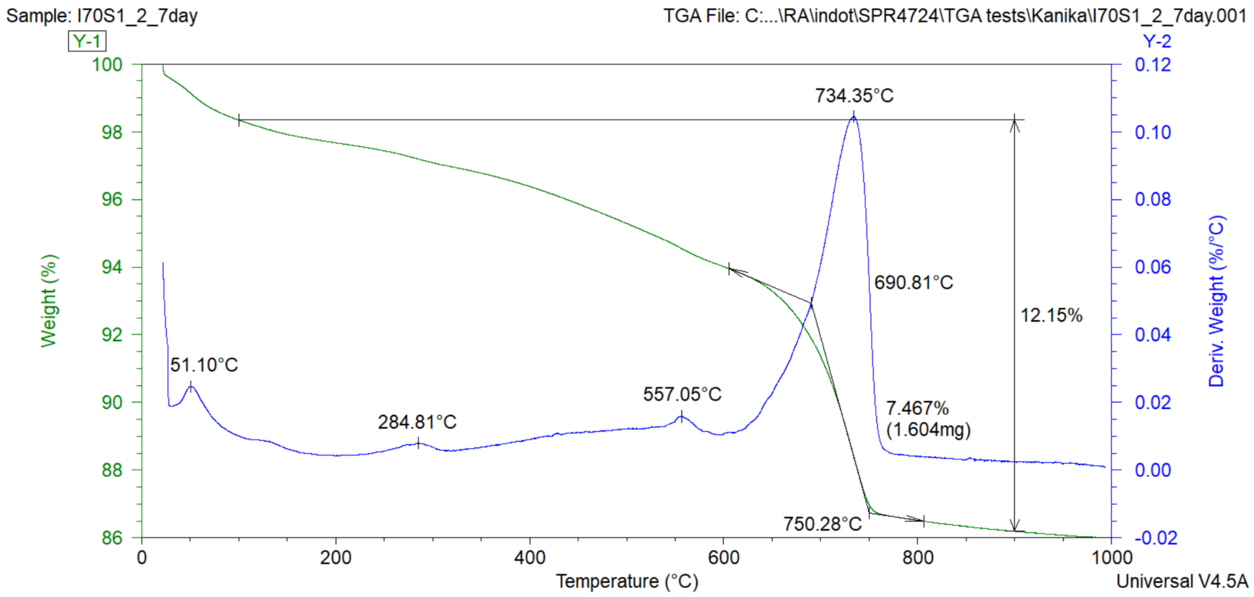

Figure A.3 TGA for I-70 Sample 1 treated with LKD_Bu (curing 7 days) Test 2.

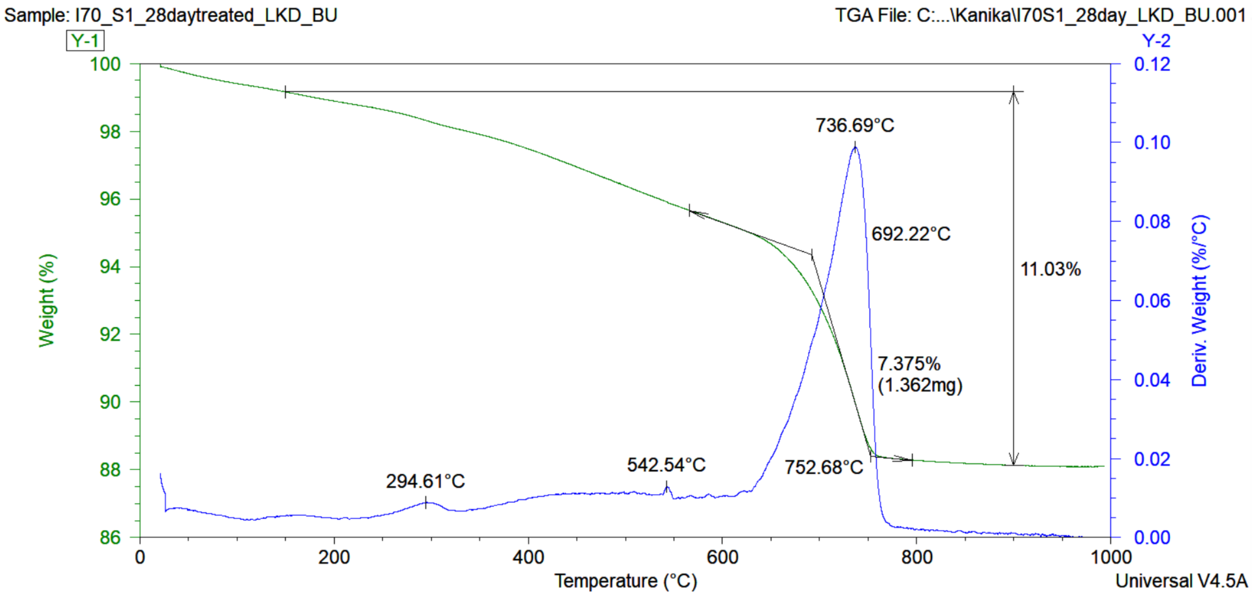

Figure A.4 TGA for I-70 Sample 1 treated with LKD_Bu (curing 28 days).

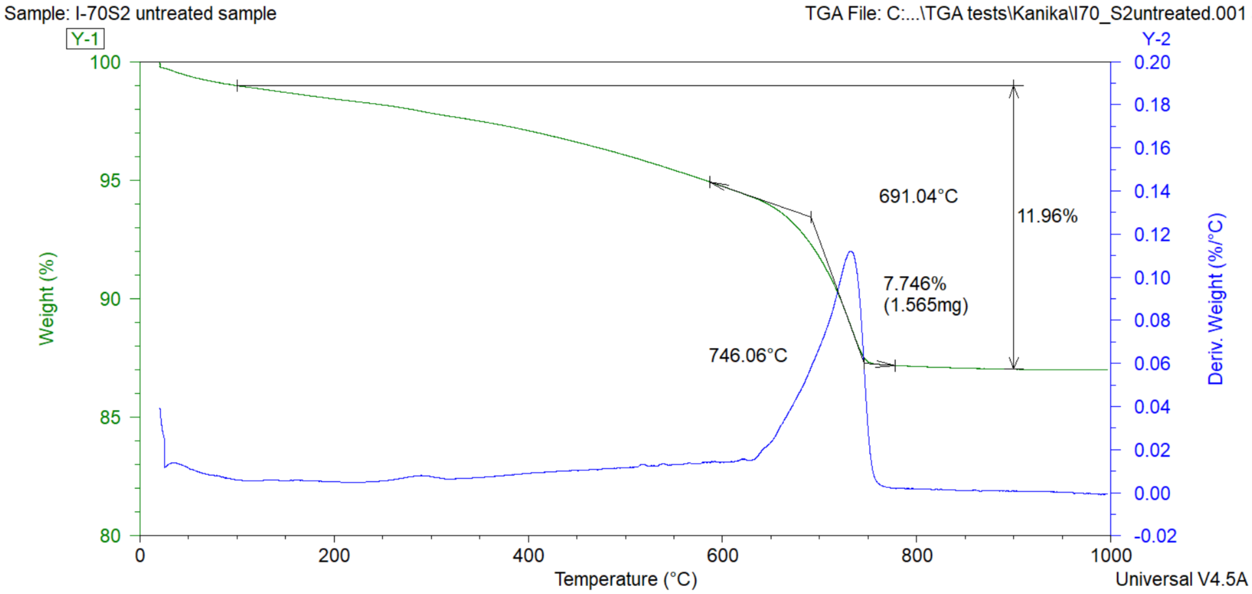

Figure A.5. TGA for I-70 Sample 2 (untreated).

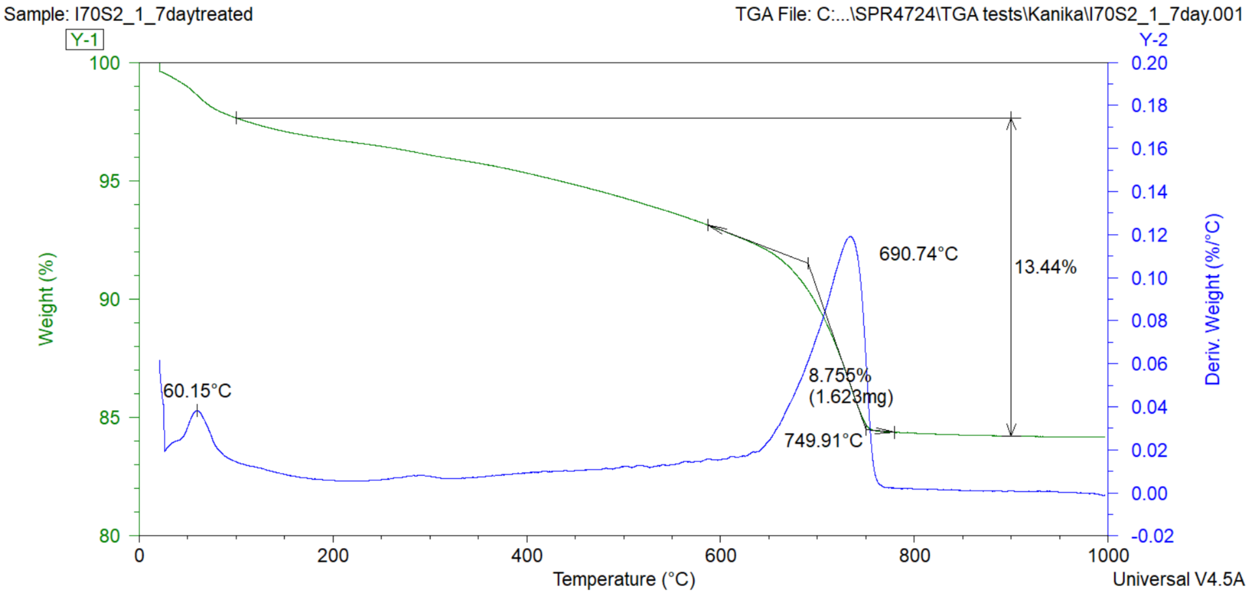

Figure A.6 TGA for I-70 Sample 2 treated with LKD_Bu (curing 7 days) Test 1.

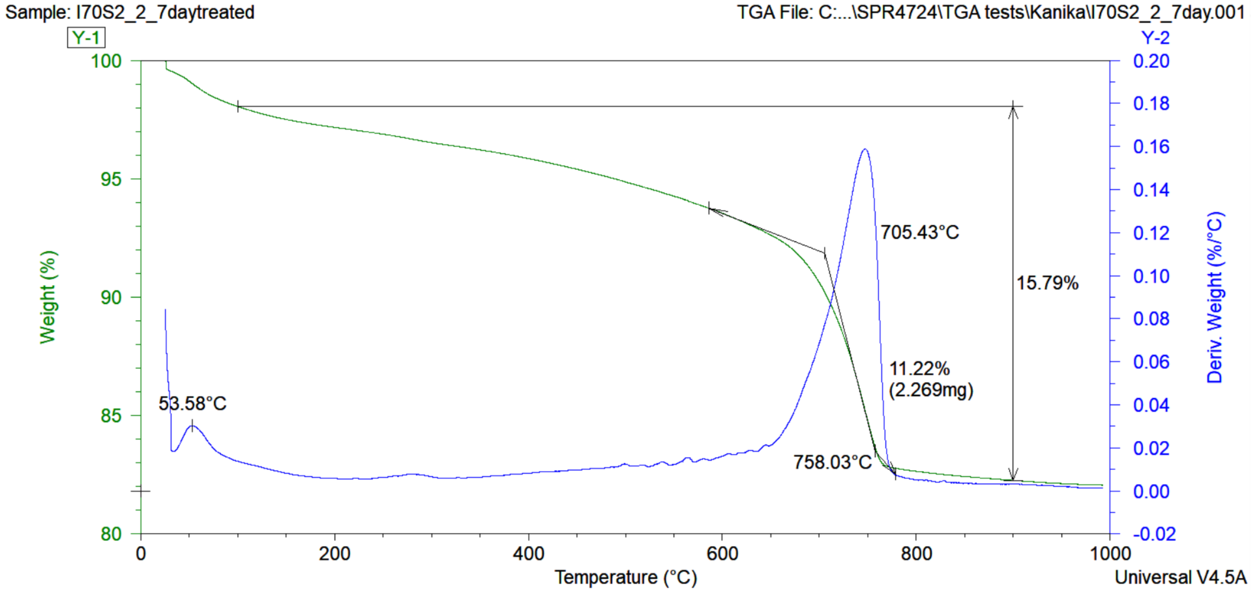

Figure A.7 TGA for I-70 Sample 2 treated with LKD_Bu (curing 7 days) Test 2.

