EVALUATION OF J-ROX AS A SUPPLEMENTARY CEMENTITIOUS MATERIAL FOR CONCRETE APPLICATIONS

by

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ABSTRACT

ALLISON ANNE SUMMERS. Evaluation of J-Rox as a Supplementary Cementitious Material for Concrete Applications. (Under the direction of DR. TARA L. CAVALLINE)

Supplementary cementitious materials (SCMs) provide a variety of benefits to fresh and hardened concrete. These materials can be found in naturally occurring substances or from by-products or co-products of a variety of industries. However, some of the industries that have been a large source of SCMs over past decades are not as prevalent today and therefore are causing a decrease in availability of popular SCMs, particularly fly ash. The main reason for the decreases in production have been concern over the environmental impacts that the mechanical process to derive fly ash, coal burning, is causing. Research has now turned to the identification of new SCMs that provide the same performance benefits as materials such as fly ash but without the environmental impacts. Evaluation of an alternative SCM produced as a byproduct of the phosphorous industry for use in concrete is the goal of this project.

The material being tested in the following research project is a co-product of producing phosphoric acid called J-Rox, produced by a phosphoric acid producer in Florida. The material was tested at 15% and 25% rates in paste, mortar, and concrete samples to determine the benefits it could provide as a SCM compared to the benefits of samples that did not use an SCM and samples that had a 20% replacement of fly ash. Through the process of testing the paste and mortar samples, different variations were created based on results of previous versions. This was done to determine which versions would potentially perform the most similar to fly ash when used in concrete. The versions determined best for concrete use were J-Rox 3 and 4.

The main concerns for similar performance among J-Rox and other SCMs like fly ash was the differences in chemical composition and fineness. J-Rox was found to have a higher

P_2O_5 percent weight which could potentially affect its performance. However, despite these differences J-Rox performed very similarly to the control samples. Compressive strength test results for J-Rox mortar and concrete were lower than control samples at the 28 day test period, but the J-Rox mixtures continued to show late age strength gain, reaching values similar to those of the controls at later ages. J-Rox concrete mixtures showed resistivity gain in late age testing similar to that which is seen in fly ash mixture, and therefore may provide durability benefits. No notable issues with concrete performance due to the higher P_2O_5 content were observed for the mixtures and tests performed as part of this study.

For all mixtures prepared as part of this work, shrinkage values were moderate to relatively high among all samples. However, this could be adjusted through the use of a lower w/c ratio, should be used in mixtures produced during future testing. Since this material is targeted for use in Florida, the addition of the chloride diffusion test to the experimental program of future work may also be beneficial.

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LIST OF ABBREVIATIONS

AASHTO	American Association of State Highway and Transportation Officials
AC	air cooled
ACI	American Concrete Institute
ASTM	American Society for Testing and Materials
°C	degrees Celsius
cf	cubic feet
cwt	hundred weight of cement
cy	cubic yard
°F	degrees Fahrenheit
FDOT	Florida Department of Transportation
FHWA	Federal Highway Administration
ft	foot
g	gram
gal	gallon
hr	hour
in	inch
JR	J-Rox
JR1F	J-Rox 1 Furnace
JR1K	J-Rox 1 Kiln
JR2	J-Rox 2
JR3	J-Rox 3
JR4AC	J-Rox 4 Air Cooled

JR4WQ	J-Rox 4 Water Quenched
kg	kilogram
lb	pound
m	meter
μ	micro
mL	milliliter
mm	millimeter
MOE	modulus of elasticity
OPC	ordinary portland cement
oz	ounce
pcy	pounds per cubic yard
pcf	pounds per cubic foot
psi	pounds per square inch
RH	relative humidity
RHF	rotary hearth furnace
σ	stress
ε	strain
SCM	supplementary cementitious material
V	volts
UNC	University of North Carolina
yd	yard
WQ	water quenched

CHAPTER 1 : INTRODUCTION

1.1 Problem Statement

Pozzolanic materials have been used in concrete in order to provide benefits in both fresh and hardened concrete, such as workability improvements and increased durability. According to the American Concrete Institute, pozzolans are siliceous or silico-aluminous materials that are classified as cementitious materials due to their ability to form compounds similar to cementitious materials during the reactions that occur when mixing concrete (ACI, 2017). Pozzolanic materials provide benefits to concrete that include improved strength and chemical resistance, the ability to reduce the rate of heat evolution during the hydration reactions and slow the strength development but greater overall strength after curing (Sutter, 2020). Although some pozzolanic materials naturally exist, some byproducts of a variety of industrial processes are also pozzolans.

The procurement of most pozzolans usually occurs through mechanical processes that produce the pozzolan materials as a byproduct. An example of this process that is widely used in concrete production is fly ash, a byproduct of coal burning. However, as coal burning becomes less commonly used as a source of energy in the United States, the ability to source this material becomes more difficult (Sutter, 2020). As the United States switches its preferred raw material for energy production from coal to natural gas, coal-fired power plants are being decommissioned. Thus, the production of fly ash will continue to gradually decrease over the next 20 years, leaving the majority of reserves being sourced from landfills and ponds where the material was previously stored before a use for it was determined (Sutter, 2020). This poses a severe problem for concrete production due to the need of fly ash in concrete to increase workability and sulfate resistance, reduce the cost, decrease shrinkage, and increase late strength (Sutter, 2020). In fact, many states require SCMs such as fly ash in certain types of infrastructure or in elements exposed to certain conditions. Therefore, concrete producers and users need to identify other materials that can perform in a manner similar to fly ash in concrete in order to maintain those benefits. As Sutter (2020) states, “concerns center on the fact that no other material is available with the reserves that fly ash historically has provided”. While there are already other supplementary cementitious materials that are used in a similar manner as fly ash, such as slag cement or other pozzolans, they are less available in some areas of the United States than fly ash, more costly, or do not provide the exact benefits that fly ash can support (Sutter, 2020).

Therefore, new alternatives must be investigated since the use of these supplementary cementitious materials (SCMs) are “essential to concrete durability”. One of these alternatives consists of a byproduct of making phosphoric acid, called J-Rox. J-Rox has been identified as a material that could possibly be proved to improve fresh and hardened concrete in a similar way as fly ash. In order to determine if this new material can potentially be used in practices that other pozzolanic materials such as fly ash have been, the chemical and physical properties of both the J-Rox material and the cementitious products produced using J-Rox (such as paste, mortar, and concrete) must be analyzed and compared. This is done through chemical analyses of the J-Rox material, as well as a variety of tests outlined in the American Society of Testing and Materials (ASTM) and American Association of State Highway and Transportation Officials (AASHTO) standards for properties of paste, mortar, and fresh and hardened concrete.

1.2 Objectives and Scope of Study

The specific objectives of the study are:

- To determine if J-Rox will contribute pozzolanic reactions to increase hydration in concrete which benefit strength and durability,
- To test and compare the chemical and physical properties to determine what changes in fresh and hardened concrete J-Rox causes, and
- To determine if there are adverse effects of using the product in fresh and hardened concrete.

1.3 Organization of Thesis

The organization of this thesis will be separated into six chapters beginning with a literature review (Chapter 2) to provide greater background and information on the need for this project to be completed, followed by the methodology (Chapter 3) used to gather and interpret the information. The results of the necessary tests needed to successfully study this new material will then be presented (Chapter 3) followed by an in-depth analysis of the results (Chapter 4). An analysis of the impact of phosphorous content on the results is presented (Chapter 5), followed finally by the conclusions and recommendations for future projects (Chapter 6).

CHAPTER 2 : LITERATURE REVIEW

2.1: Environmental Impacts

The production of cement has increased greatly over the past century due to the increased demands from the infrastructure industry around the world. It is projected that by 2050 the amount of cement production worldwide will reach 5.8 billion tons per year (Juenger & Siddique, 2015). This amount of production is not only concerning for environmental reasons due to the negative effects of production (such as consumption of natural resources and emission of greenhouse gasses), but the ability for supply to meet society's demand is an issue as well. Both of these concerns can be addressed through the use of SCMs as a partial replacement to cement.

2.1.1: Benefit of Reducing Cement Content

The annual world cement production has experienced a growth of 0.7 billion tons in recent years (Yang et. al, 2015). While this has been beneficial for the construction industry as there is a greater supply of the material, it has caused environmental impacts which are not insignificant. Based on the growth of the cement production industry it is estimated that the industry produces 7% of the worldwide production of CO_2 (Yang et. al, 2015). Approximately 60% of the CO_2 produced is generated during the decarbonation of limestone during the creation of cement clinker, with the remaining 40% coming from the fuel used to power the production processes (Skibsted, 2019). There are also other environmental impacts from dust pollution and degradation of land during the mining process for source materials.

Of the options provided for reducing the amount of cement produced, the addition of SCMs is the most economical and practical, as well as having a straightforward process for application (Yang et.al, 2015). The use of SCMs not only reduces the amount of CO_2 emissions significantly, but it also provides a use for by-products that are already being produced from a variety of manufacturing processes (Lothenbach et al., 2011). The replacement of cement with SCMs provides one important means of reducing the amount of CO_2 emissions between 30 and 40% without affecting the performance characteristics of the concrete produced (Skibsted & Snellings, 2019).

2.2: Supplementary Cementitious Materials (SCMs)

Supplementary Cementitious Materials (SCMs) have been used in concrete applications to provide various performance benefits. As of 2015, over 60% of ready-mix concrete made in the United States utilized some form of SCMs (Juenger & Siddique, 2015). In order to achieve these benefits, SCMs are used in conjunction with portland cement in concrete mixtures to initiate either hydraulic or pozzolanic reactions (PCA, 2011). SCMs require exposure to moisture for a longer period of time, thus allowing the chemical reactions that provide beneficial characteristics of hardened concrete to continue longer than they normally would in cement-only mixtures (Mehta & Monterio, 2014). The main focus of this section will be on fly ash, since that is the material being used for comparison to the test material in this study; however, for informational purposes a variety of SCMs will also be occasionally mentioned or discussed.

2.2.1: Types of SCMs

Types of SCMs, also referred to as mineral admixtures, can be separated into two main categories, natural pozzolanic materials and by-product materials (Mehta & Monterio, 2014). Natural pozzolanic materials are usually sourced from volcanic rocks, volcanic glasses, or minerals (Mehta & Monterio, 2014). By-product materials include

SCMs such as fly ash, iron-blast furnace slag, and silica fume. These byproducts are produced along with the primary products of certain industrial processes, and either must be disposed of after produced or recycled into a new material, such as concrete.

The most commonly used SCM in concrete is fly ash, which can replace cement content by 15% to 35% by weight (Al-Shmaisani et. al, 2019). The industrial process that creates this by-product is the combustion of coal in coal-fired power plants. Fly ash is separated into two categories based that are primarily dependent on the type of coal that was burned to produce the ash, which overall controls the amount of CaO (Mehta & Monterio, 2014). These categories are denoted by Class F fly ash, or low-calcium fly ash with larger proportions of silica and aluminum, and Class C fly ash, or high-calcium fly ash (Mehta & Monterio, 2014). Positive impacts of the use of fly ash in both fresh and hardened properties of concrete include but are not limited to increasing workability, reducing permeability, and increasing later strength gain; as well as it being less costly than portland cement (Al-Shmaisani et. al, 2019). Class F type fly ashes have also shown the ability to mitigate alkali-silica reactivity, a material-related distress mechanism that must be addressed in a number of states across the United States due to the presence of reactive aggregates.

Often, fly ash also reduces the amount of water by 1 to 10% required to reach the needed workability of the concrete mixture compared to those that only use portland cement (PCA, 2011). This is due to the spherical shape of fly ash particles, which increases the workability of the concrete mixture. Also, the smaller particle size of fly ash creates a larger net surface area, and therefore a greater number of nucleation sites, for water to be absorbed and reactions to occur (PCA, 2011). This results in an improvement in concrete's microstructure, with the nucleation sites promoting dense formation of hydration products. In other words, the smaller particle sizes and round shape provide performance benefits.

Iron blast-furnace slag is a by-product of producing pig iron, with its initial state being in a liquid form. The liquid slag is then either water-quenched or air-quenched (with a small amount of water) to form granulated slag or pelletized slag respectively (Mehta & Monterio, 2014). The various types of SCMs provide their own benefits which determine the application in which they are used, but their accessibility varies by region. For example, iron production is more prominent in the northeastern United States, resulting in the prevalence of slag cements used in that area.