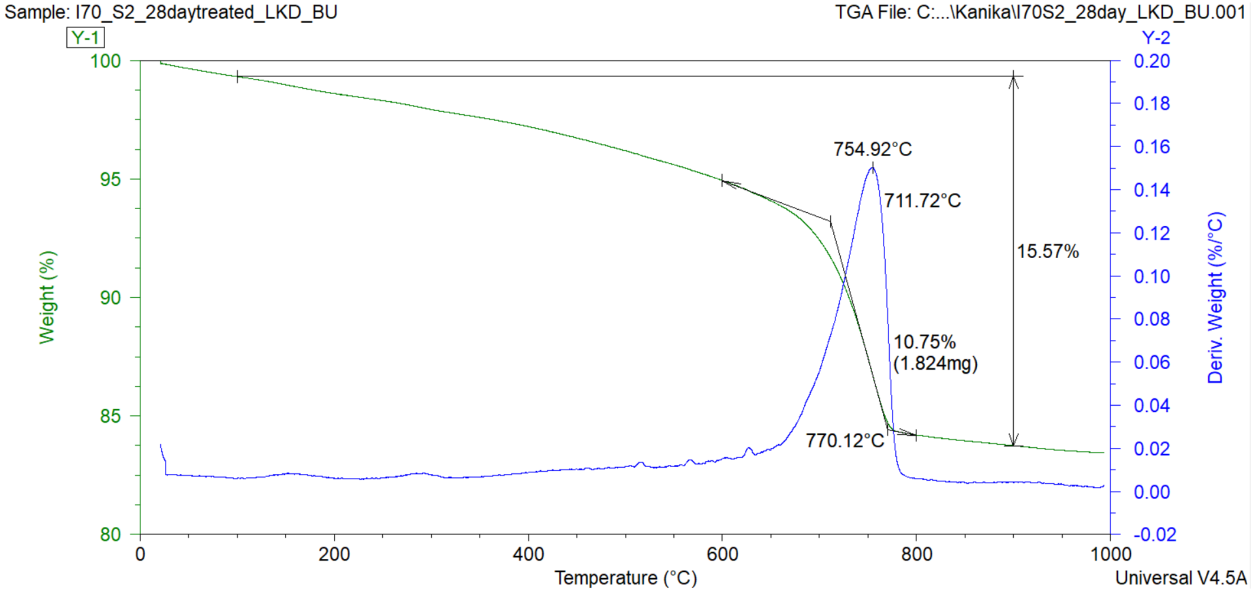

Figure A.8 TGA for I-70 Sample 2 treated with LKD_Bu (curing 28 days).

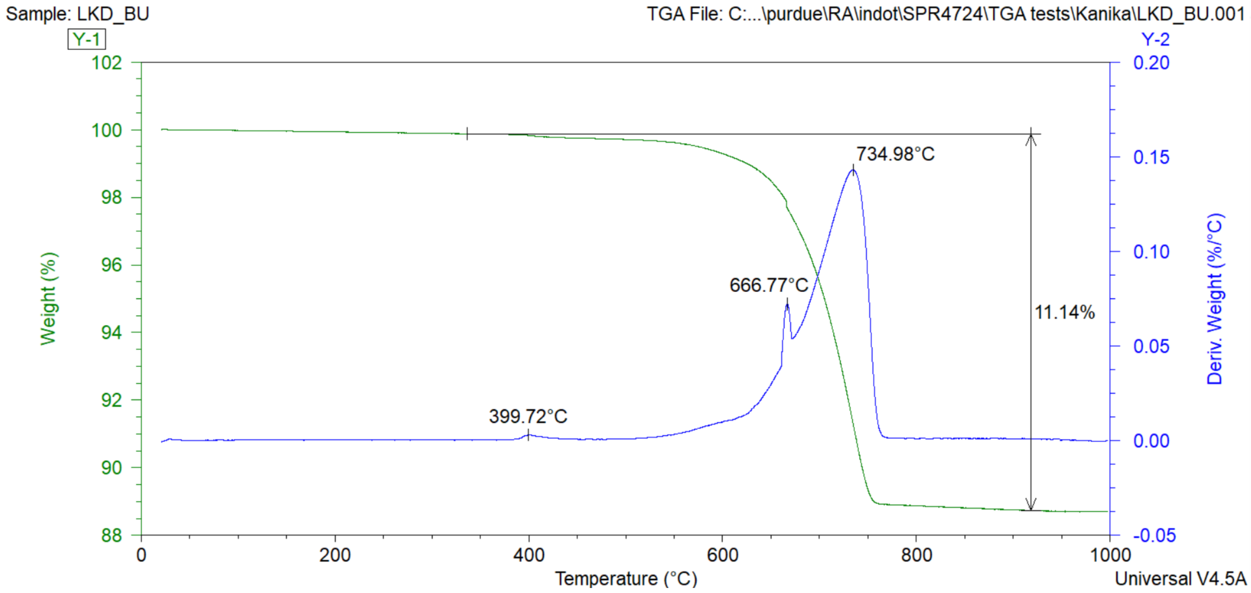

Figure A.9 TGA for lime kiln dust from Buffington plant (LKD_Bu).

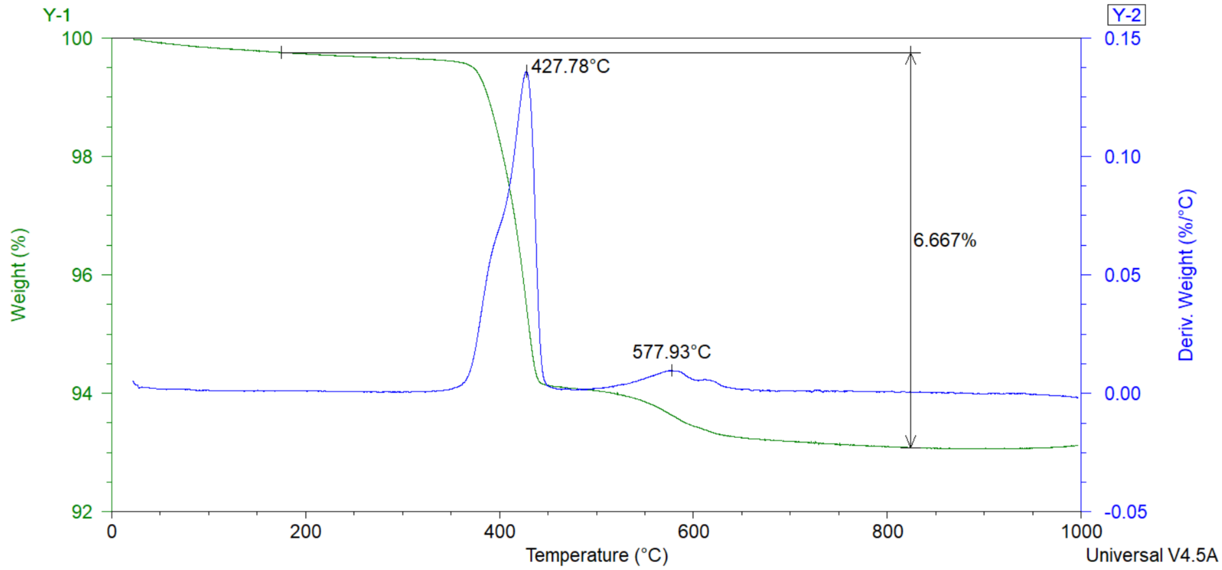

Figure A.10 TGA for quicklime from Black River (QL_BR).

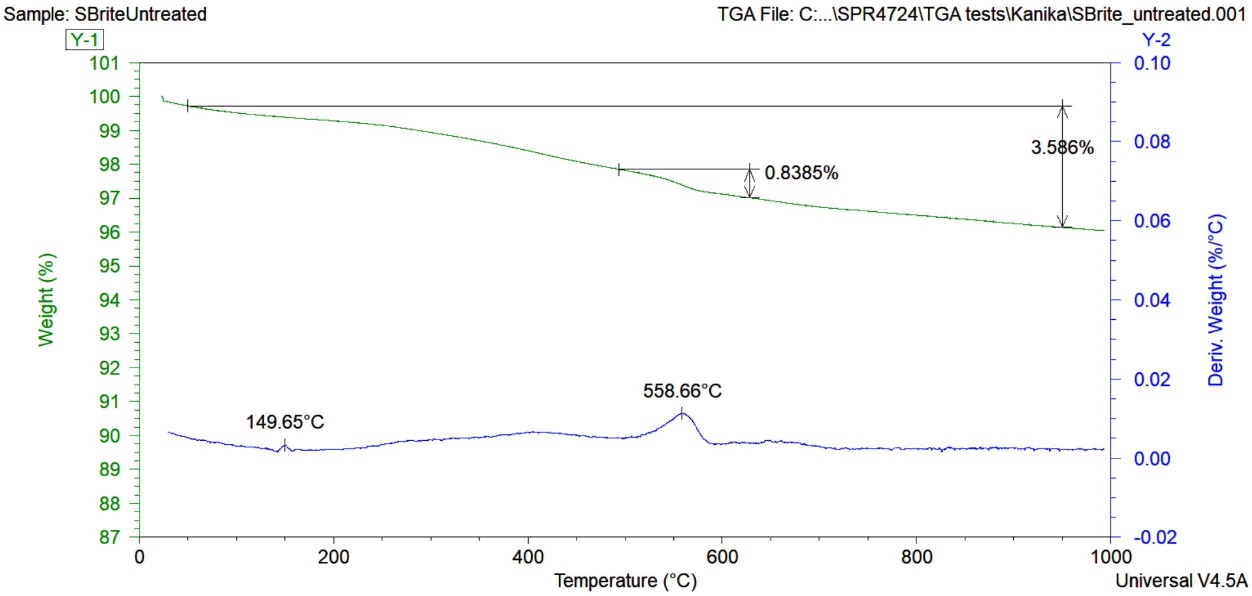

Figure A.11 TGA for SBRITE (untreated).

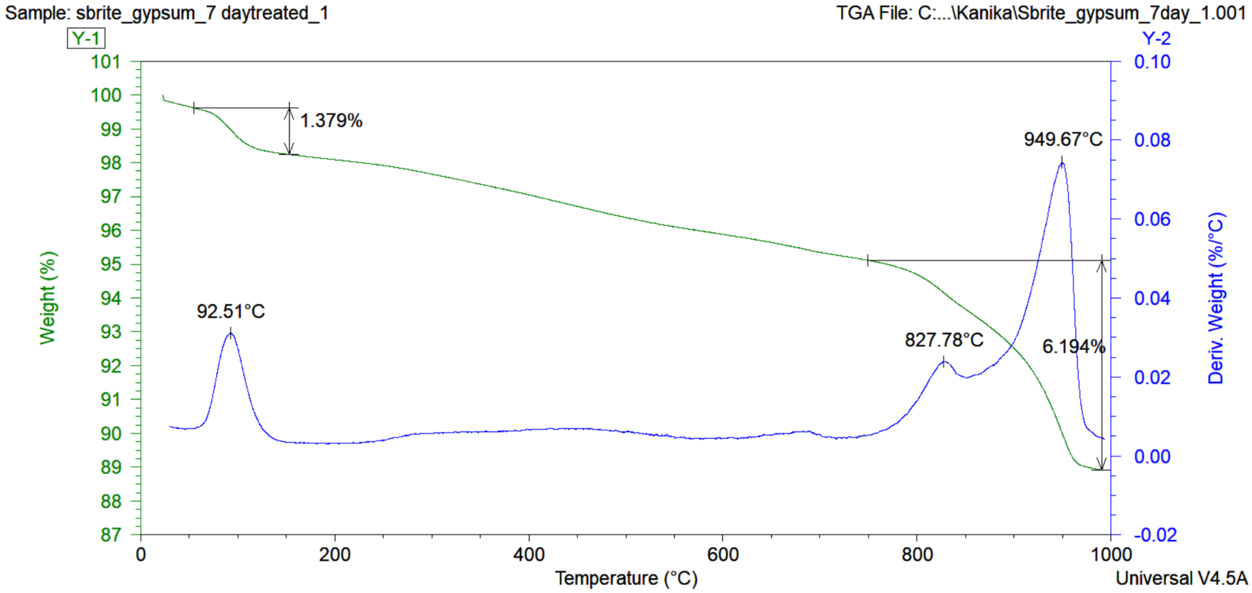

Figure A.12 TGA for SBRITE soil treated with gypsum (curing 7 days) Test 1.