2.2.2: Chemical Compositions of SCMs

Multiple chemical reactions occur during the concrete production process that change when an SCM is introduced into the mixture, beginning with the hydration of portland cement which produces calcium hydroxide (CH) (Glosser et al., 2019). Pozzolanic reactions are caused when the pozzolan itself reacts with calcium hydroxide to form calcium silicate hydrates (C-S-H) (PCA, 2011).

One of the more important characteristics of SCMs is the amount of silica due to its ability to influence the type or amount of hydrates formed during the chemical reactions that occur during mixing (Lothenbach et al., 2011). As stated previously, as currently specified in ASTM 618, "Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete" (ASTM, 2019), and ASTM 311, "Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete (ASTM, 2018), fly ash classification depends on the amount of calcium present in the material but the material itself consists mainly of SiO_2 (Lothenbach et al., 2011). The main reactive elements of fly ash and slag cements consist of aluminosilicate or calcium aluminosilicate glasses, which are mainly composed of SiO_4 and AlO_4 (Skibsted, 2019).

2.2.3: Use and Effects in Concrete

The use of SCMs in concrete can either occur through using blended cements or adding the SCM separately during the mixing process. The following figure summarizes how a variety of fresh properties are affected by the various types of SCMs.

	Fly ash		Slag cement	Silica fume	Natural pozzolans		
	Class F	Class C			Calcined shale	Calcined clay	Metakaolin
Water demand	↓	↓	↓	↑	↔	↔	↑
Workability	↑	↑	↑	↓	↑	↑	↓
Bleeding and segregation	↓	↓	↕	↓	↔	↔	↓
Setting time	↑	↕	↑	↔	↔	↔	↔
Air content	↓	↓	↔	↓	↔	↔	↓
Heat of hydration	↓	↕	↓	↔	↓	↓	↔

Key: ↓ Lowers ↑ Increases ↕ May increase or lower ↔ No impact

* The properties will change dependant on the material composition and dosage and other mixture parameters. Adapted from Thomas and Wilson (2002).

Figure 1: Effects of SCMs on Fresh Concrete (from Kosmatka and Wilson 2016)

One of the key benefits of the introduction of fly ashes into fresh concrete is the decrease in water needed while simultaneously providing an increase in the workability. Class F fly ashes also increase the strength of hardened concrete, but the benefit is not usually seen until at least two weeks of curing has occurred; however, the strength benefits of using a slag product can be seen within the first seven days (Mehta & Monteiro, 2014).When examined under a microscope, it is apparent that fly ash particles are spherical in shape, giving the material the ability to provide a “ball bearing” effect in concrete that overall improves workability and pumpability (Tritsch et al., 2020).

SCMs also typically increase the long-term durability performance of concrete, which typically increases the lifespan of the concrete structure and reduces maintenance needs, in addition to improving mechanical performance (Juenger et al., 2019). When used as a substitute for a portion of portland cement, fly ash is able to decrease concrete’s permeability which prevents against harmful agents attacking the concrete pore structure and the corrosion of any rebar present (Tritsch et al., 2020).

2.2.4: Impacts from Previously Used SCMs

Although only one of the available options for reducing the amount of cement produced, the addition of SCMs is the most economical and practical and has a straightforward process for application (Yang et.al, 2015). In a comprehensive study that created a database of 5,294 laboratory concrete mixes and 3,915 concrete plant mixtures, it was determined that the type of SCM used, and its substitution rate can both be simply selected in order to achieve a certain strength and rate of reduction for CO_2 (Yang et al., 2015). When the rate of replacement of an SCM is between 15% to 20%, the amount of CO_2 produced decreases sharply and then gradually with further increases in replacement rates (Yang, 2020).

However, due to environmental concerns from the burning of coal, new emission standards have been implemented by a number of states in the United States. Additionally, due to the rise in the desirability of natural gas as a source for power production, there has not been new construction of coal-fired power plant since 2013 in the United States (Al-Shmaisani et. al, 2019). In the period of time between 2010 and

2017 the amount of fly ash used in concrete has increased by 28% despite the fact that production of the material has fallen by 44% in the same time period, along with the new emission control systems installed in coal-fired plants due to new emission standards causes a reduction in the quality of fly-ash produced (Al-Shmaisani et al., 2019).

2.3: J-Rox

2.3.1: Source Materials of J-Rox

J-Rox is an industrial byproduct that is produced during the production of phosphoric acid (Novaphos, 2020). In 2006, the four main states that mined phosphate rock used to produce phosphoric acid were Florida, North Carolina, Idaho, and Utah (OAR, 2009). Phosphoric acid is the second leading inorganic acid produced and consumed following sulfuric acid in terms of volume (IHS Markit, 2018). In 2017, the United States produced 8.4 million tons of phosphoric acid that were distributed among many important industries in the country, such as farming (ECI, 2017). Up to 90% of the phosphoric acid produced is converted into three phosphate salts that are used for fertilizers, with the remaining 10% being used in various ways such as supplements for livestock feed (ECI, 2017).

The general process flow to support production of phosphoric acid begins with the mining of base material of phosphate ore, which includes mine tailings (including 3-20% P_2O_3), silica sand, and per coke or coal (Novaphos, 2020). While many SCMs are by-products of other production materials, J-Rox is an actual co-product of the production process in addition to producing both Tech Grade and SPA phosphoric acid (Novaphos, 2020).

2.3.2: Production Process

J-Rox is produced by a company producing phosphoric acid located in Fort Meade, Florida. To date, there have been three main processes used commercially for production of phosphoric acid: wet, thermal, and dry kiln (Guichon Valves, 2019). J-Rox has been produced in both a kiln process and furnace process, using a dual-kiln demo plant being built in 2017 and 2018 (Novaphos, 2020). The dual-kiln process included the coupling of an induration kiln ahead of the reduction kiln in order to reduce the amount of dust that was created by the kiln process (Novaphos, 2020). However, in the late summer of 2018 the kilns were de-coupled, and the process was changed to the Rotary Hearth Furnace (RHF) which was able to solve the issues that were experienced with the kiln production method (Novaphos, 2020). The following figures show the production process.