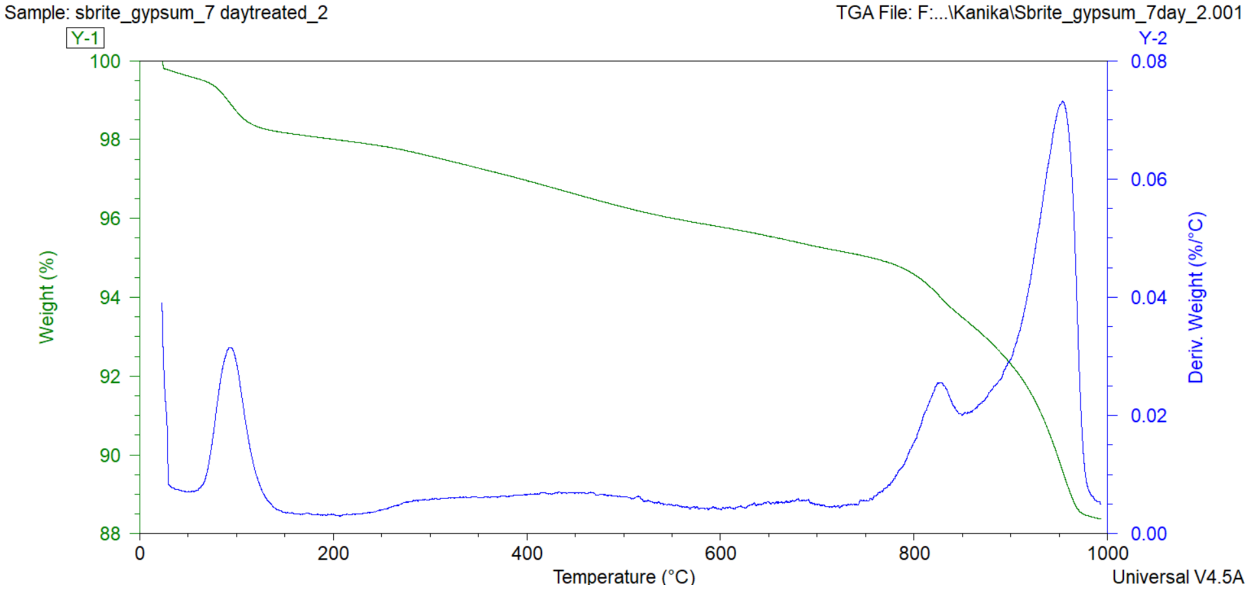

Figure A.13 TGA for SBRITE soil treated with gypsum (curing 7 days) Test 2.

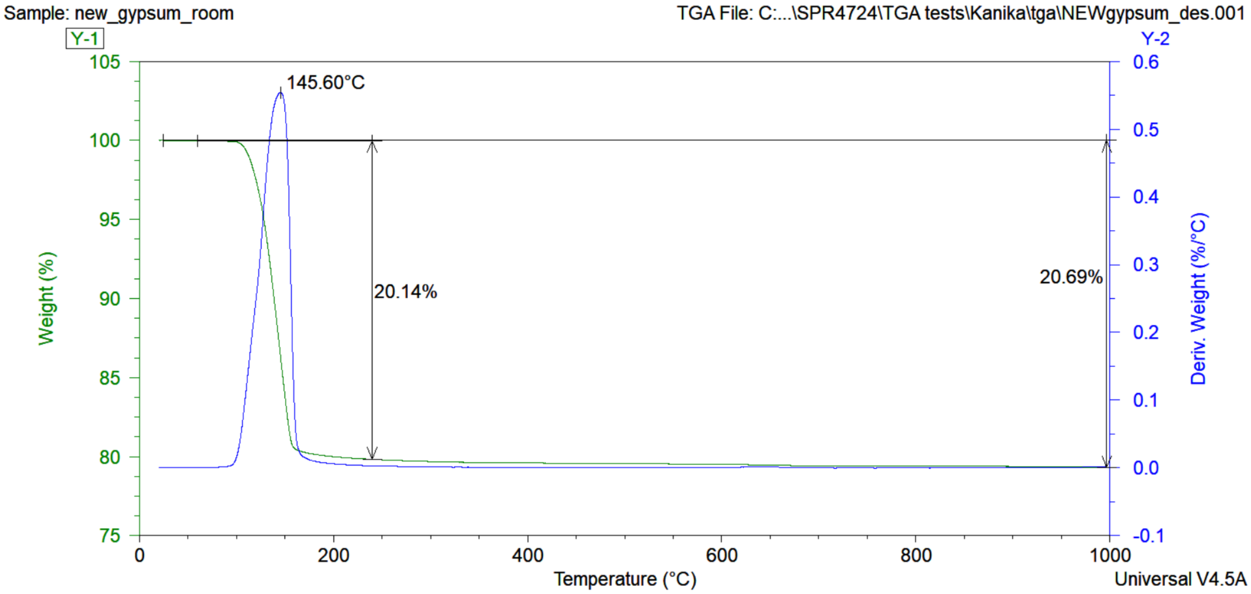

Figure A.14 TGA for gypsum kept at room temperature.

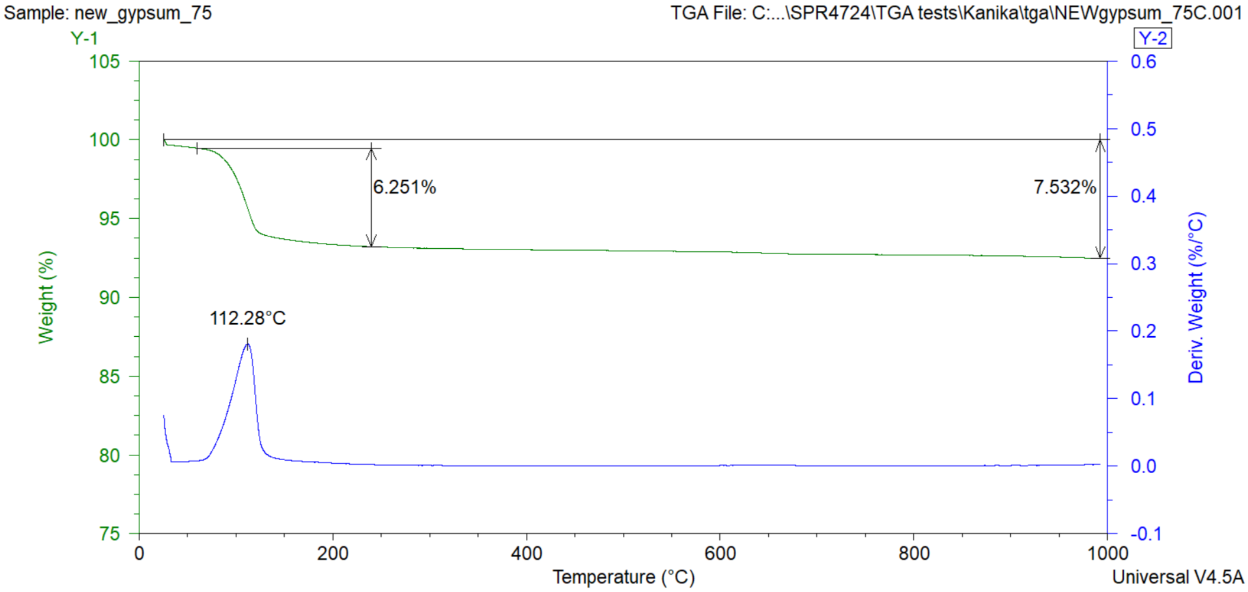

Figure A.15 TGA for gypsum kept at 75°C.

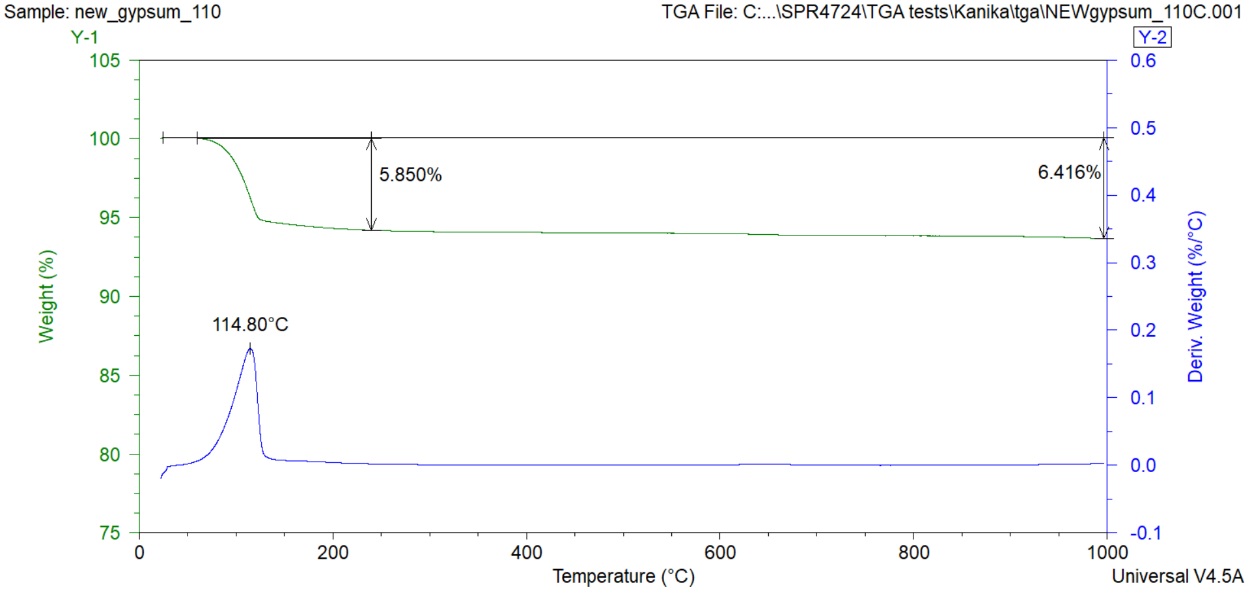

Figure A.16 TGA for gypsum kept at 110°C.

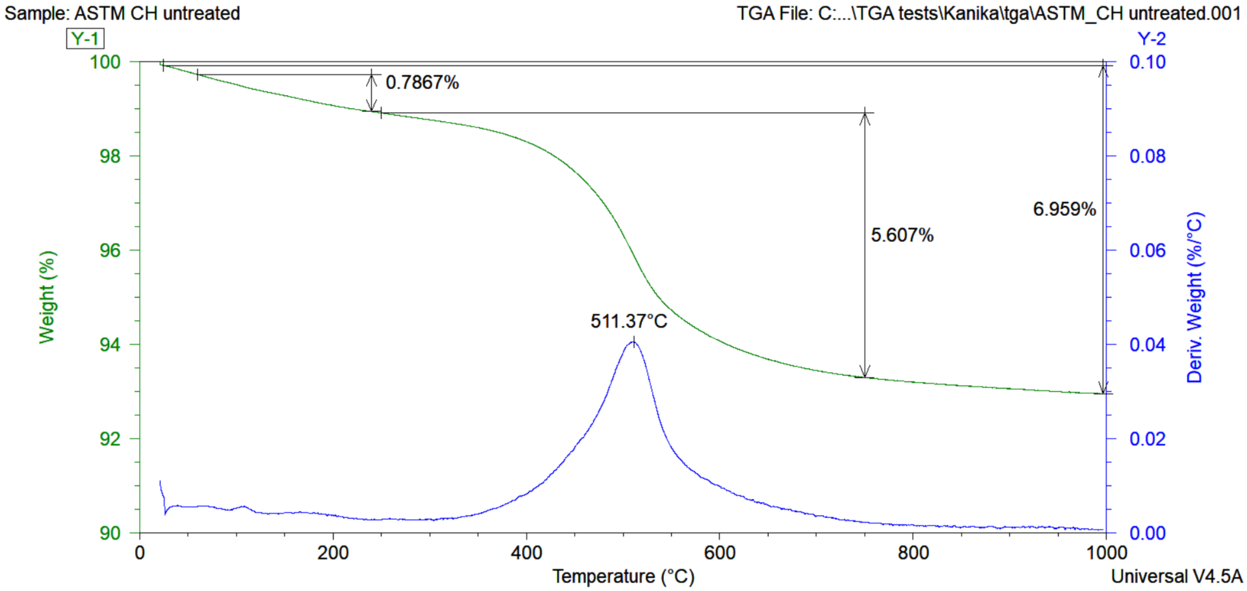

Figure A.17 TGA for ASTM CH soil (untreated).

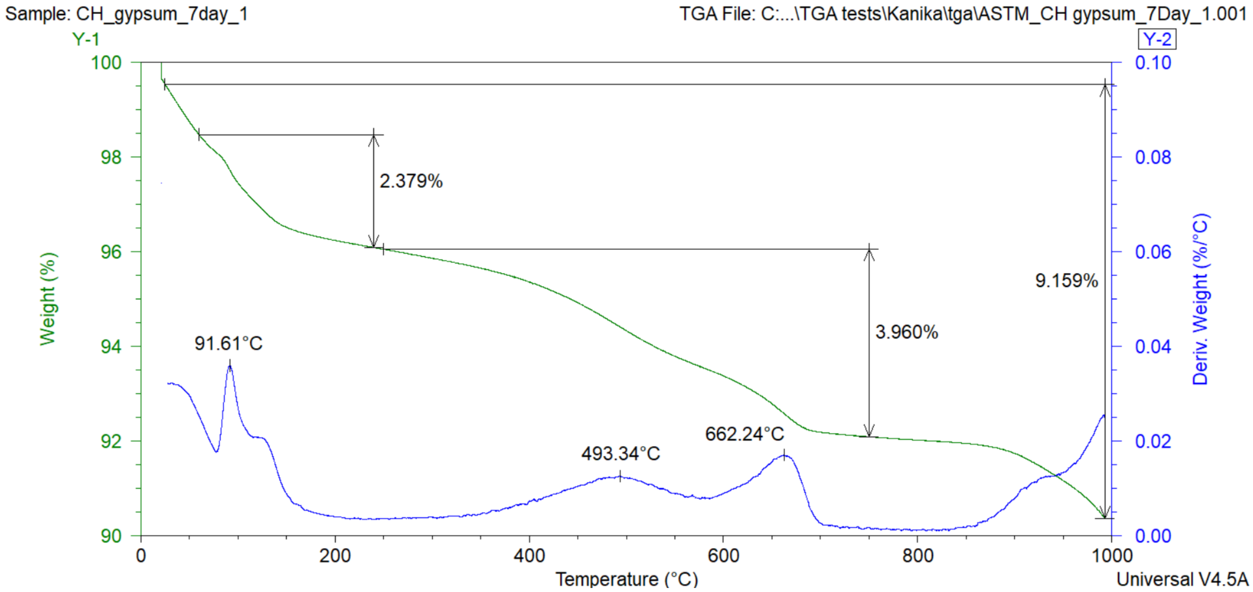

Figure A.18 TGA for ASTM CH soil treated with gypsum (curing 7 days) Test 1.

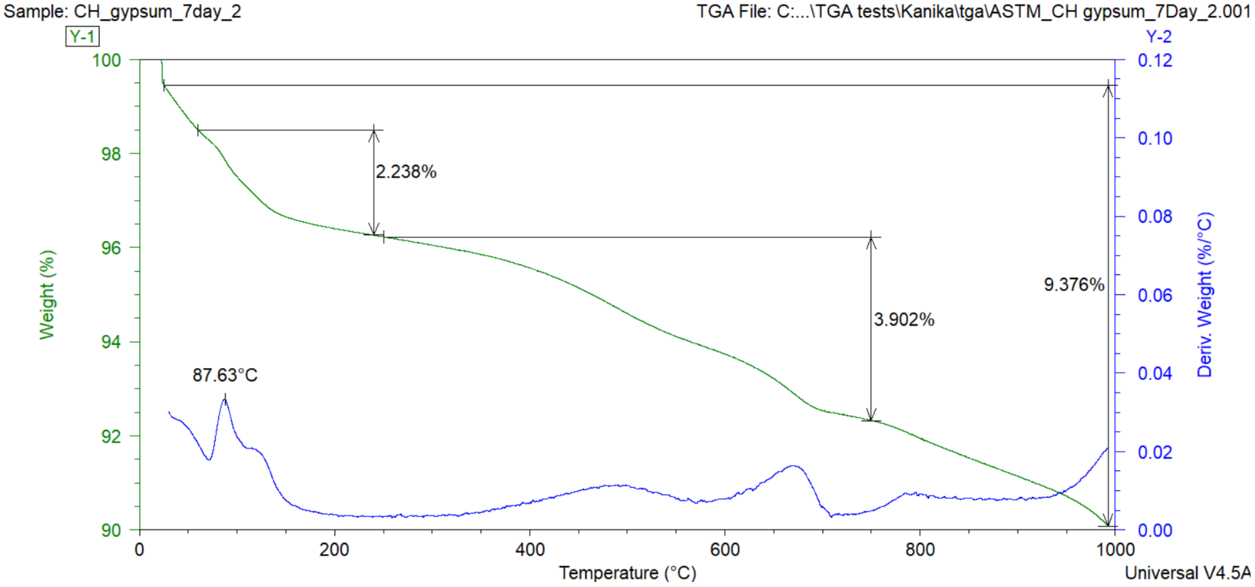

Figure A.19 TGA for ASTM CH soil treated with gypsum (curing 7 days) Test 2.

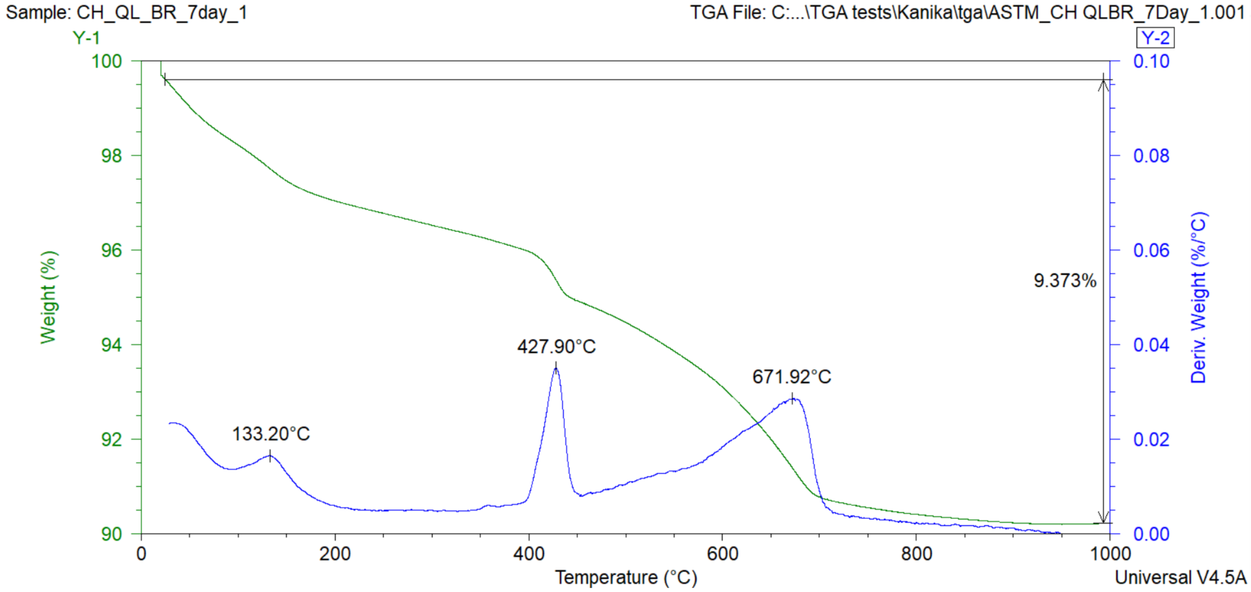

Figure A.20 TGA for ASTM CH soil treated with QL_BR (curing 7 days) Test 1.

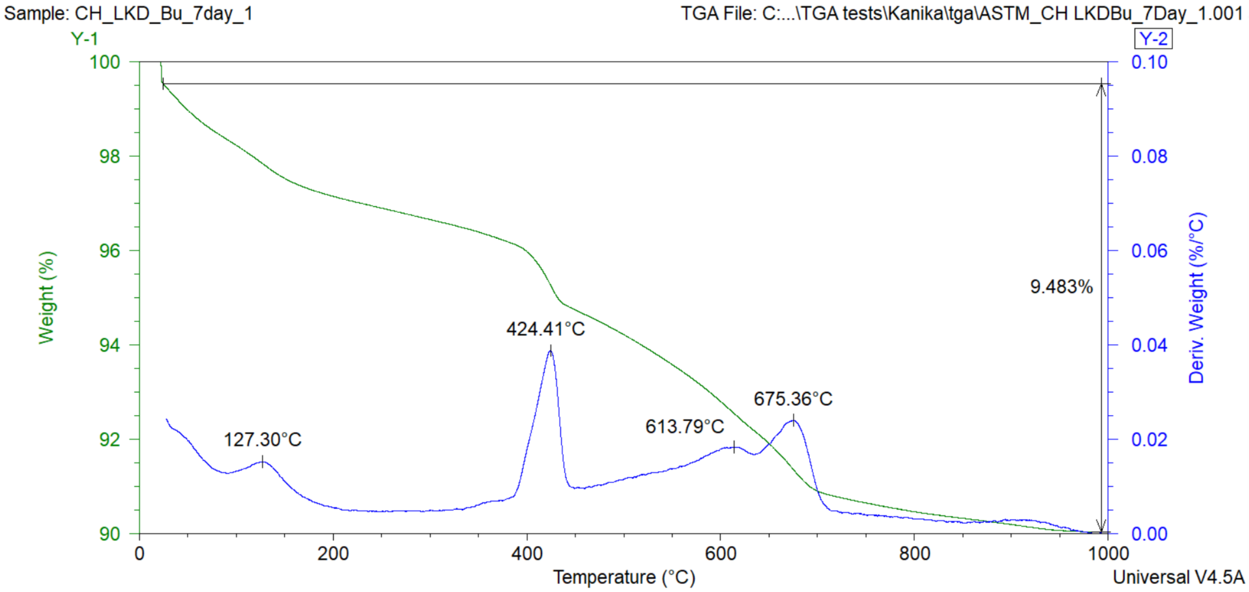

Figure A.21 TGA for ASTM CH soil treated with LKD_Bu (curing 7 days) Test 1.

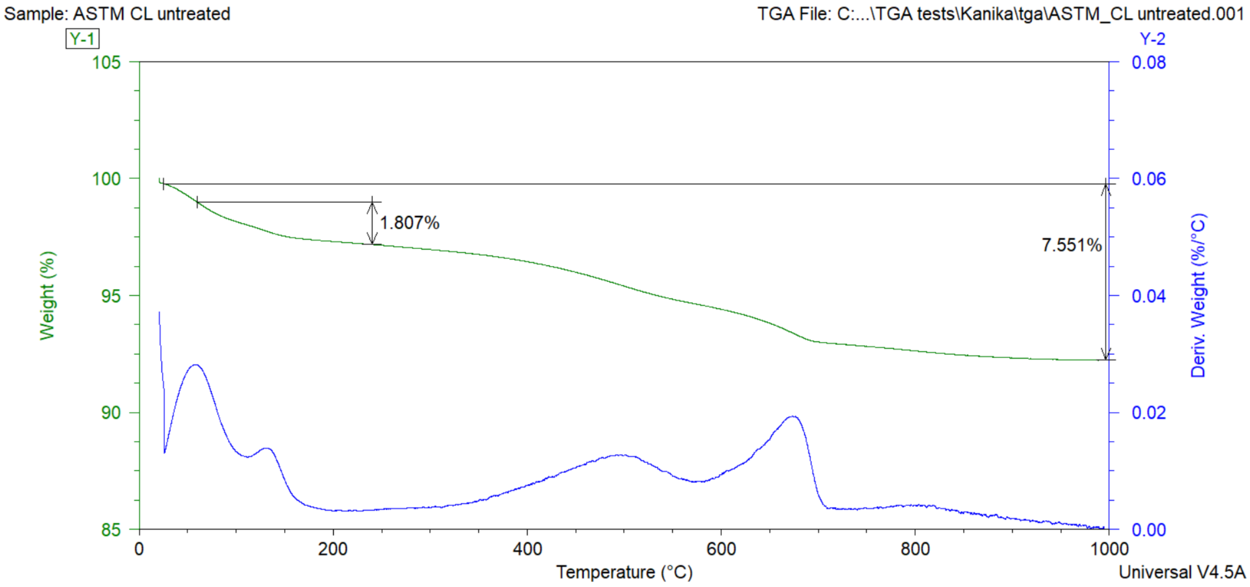

Figure A.22 TGA for ASTM CL soil (untreated).