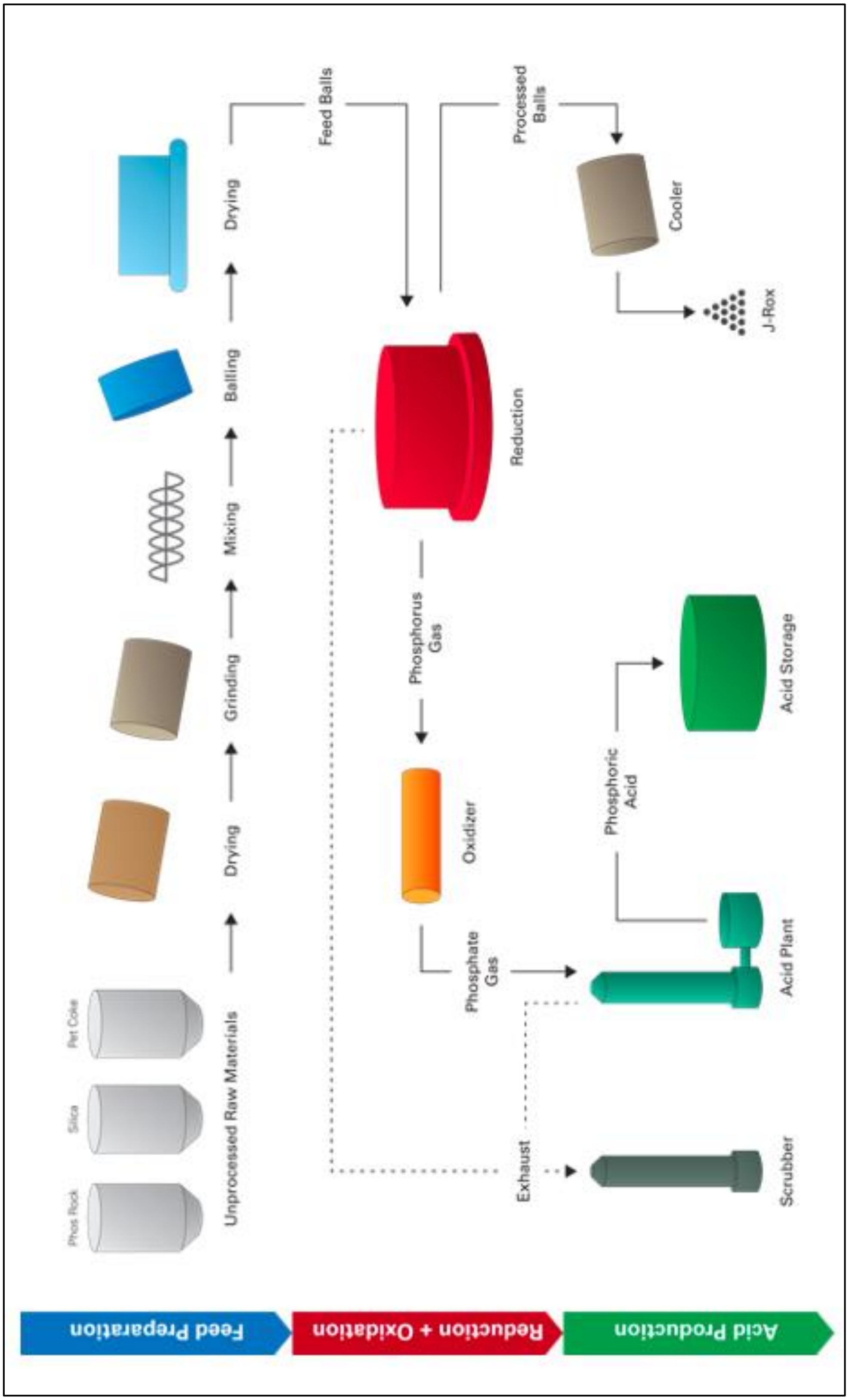

Figure 2: J-Rox Production Process (Novaphos, 2020)

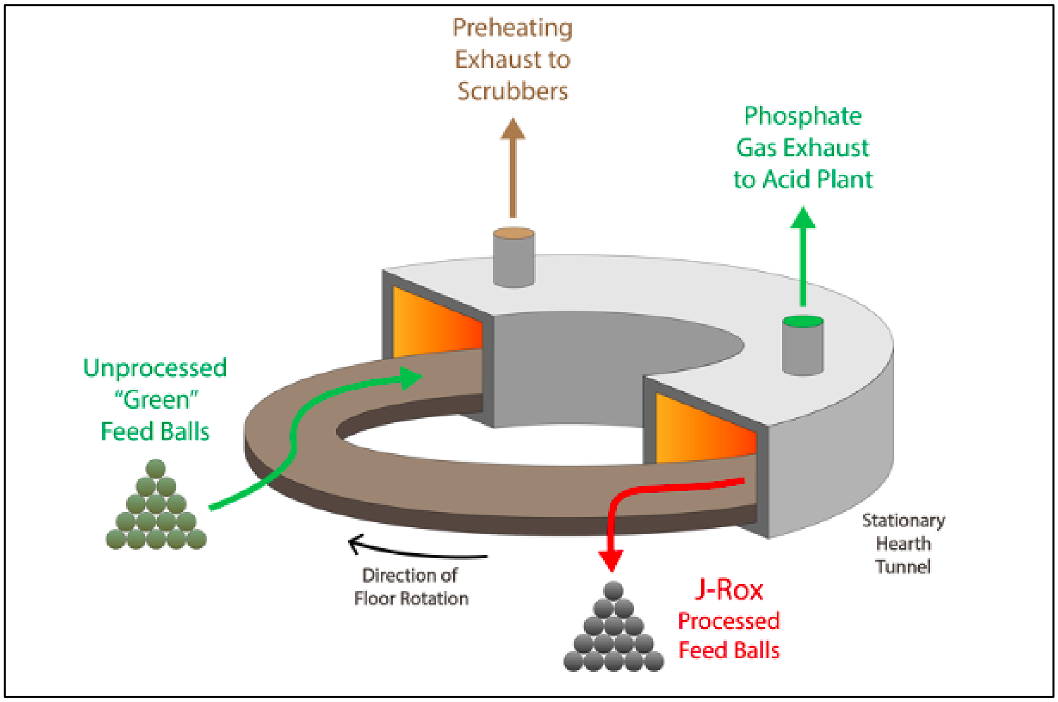

Figure 3: Rotary Hearth Furnace (Novaphos, 2020)

At time of writing of this thesis, the production process is still being improved upon. The design and installation of a larger, annular, and segmented pilot scale rotatory hearth was one of the company's goals when testing of this product began (Novaphos, 2020). This includes designing and building a 16-foot diameter hearth, furthering the development of commercial models, along with the testing of the material for potential application purposes (Novaphos, 2020). The new hearth was completed in March of 2021 and was continued to be updated as material testing proceeded during this research study.