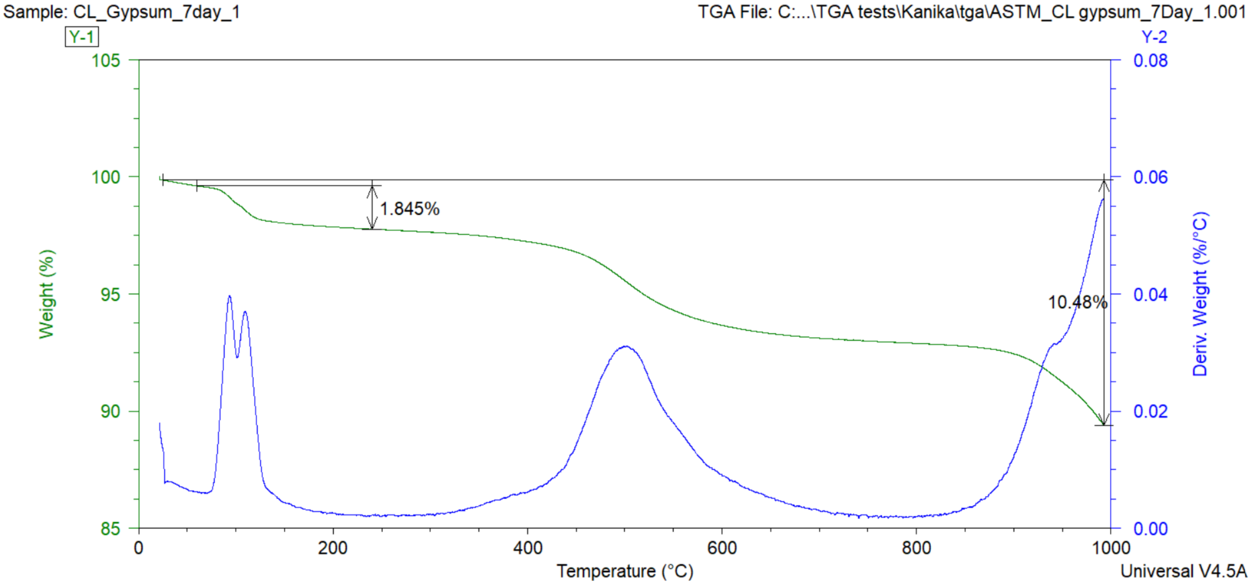

Figure A.23 TGA for ASTM CL soil treated with gypsum (curing 7 days) Test 1.

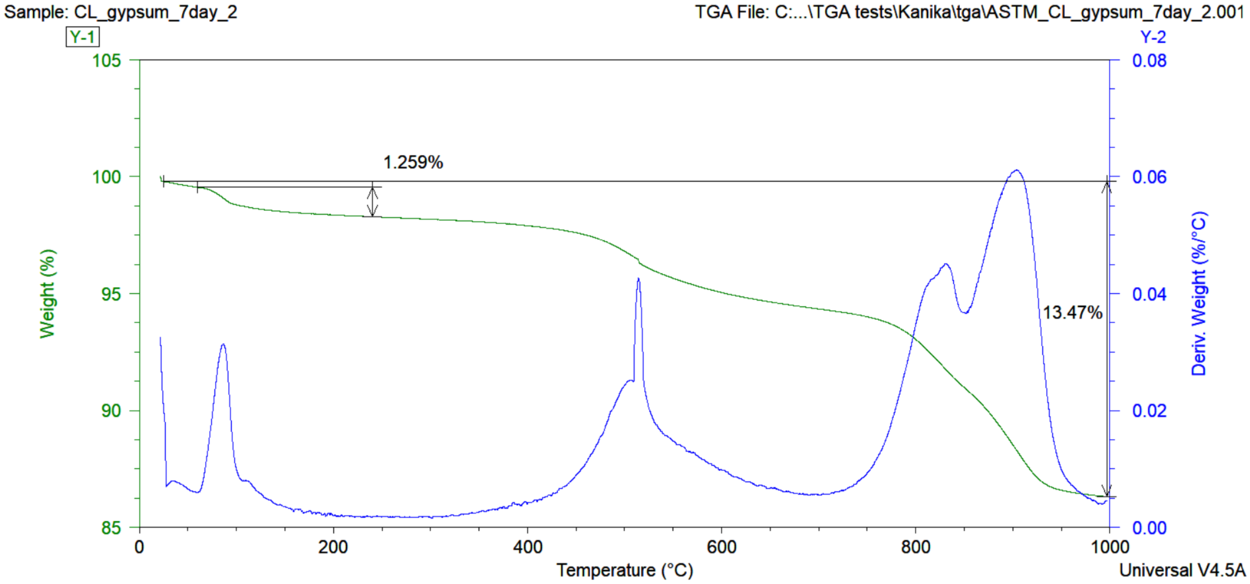

Figure A.24 TGA for ASTM CL soil treated with gypsum (curing 7 days) Test 2.

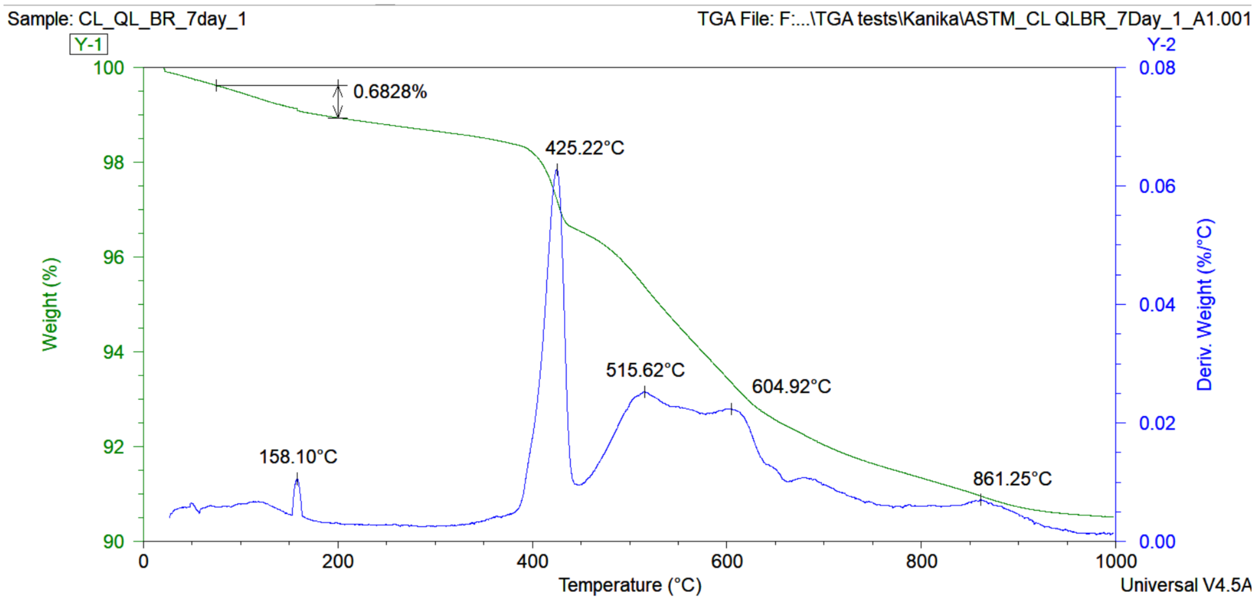

Figure A.25 TGA for ASTM CL soil treated with QL_BR (curing 7 days) Test 1.

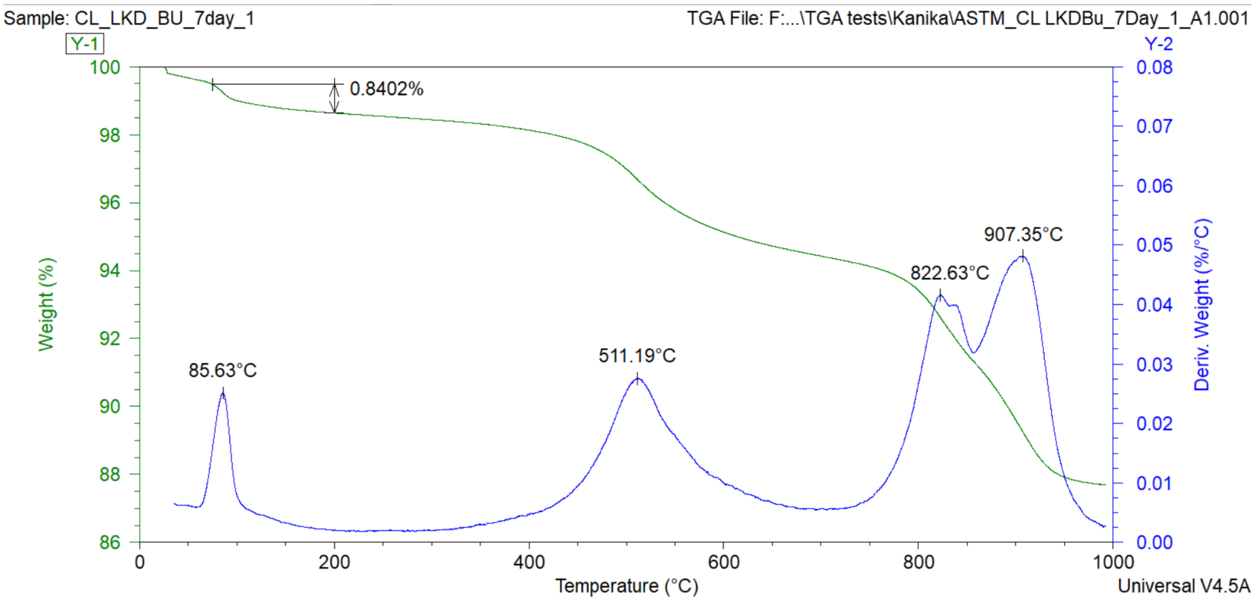

Figure A.26 TGA for ASTM CL soil treated with LKD_Bu (curing 7 days) Test 1.

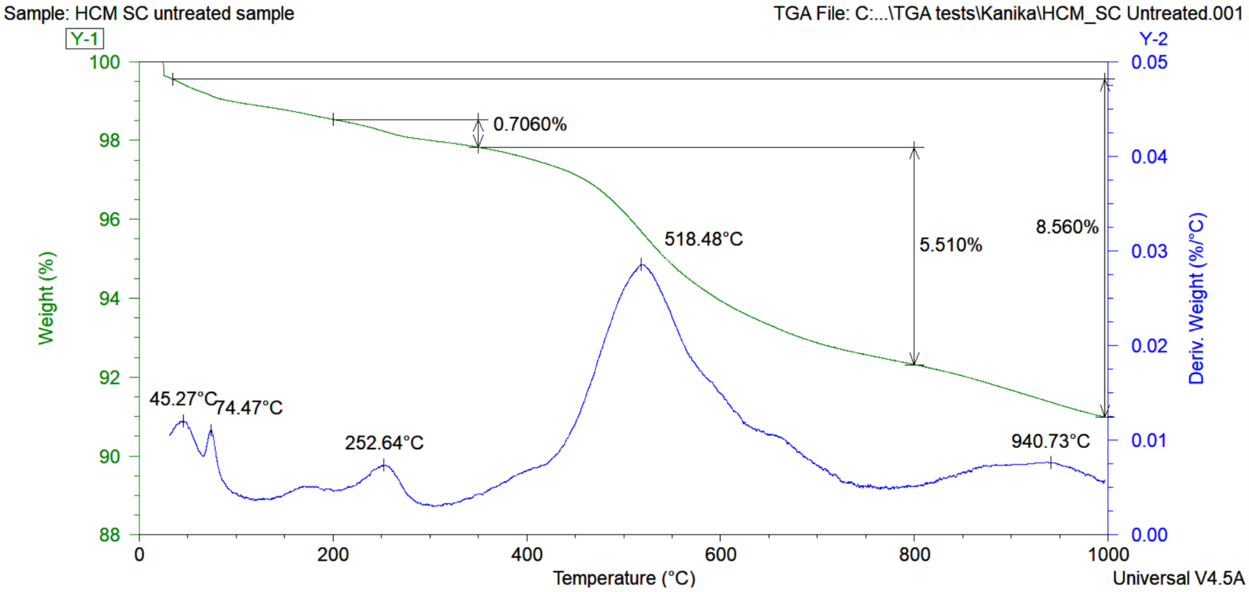

Figure A.27 TGA for HCM_SC (untreated).

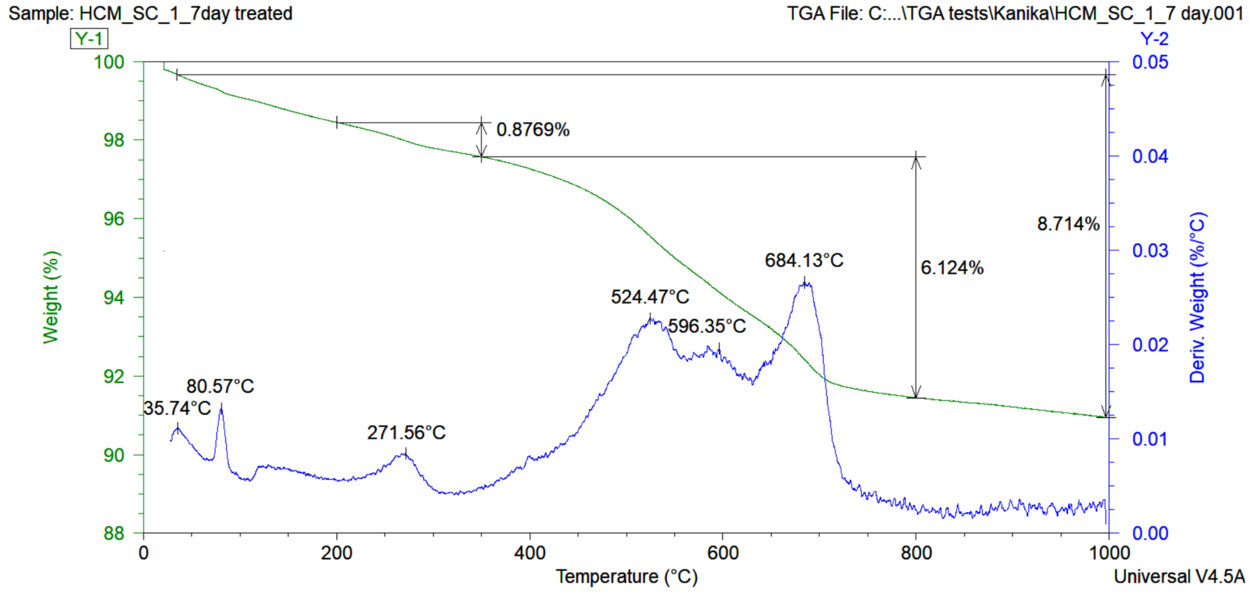

Figure A.28 TGA for HCM_SC treated with LKD_Bu (curing 7 days) Test 1.

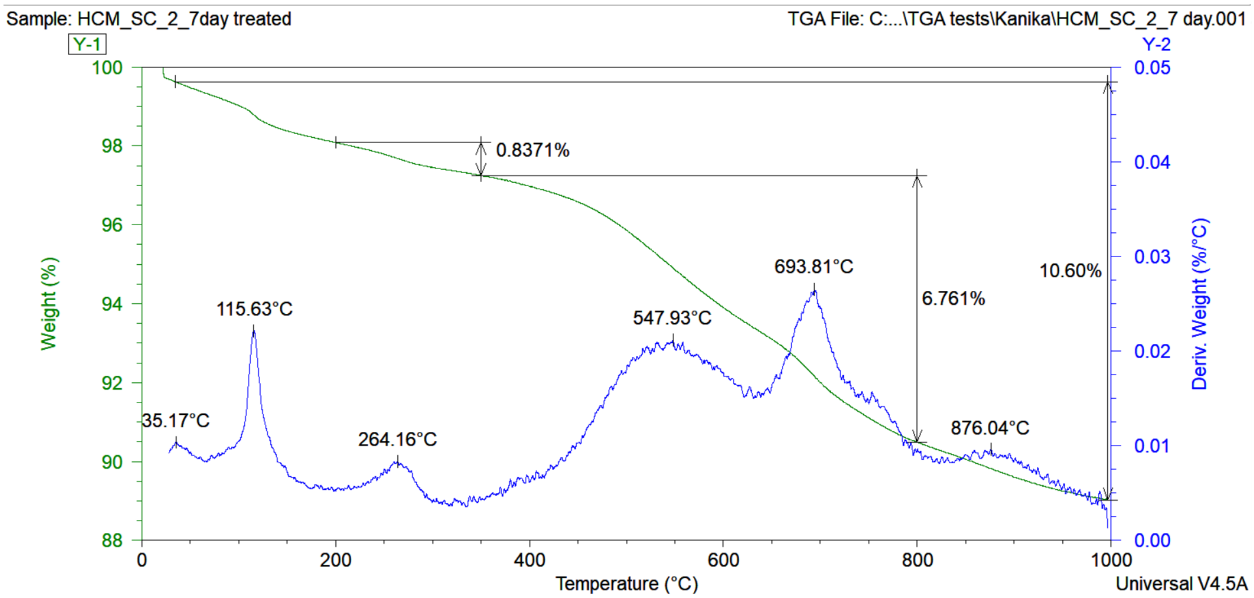

Figure A.29 TGA for HCM_SC treated with LKD_Bu (curing 7 days) Test 2.

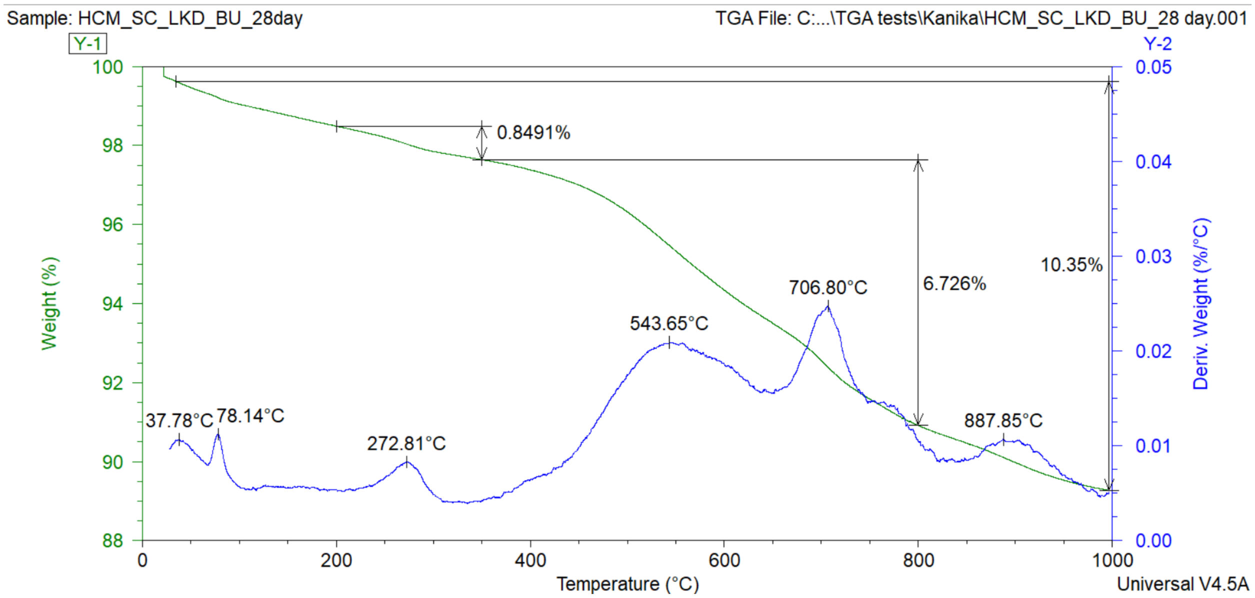

Figure A.30 TGA for HCM_SC treated with LKD_Bu (curing 28 days).

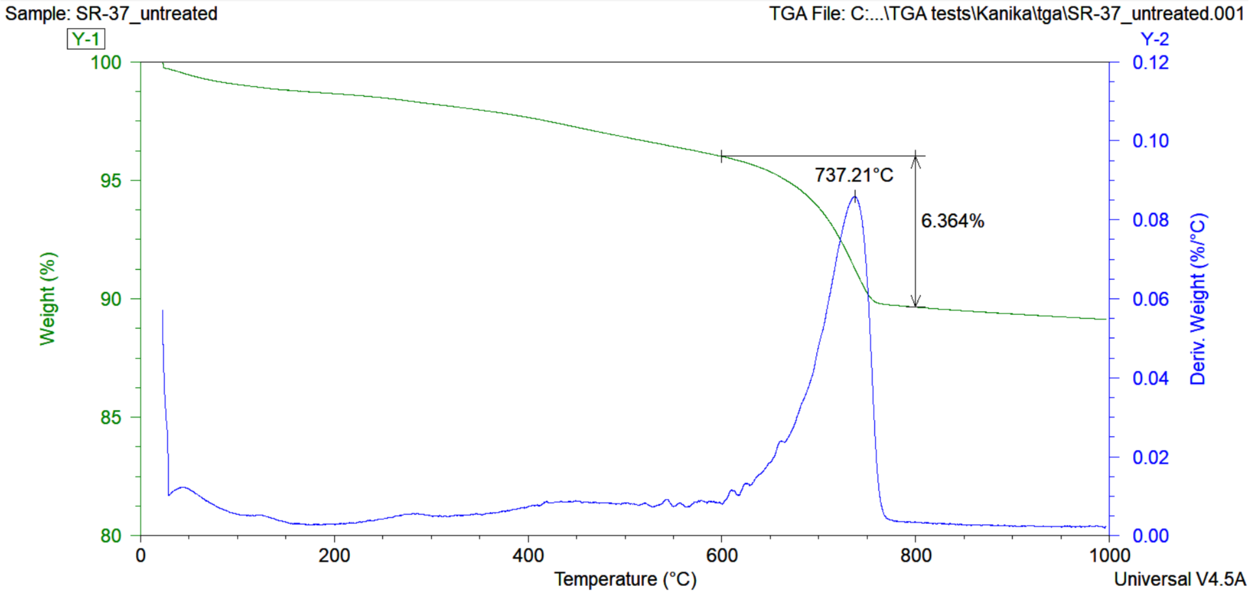

Figure A.31 TGA for SR-37 (untreated).

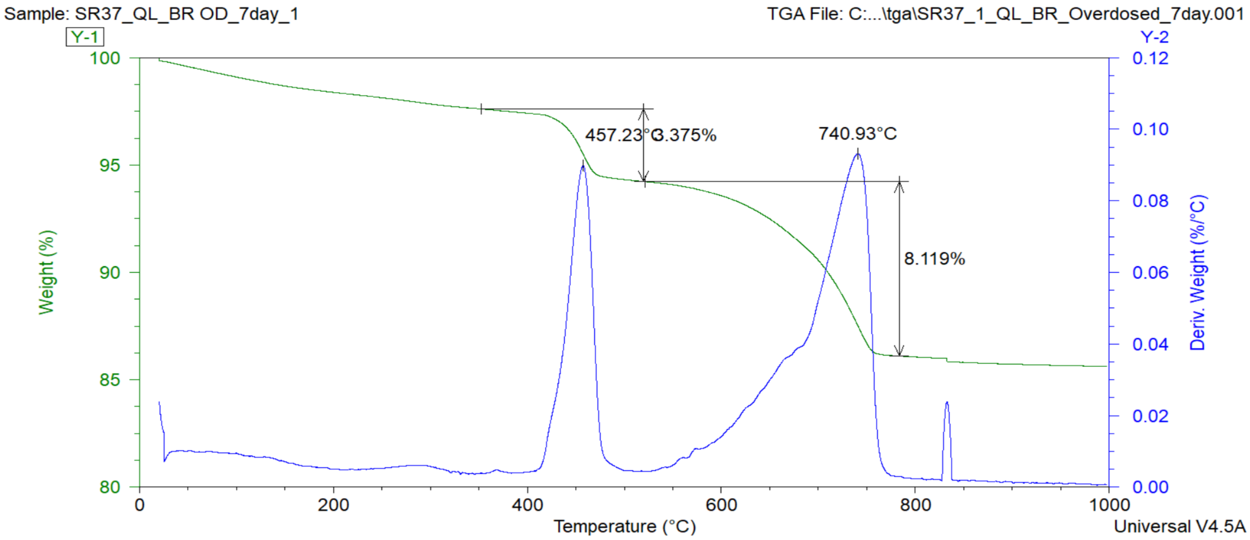

Figure A.32 TGA for SR-37 treated with QL_BR (curing 7 days) Test 1.

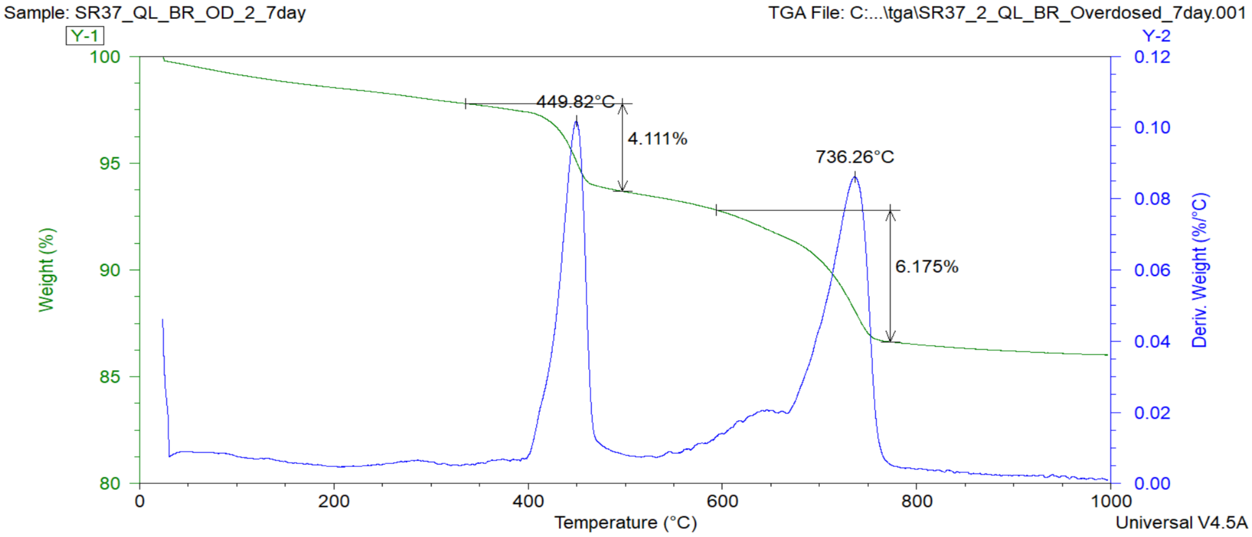

Figure A.33 TGA for SR-37 treated with QL_BR (curing 7 days) Test 2.