Since the testing process the production process was adjusted several times in order to achieve a certain chemical composition of the J-Rox, several iterations of J-Rox with varying chemical compositions and particle characteristics were produced and tested in this project. Changes to the process included the addition of more silica during production as well as switching the furnace from gas heated to electrically heated in an attempt to reduce the amount of CO_2 that was being produced. Further discussion on these process enhancements and the impact on the J-Rox byproduct produced after each enhancement effort is presented in Chapter 3 and Chapter 4.

2.3.3: Chemical Composition of J-Rox

The figure below shows the primary chemical reactions that occur during the production process of phosphate, phosphoric acid, and the source material for J-Rox. The figure shows how SiO is added to the production process to absorb the leftover calcium (Ca) which creates a chemical bond similar to those seen in other SCMs. The chemical composition was the main factor that was altered during the testing process of J-Rox in order to achieve certain performance results. The amount of silica in the final J-Rox byproduct played a large role in how the material performed overall, and therefore was targeted during production altering.

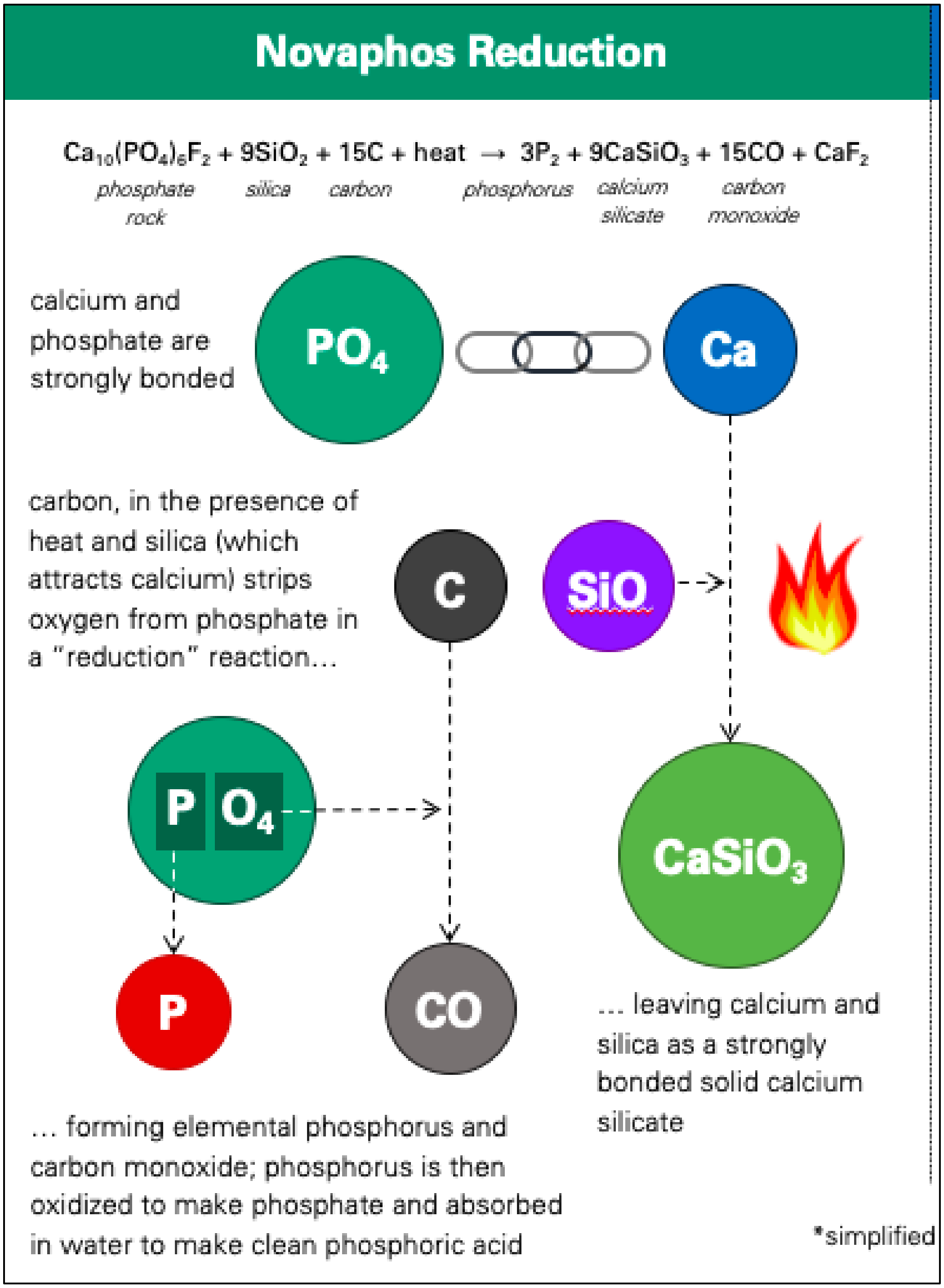

Figure 4: Chemical Reactions During Production (Novaphos, 2020)

2.3.4: Impacts from Using J-Rox

Due to the reduced availability of fly ash and the environmental issues caused by the production of portland cement, it is important that new materials that can perform the same benefits to concrete. Of the 14 phosphoric acid plants across the United States that were in use in 2006, a total of only 1.17 tons of CO_2 were produced (OAR, 2009). The market for phosphoric acid showed growth of 4.5% from 2008 to 2018 (IHS Market, 2018). Assuming that the 4.5% growth rate can be applied to both 2006 and 2007 as well, it can be assumed that the total tonnage of CO_2 in 2018 produced from those 14 plants only grew to 14.7 tons.

The use of phosphorous in concrete and how it affects hydration and overall property development has previously been an interest of the industry. Research has been conducted on levels of P2O5 in cement, however the total composition of that chemical compound has been between 0.5 to 1.1% (Boughanmi et al., 2018). At this level, there is not a significant effect on the concrete produced or the hydration process, and therefore it is important to continue testing the potential effects of this compound when it exists in higher levels as is the case with some J-Rox types.