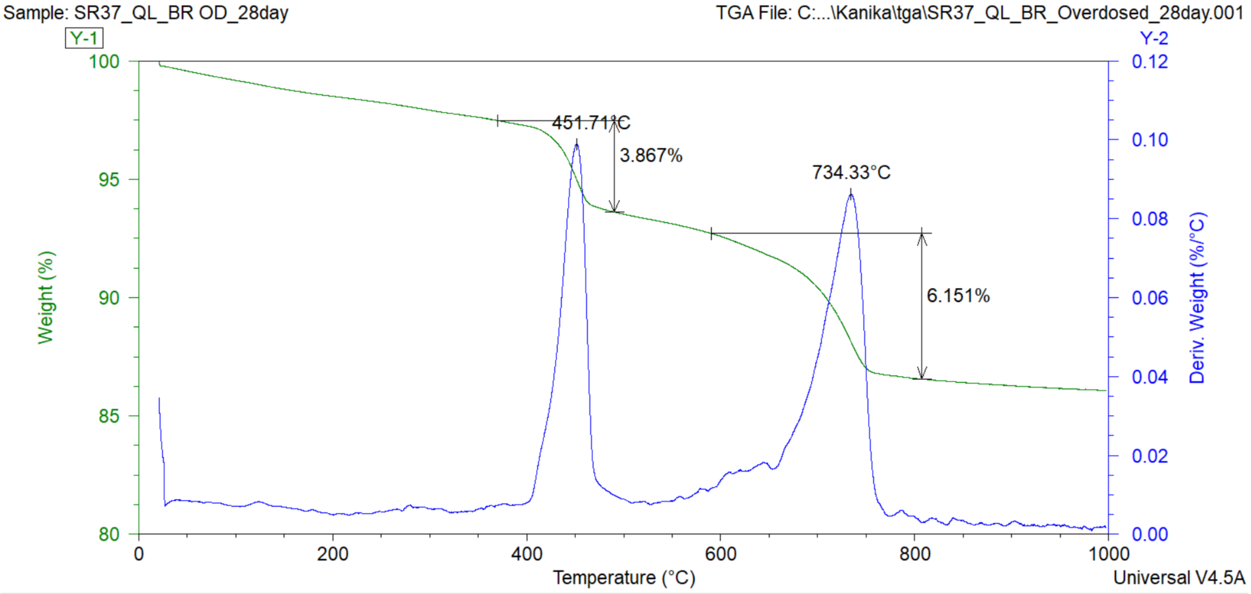

Figure A.34 TGA for SR-37 treated with QL_BR (curing 28 days).

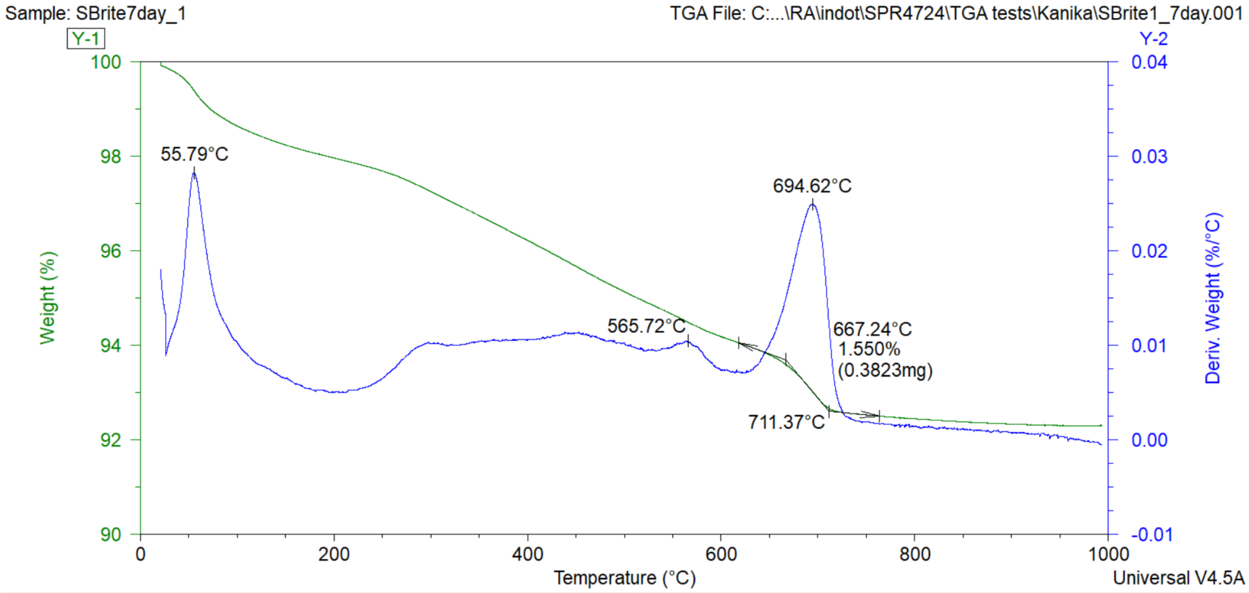

Figure A.35 TGA for SBRITE treated with LKD_Bu (curing 7 days) Test 1.

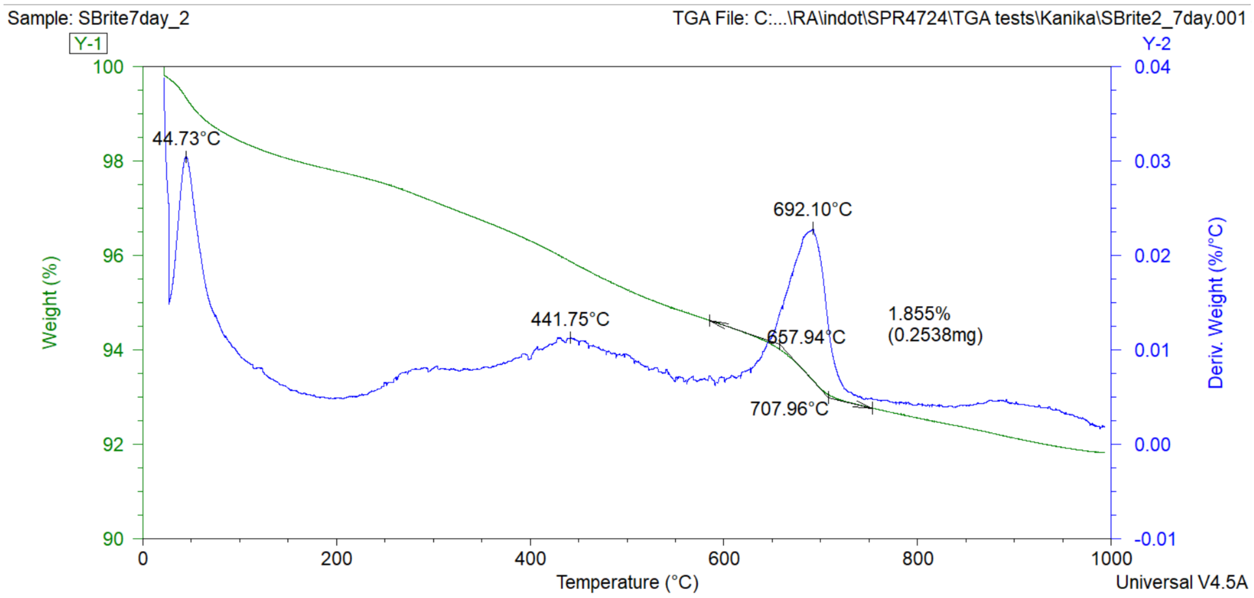

Figure A.36 TGA for SBRITE treated with LKD_Bu (curing 7 days) Test 2.

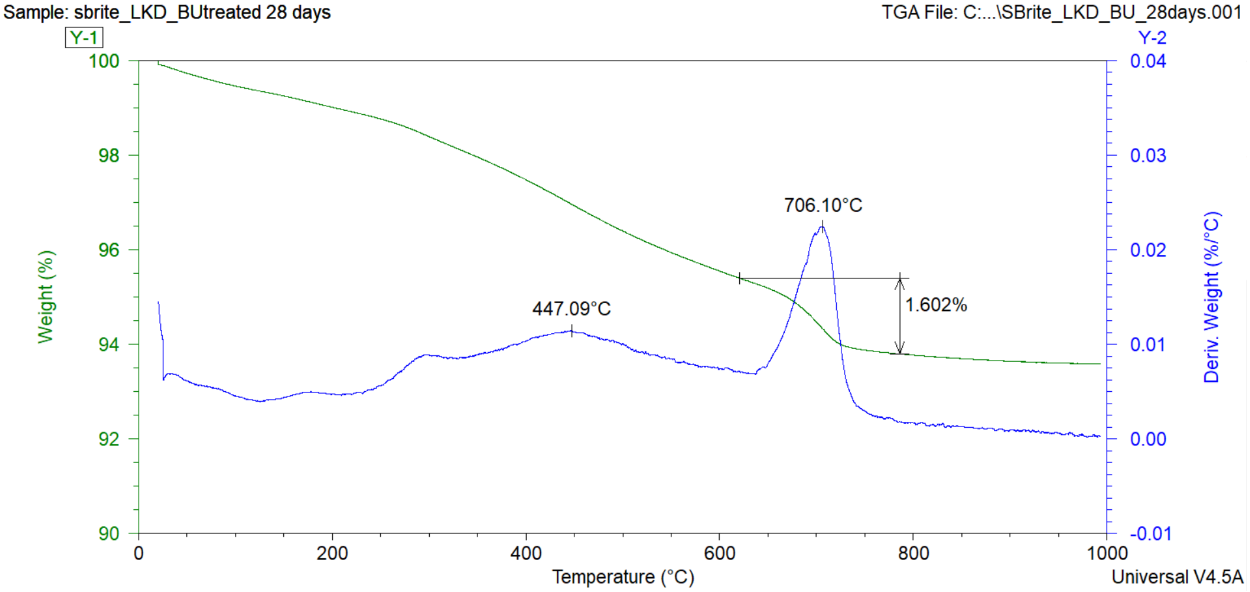

Figure A.37 TGA for SBRITE treated with LKD_Bu (curing 28 days).

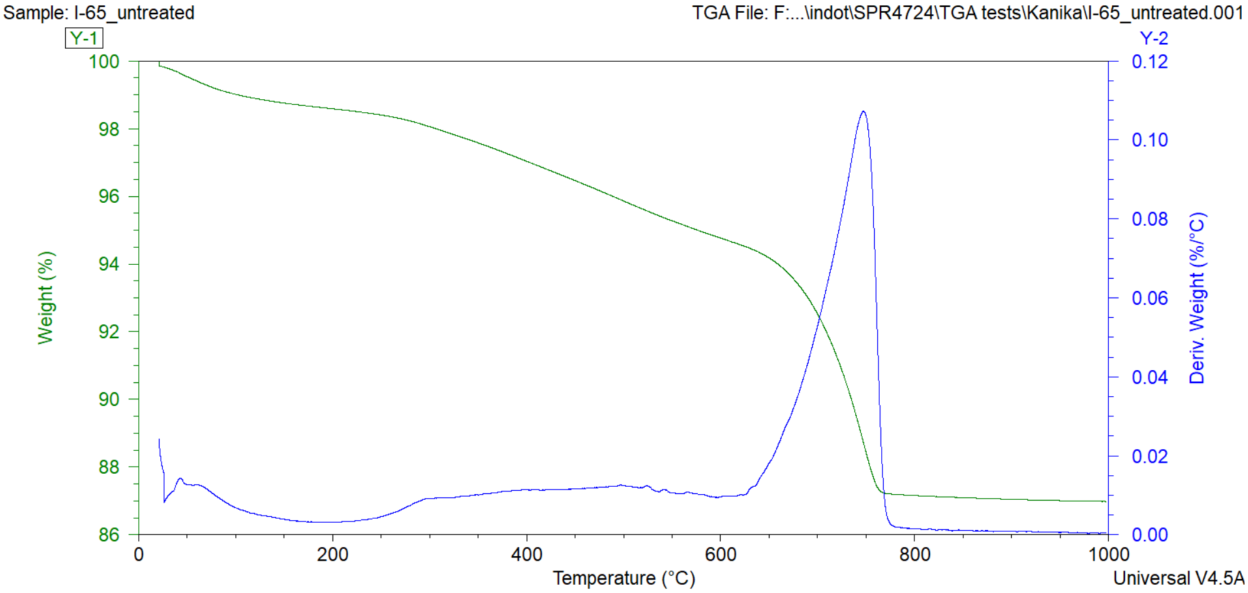

Figure A.38 TGA for I-65 (untreated).

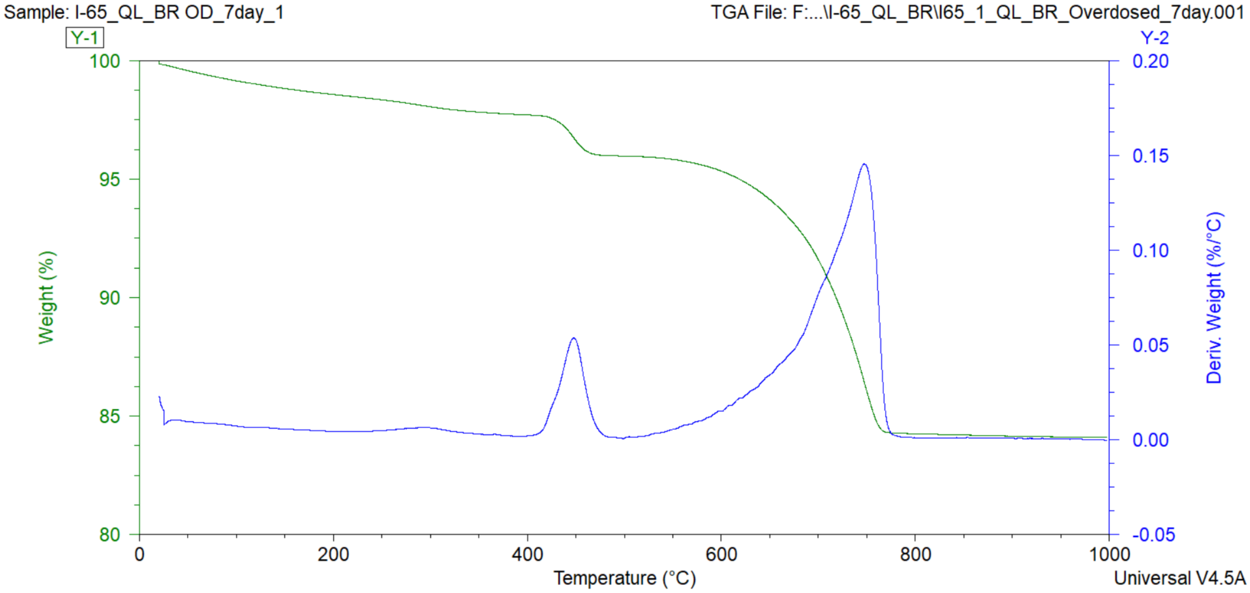

Figure A.39 TGA for I-65 treated with QL_BR (curing 7 days) Test 1.

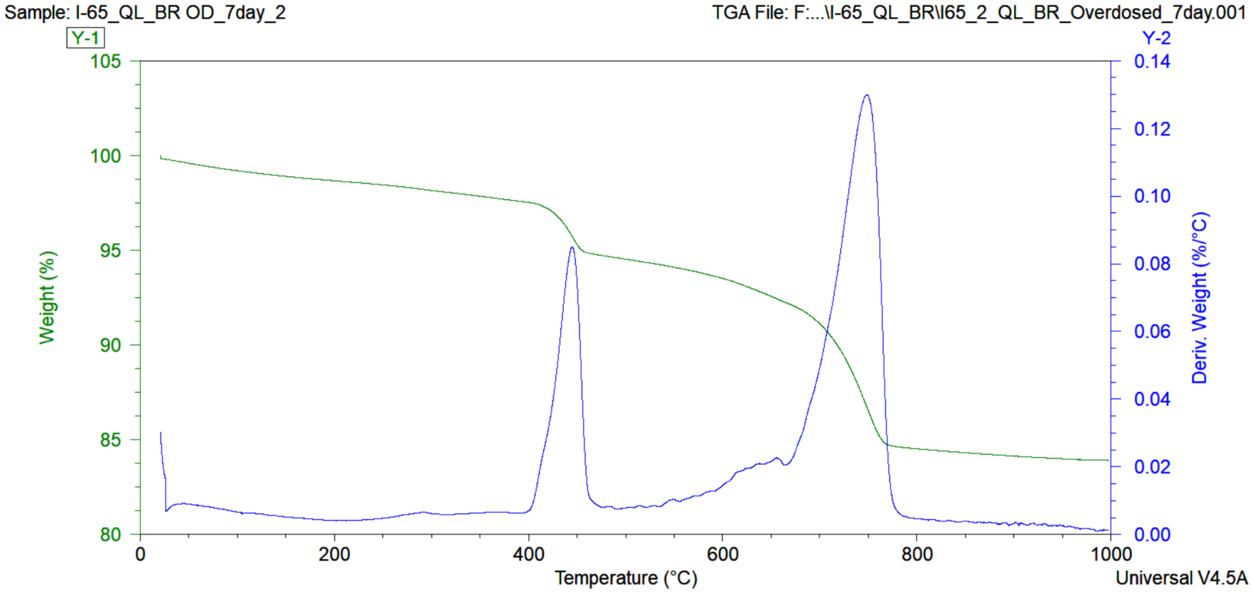

Figure A.40 TGA for I-65 treated with QL_BR (curing 7 days) Test 2.

APPENDIX B. ITM-510 AND IC TEST RESULTS

The following table presents results of ITM-510 and IC tests performed on soil, lime and soil-lime mixes. Both tests are performed on filtrate obtained from the respective soil-water slurries. The sample concentration is then measured upon multiplication with the respective dilution factor used in the preparation of the soil-water slurries.

Table B.1 Comparison of sulfate content measured from ITM-510 and ion chromatography tests

		Sulfate Concentration, ppm			
		Filtrate		**Sample**	
	Sample Designation	**IC**	**ITM-510**	**IC**	**ITM-510**
1	ASTM CL 1:20	159	159	3,173	3,173
2	ASTM CL 1:40	100	66	4,005	2,640
3	CL+LKD_Bu 7DS1 1:20	159	132	3,171	2,633
4	CL+LKD_Bu 7DS2 1:20	177	143	3,550	2,867
5	CL+LKD_Bu 28D 1:20	184	133	3,672	2,667
6	CL+LKD_Bu 7DS1 1:40	97	43	3,882	1,733
7	CL+LKD_Bu 7DS2 1:40	134	97	5,367	3,880
8	CL+LKD_Bu 28D 1:40	97	59	3,863	2,360
9	ASTM CL 1:20	192	157	3,837	3,147
10	ASTM CL 1:40	95	81	3,788	3,227
11	CL+QL_BR 7DS1 1:20	65	28	1,295	560
12	CL+QL_BR 7DS2 1:20	55	41	1,091	813
13	CL+QL_BR 28D 1:20	47	41	942	813
14	CL+QL_BR 7DS1 1:40	44	44	1,749	1,773
15	CL+QL_BR 7DS2 1:40	31	25	1,244	1,013
16	CL+QL_BR 28D 1:40	37	34	1,496	1,360
17	CL+LKD_CK2 7DS1 1:20	128	104	2,569	2,080
18	CL+LKD_CK2 7DS2 1:20	101	66	2,029	1,320
19	CL+LKD_CK2 28D 1:20	110	81	2,195	1,620
20	CL+LKD_CK2 7DS1 1:40	90	47	3,616	1,867
21	CL+LKD_CK2 7DS2 1:40	66	69	2,627	2,773
22	CL+LKD_CK2 28D 1:40	80	81	3,195	3,227
23	ASTM CH 1:20	4	0	72	0
24	CH+LKD_Bu 7DS1 1:20	90	89	1,803	1,773
25	CH+LKD_Bu 7DS2 1:20	97	105	1,934	2,093
26	CH+LKD_Bu 28D 1:20	99	103	1,976	2,067
27	ASTM CH 1:20	2	0	48	0
28	CH+LKD_Bu 7DS1 1:40	45	45	1,798	1,813
29	CH+LKD_Bu 7DS2 1:40	44	42	1,752	1,680
30	CH+LKD_Bu 28D 1:40	43	41	1,724	1,653
31	CH+QL_BR 7DS1 1:20	6	0	117	0
32	CH+QL_BR 7DS2 1:20	15	6	290	127
33	CH+QL_BR 28D 1:20	6	0	118	0

34	SBRITE 1:20	1	0	27	0
35	SBRITE+LKD_CK2 7DS1 1:20	62	38	1,244	760
36	SBRITE+LKD_CK2 7DS2 1:20	67	40	1,336	800
37	SBRITE+LKD_CK2 28D 1:20	75	40	1,495	800
38	SBRITE+LKD_CK2 7DS1 1:40	29	22	1,180	893
39	SBRITE+LKD_CK2 7DS2 1:40	33	26	1,326	1,053
40	SBRITE+LKD_CK2 28D 1:40	65	76	2,611	3,040
41	SBRITE - HC85_BV 7DS1 1:20	5	0	90	0
42	SBRITE - HC85_BV 7DS2 1:20	4	0	81	0
43	SBRITE - HC85_BV 28D 1:20	5	0	105	0
44	I-65 1:20	14	0	270	0
45	I-65+LKD_CK2 7DS1 1:20	88	53	1,766	1,067
46	I-65+LKD_CK2 7DS2 1:20	85	53	1,696	1,060
47	I-65+LKD_CK2 28D 1:20	95	75	1,908	1,500
48	I-65+LKD_CK2 7DS1 1:40	43	41	1,701	1,627
49	I-65+LKD_CK2 7DS2 1:40	55	60	2,196	2,413
50	I-65+LKD_CK2 28D 1:40	52	54	2,060	2,173
51	I-65 - HC85_BV 7DS1 1:20	14	0	285	0
52	I-65 - HC85_BV 7DS2 1:20	14	0	289	0
53	I-65 - HC85_BV 28D 1:20	15	0	309	0
54	HCM_SC 1:20	437	267	8,737	5,333
55	HCM_SC 1:40 S1	316	320	12,621	12,800
56	HCM_SC 1:40 S2	421	434	16,834	17,360
57	HCM_SC+LKD_CK2 7DS1 1:20	605	528	12,108	10,560
58	HCM_SC+LKD_CK2 7DS2 1:20	744	624	14,873	12,480
59	HCM_SC+LKD_CK2 28D 1:20	620	501	12,406	10,027
60	HCM_SC+LKD_CK2 7DS1 1:40	393	396	15,733	15,840
61	HCM_SC+LKD_CK2 7DS2 1:40	382	288	15,270	11,520
62	HCM_SC+LKD_CK2 28D 1:40	315	264	12,594	10,560
63	HCM_SC+LKD_Bu 7DS1 1:20	751	731	15,017	14,613
64	HCM_SC+LKD_Bu 7DS2 1:20	678	629	13,557	12,587
65	HCM_SC+LKD_Bu 28D 1:20	685	683	13,690	13,653
66	HCM_SC+LKD_Bu 7DS1 1:40	371	450	14,825	18,000
67	HCM_SC+LKD_Bu 7DS2 1:40	420	402	16,781	16,080
68	HCM_SC+LKD_Bu 28D 1:40	367	339	14,683	13,560

About the Joint Transportation Research Program (JTRP)

On March 11, 1937, the Indiana Legislature passed an act which authorized the Indiana State Highway Commission to cooperate with and assist Purdue University in developing the best methods of improving and maintaining the highways of the state and the respective counties thereof. That collaborative effort was called the Joint Highway Research Project (JHRP). In 1997 the collaborative venture was renamed as the Joint Transportation Research Program (JTRP) to reflect the state and national efforts to integrate the management and operation of various transportation modes.

The first studies of JHRP were concerned with Test Road No. 1—evaluation of the weathering characteristics of stabilized materials. After World War II, the JHRP program grew substantially and was regularly producing technical reports. Over 1,600 technical reports are now available, published as part of the JHRP and subsequently JTRP collaborative venture between Purdue University and what is now the Indiana Department of Transportation.

Free online access to all reports is provided through a unique collaboration between JTRP and Purdue Libraries. These are available at http://docs.lib.purdue.edu/jtrp.

Further information about JTRP and its current research program is available at http://www.purdue.edu/jtrp.

About This Report

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