2.4: Tests to Evaluate Performance of Cementitious Materials

The use of different cementitious materials in concrete mixtures can create various outcomes in fresh and hardened concrete which are analyzed through a variety of test methods. In order to determine the viability of J-Rox as a SCM that could potentially be used as a replacement for materials such as fly-ash in concrete used for transportation and other applications, a variety of material testing must be completed per the ASTM and

AASHTO standards typically used by state highway agencies, other owners, and concrete producers to evaluate its performance.

2.4.1 Assessing similarity to other pozzolans

To be a pozzolan, the SCM must have a chemical composition that enables it to perform the necessary chemical reactions when introduced to water and cement. The set times of SCMs vary depending on their type and the amount of the replacement rate used. Fly ash typically will extend the set time of the concrete exponentially as the rate of replacement is increased (Tritsch et al., 2020). This phenomenon can be individually tested through tests such as ASTM C191 “Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle” (ASTM, 2017).

2.4.2 Fresh property tests

Fresh property tests include slump, air content, unit weight and temperature of freshly mixed concrete. Concrete mixtures must typically meet a range of specified slump and air content values. The actual test results will vary depending on the climate, weather, the process used for concrete placement, and other conditions occurring while concrete is in the fresh state.

There is a variety of information that can be determined about both the fresh and hardened sample through the use of fresh property testing. Slump and unit weight help to ensure that the overall mixture includes accurate proportions of the different materials necessary to make concrete, which in turn gives insight to what the future compressive strength will be. Air content gives insight to how the specimen will act under different environmental conditions such as freezing and thawing when wet. The temperature that the concrete is created and cures in also plays a role on its performance since extreme temperatures can cause premature shrinking or cracking if the proper precautions to keep the concrete at an acceptable temperature for heat of hydration.

2.4.3 Mechanical property tests

Mechanical properties of concrete are important to understand how the material is going to perform during its lifetime. These properties include modulus of elasticity, shrinkage, and compressive strength. Both modulus of elasticity and shrinkage are affected by the characteristics of the materials used and their proportions in the concrete mixture (Mehta & Monteiro, 2014). For example, the porosity of aggregate and the cement paste matrix are two factors that can increase the elasticity of a concrete specimen (Mehta & Monteiro, 2014). Also, the addition of pozzolans in concrete increases the amount of fine pores which can increase the amount of shrinkage that occurs; however, the use of water reducers can ensure shrinkage does not increase (Mehta & Monteiro, 2014).

Compressive strength is dependent on the water-cement ratio as well as the porosity of the sample. A higher water-cement ratio and higher porosity will both lead to decreases in strength. Air entrainment can also have an effect on compressive strength due to the fact that it is introducing more pores into the system, but extreme adverse effects on strength are not usually seen until the water-cement ratio exceeds 0.50 (Mehta & Monteiro, 2014). Compressive strength is also subject to the conditions that it experiences during curing and loading, as well as the time it has to cure before compression to failure occurs.

2.4.4 Durability performance tests

The concrete durability performance benefits obtained through the use of SCMs has been thoroughly studied. However, for many new and emerging SCMs, the effect on concrete durability is still unknown. Despite the lack of research however, there is a correlation between the type of hydrates formed and the durability of concrete produced, so therefore it may be possible to assume that new types of SCMs will have the same impact on durability (Juenger et al., 2019). This correlation allows developers of alternative SCMs to optimize the chemical composition, texture, and fineness of their materials to ensure strong odds of providing adequate mechanical performance and durability performance benefits.

The durability performance of concrete is heavily reliant on its microstructure and its ability to resist the penetration of harmful materials, and a useful test of this is electrical resistivity. As the microstructure ages during curing the porosity of it decreases causing electrical resistivity to increase (Azarsa & Gupta, 2017). Therefore, having a high resistivity indicates a lower pore system connectivity for harmful agents to enter and negatively impact the durability of the concrete. The relationship between resistivity values for fly ash and concrete have been tested and it is known that the resistivity values of concrete with fly ash generally show a sharp increase in later age (>28 days) testing. Based on the previous statement that it can be assumed new types of SCMs should show the same durability characteristics, it could reasonably be assumed that they will also show a large increase in resistivity values in later age testing when compared to concrete without the presence of SCMs.

2.5: Research Needs

As discussed in this chapter, the effects of SCMs such as fly ash and slag cements on concrete performance has been extensively researched to determine the benefits they provide. With the increasing need to find new materials that can be used as SCMs in concrete due to environmental concerns and future material shortages (particularly of fly ash), new SCMs need to be identified and tested with a sense of immediacy.

The goal of this project was to determine if an industrial byproduct material (J-Rox) produced by a phosphoric acid producer is suitable for use as a SCM in concrete applications. The suitability of this byproduct for use as an SCM was evaluated by performing a variety of tests on paste, mortar, and concrete specimens using certain percentages of replacement of cement by weight with J-Rox and comparing those results with those of a 100% cement control as well as a 20% fly ash replacement control. The results from this project will assess byproduct of the phosphoric acid production for a beneficial reuse that will benefit both the company and future concrete that is produced with the material. This research should also provide more information to support the potential uses of other byproducts, co-products, or recycled materials for use in concrete if they have the same (or similar) chemical structure or properties of the J-Rox material.

CHAPTER 3 : METHODOLOGY

3.1 Introduction

The most promising J-Rox chemical composition and particle characteristics were determined based on the results for paste, mortar, and concrete properties obtained from several tests discussed in Chapter 2. To evaluate the pertinent properties, specific test methods were performed on samples of paste, mortar, and concrete. The effects of J-Rox on mortar and concrete were compared to mixtures produced using cementitious materials that are currently used in concrete mixtures: a 100% portland cement control mixture and a 20% replacement of fly ash control mixture. In addition to those controls, the different types of J-Rox were added in replacement increments of 15%, 20%, and 25%. The replacement percentage and the type of J-Rox used were to two variables that changed throughout the testing procedures. The following chapter describes the methodology used in the laboratory and testing program, as well as a description of the materials used in these processes.

3.2 Materials Description and Characteristics

The following section discusses the materials used for the variety of test procedures. The source location of these materials and key material properties that were either determined through in-house testing or test results provided by the material supplier are also included.

3.2.1 Cementitious and Supplementary Cementitious Materials

The cementitious material used in testing was an ordinary portland cement. The two supplementary cementitious materials used were fly ash and a variety of samples of the J-Rox material. Further discussion of their source and characteristics continues below.

3.2.1.1. Cementitious Materials

Ordinary portland cement was used for the creation of all paste, mortar, and concrete samples. This cement is classified as an OPC Type I/II cement that was sourced from LafargeHolcim in Holly Hill, South Carolina and had a specific gravity of 3.15. The fly ash used is a Class F sourced from the Roxboro power plant in North Carolina with a specific gravity of 2.29. The mill certificate for the cement and the chemical analysis of the fly ash are included in Appendix B. The following table compares the chemical compositions of a typical cement and fly ash samples to those of the products used in this study.

Table 3.1: Chemical Composition Comparison of Samples (Kosmatka et al., 2014)

Chemical Composition (%)	Material Type									
	Typical Type I Cement	Typical Type II Cemen t	Typical Class F Fly Ash	Typical Class C Fly Ash	Type I/II OPC used in this study	Class F Fly Ash used in this study	J-Rox 1	J-Rox 2	J-Rox 3	J-Rox 4 Air Cooled
SiO_2	20.5	21.2	52	35	20.1	52.7	61.8	30.3	63.4	58.9
Al_2O_3	5.4	4.6	23	18	4.5	26.7	0.8	2.1	0.9	0.8
Fe_2O_3	2.6	3.5	11	6	3.5	11.12	1.5	1.8	1.5	1.7
CaO	63.9	63.8	5	21	63.6	2.1	24.1	42.6	24	20.6
MgO	2.1	2.1	-	-	1.4	1.1	2.1	4.4	2.1	1.7
SO_3	3	2.7	0.8	4.1	3.2	1.9	-	-	-	-
P_2O_5	-	-	-	-	-	0.21	4.2	11.8	2.5	11.4

3.2.1.2 J-Rox

Due to J-Rox being a material in development, the producer sent a variety of samples with different chemical compositions throughout the course of this project. After receiving results from the paste and mortar tests performed on each J-Rox sample, the company would alter certain aspects of the J-Rox production to improve the material, and then send a new sample. These changes ranged from changing the production methods (which altered the chemical composition) or changing the fineness of the material through the grinding process.

The first samples received were J-Rox 1 Furnace (JR1F) and J-Rox 1 Kiln (JR1K). Note that the sample IDs take the form JR1, or J-Rox 1. A finely ground sample and a coarsely ground sample of each was provided. Due to production concerns, Novaphos determined the kiln would not be part of their production process moving forward. Therefore, the majority of testing of this sample was on the JR1F fine and coarse materials. Paste and mortar testing was performed on these samples.

After testing of the J-Rox 1 samples, a low silica version of J-Rox was provided which was called J-Rox 2. Again, fine, and coarse samples of this material was provided and paste, and mortar testing was performed. While testing of these samples was ongoing four drums of J-Rox were delivered with similar chemical characteristics to that of J-Rox 2. These drums were named J-Rox A, J-Rox B, J-Rox O, and J-Rox X and only mortar testing was performed on these samples.

After the lower-silica J-Rox 2 showed inferior strength gain in mortar samples, production was altered to produce J-Rox with a chemical composition more similar to that of the JR1F material. This resulted in production of J-Rox 3, the clinker for which was hand-picked by the team at Novaphos for use prior to grinding to reach a desired fineness. This material was received when Novaphos was in the final stages of finalizing their new production plant. J-Rox 3 was the first J-Rox sample that was used to create concrete samples, in addition to the paste and mortar samples prepared for the previous J-Rox formulations.

Once the producer's new plant became operable, J-Rox 4 was produced. This material had a chemical composition similar to JR1F and J-Rox 3, although the P_2O_5 content was somewhat higher. J-Rox 4 and was used to create paste and mortar samples. Both water-quenched (JR4W) and air-quenched (JR4A) versions of this material were provided in order to determine the differences in performance that could be observed between J-Rox produced using the two different process-finishing methods. The production process of this material also included a heating process that was powered through gas resulting in a higher quantity of CO_2 existing during production; and overall resulting in a higher level of P_2O_5 in the J-Rox 4 samples.

3.2.2 Aggregates

Two coarse aggregates were tested for use in concrete for this project. The first coarse aggregate used was a coastal limestone aggregate sourced from the Martin Marietta's Castle Hayne Quarry in North Carolina. This material was initially planned to be the aggregate used for all mixes in order to create a design that was more similar to those used by Florida Department of Transportation (FDOT). However, due to the quarry ceasing production of No. 67 graded aggregates, only the two control mixtures could be produced using this material. The coarse aggregate was then switched to a granitic gneiss sourced from the Wake Stone's Triangle Quarry in Cary, North Carolina. All mixtures that were produced using the coastal limestone prior to this switch were re-batched and re-tested with the granite coarse aggregate. Concrete and mortar mixtures used a natural silica sand was sourced from a pit in Lemon Springs, North Carolina, which meets ASTM C33 "Standard Specification for Concrete Aggregates" (ASTM, 2018). All aggregates were allowed to air dry prior to being used in mixes, so that a steady moisture state could be accounted for with the batch water.

3.2.3 Chemical Admixtures

Use of J-Rox is being targeted in Florida, since that is the state where it is produced. Due to the lack of significant freeze-thaw conditions in Florida, there is not an entrained air target for FDOT concrete mixtures. Therefore, mixtures did not contain an air entraining admixture, and the only chemical admixture used was a water reducer (WRA). MasterPolyheed 997 is a mid-range water reducer manufactured by BASF Construction Chemicals in Denver, Colorado. This use of this material in the mixture design was to increase workability in the concrete to help ensure quality samples for hardened property testing were produced.

3.3 Testing Program

The testing program was separated into four sections: testing of paste samples, testing of mortar samples, testing of fresh concrete properties, and finally testing of hardened concrete properties. The following table shows each section and the test methods that are associated with them. Test methods were performed in accordance with ASTM and/or AASHTO standards. Each test shown was performed on both control samples followed by the J-Rox replacement samples.

Table 3.2: Testing Program

Sample Type	Name of Test	Test Standard	Testing ages (days)	Number of Replicates
Paste	Set Time	ASTM C191	1	1
Mortar	Strength Testing of Mortar Cubes	ASTM C109	3, 7, 28, 90	3
Fresh Concrete	Temperature	AASHTO T 309	Fresh	1
	Slump	ASTM C143	Fresh	1
	Air Content	ASTM C231	Fresh	1
	Fresh Density (Unit Weight)	ASTM C138	Fresh	1
Hardened Concrete	Resistivity	AASHTO T 358	3, 7, 28, 56, 90	3
	Compressive Strength	ASTM C39	3, 7, 28, 56, 90	3
	Modulus of Elasticity and Poisson's ratio	ASTM C469	28	2-3
	Shrinkage	ASTM C157	Per Standard	3

3.4 Batching and Mixing Paste Samples

The first step taken to determine any similar characteristics between the test material J-Rox and its control cementitious materials—ordinary portland cement and fly ash—was to test the paste samples of each to determine the initial and final set times of each material. This was completed through the use of ASTM C191 “Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle” (ASTM, 2019) and ASTM C305 “Practice for Mechanical Mixing of Hydraulic-Cement Pastes and Mortars of Plastic Consistency” (ASTM, 2020). The apparatus used for creating paste samples was a Hobart mortar mixer.

Table 3.2 below shows the mixture proportions and mixture IDs for the paste samples. As can be seen in Table 3.2, the water content used in each mixture varies slightly. This is due to the fact that the paste sample must be of normal consistency as per the ASTM C187 “Standard Test Method for Amount of Water Required for Normal Consistency of Hydraulic Cement Paste” (ASTM, 2016). This standard defines normal consistency of the paste sample to be when the rod of the Vicat needle apparatus falls from the surface of the sample to a point on the graduated scale that is 10 +/- 1 mm below the surface of the sample in the first 30 s after mixing (ASTM, 2016).

Table 3.3: Paste Mixture Proportions and IDs

Sample	OPC (g)	Fly Ash (g)	Water (g)	J-Rox furnace fine-grind (g)	J-Rox furnace coarse grind (g)
100% Cement Control	650	0	195	0	0
20% Fly Ash Control	520	130	185	0	0
15% J-Rox Furnace fine-grind	550	0	185	100	0
15% J-Rox Furnace coarse-grind	550	0	190	0	100
25% J-Rox Furnace fine-grind	490	0	185	160	0
25% J-Rox Furnace coarse-grind	490	0	190	0	160
15% Low Silica fine-grind	550	0	190	100	0
15% Low Silica coarse-grind	550	0	195	0	160
25% Low Silica fine-grind	490	0	200	100	0
25% Low Silica coarse-grind	490	0	195	0	160
15% J-Rox 3	550	0	185	100	0
25% J-Rox 3	490	0	185	160	0
15% J-Rox 4 Water Quenched	550	0	185	100	0
25% J-Rox 4 Water Quenched	490	0	185	160	0
15% J-Rox 4 Air Cooled	550	0	185	100	0
25% J-Rox 4 Air Cooled	490	0	185	160	0

3.5 Batching and Mixing Mortar Samples

Following ASTM C305, the Hobart mortar mixer was again used for the mixing of mortar samples batched with the ratio of 2.75 kg silica sand to 1.00 kg cementitious material. Mortar cube molds were sprayed with form release prior to being filled and tamped in two lifts, and finally allowed to cure for 24 hours before being demolded and placed in a curing room that applies a constant mist to the samples and is in accordance with ASTM C511, “Standard Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes” (ASTM, 2019). The cubes remained in the curing room until their specific test day arrived, in which they were then broken according to ASTM C109/109M, “Standard Test Method for Compressive Strength of Hydraulic Cement Mortars” (ASTM, 2020).

Table 3.3 shows the mixture proportions and IDs for the mortar samples. As can be seen in Table 3.3, the water content used in each mixture varies slightly. This due to the fact that ASTM C109 requires that the water content of the sample is able to produce a flow of 110 +/- 5 in 25 drops of the flow table to reach normal consistency for mortar samples following the procedure of ASTM C230/C230M-21 “Standard Specification for Flow Table for Use in Tests of Hydraulic Cement” (ASTM, 2021).

Table 3.4: Mortar Mixture Proportions and IDs

Sample	Mortar Components					
	OPC (g)	**Fly Ash (g)**	**Water (g)**	**J-Rox furnace fine-grind (g)**	**J-Rox furnace coarse-grind (g)**	**ASTM C33 Natural Silica Sand**
100% Cement Control retest	1000	0	535	0	0	2750
20% Fly Ash Control retest	800	200	515	0	0	2750
15% J-Rox fine-grind	850	0	515	150	0	2750
15% J-Rox coarse-grind	850	0	515	0	150	2750
25% J-Rox fine-grind	750	0	515	250	0	2750
25% J-Rox coarse-grind	750	0	515	0	250	2750
15% J-Rox low silica fine-grind	850	0	515	150	0	2750
15% J-Rox low silica coarse-grind	850	0	515	0	150	2750
25% J-Rox low silica fine-grind	750	0	515	250	0	2750
25% J-Rox low silica coarse-grind	750	0	515	0	250	2750
100% Cement Control - initial	1000	0	535	0	0	2750
20% Fly Ash Control - initial	800	200	515	0	0	2750
15% J-Rox 4 WQ	850	0	530	0	150	2750
25% J-Rox 4 WQ	750	0	530	0	250	2750
15% J-Rox 4 AC	850	0	530	0	150	2750
25% J-Rox 4 AC	750	0	530	0	250	2750

3.6 Batching and Mixing Concrete Samples

The mixtures produced for this project consisted of the 100% OPC control mixture, the 20% fly ash control mixture, and J-Rox mixtures with 15%, 20%, and 25% replacement rates. Additional materials—coarse aggregate, fine aggregate, cement type, and admixtures—all remained constant for each mix. The mixture proportions are shown in Table 3.4.

Table 3.5: Concrete Mixture Proportions and IDs

Mixture	**Concrete Mixture Component (pcy)**						
	OPC	**Fly Ash**	**J-Rox**	**Water**	**Coarse Aggregate**	**ASTM C33 Natural Silica Sand**	**Water Reducer (mL)**
100% Cement Control - limestone CA	658	0	0	269	1591	1419	200
20% Fly Ash Control - limestone CA	526	132	0	269	1591	1378	210
100% Cement Control - granitic gneiss CA	658	0	0	269	1814	1308	220
20% Fly Ash Control - granitic gneiss CA	526	132	0	269	1814	1267	200
15% J-Rox 3 - granitic gneiss CA	559	0	99	269	1814	1273	200
20% J-Rox 3 - granitic gneiss CA	526	0	132	269	1814	1261	200
25% J-Rox 3 - granitic gneiss CA	494	0	165	269	1814	1249	200
15% J-Rox 4 - granitic gneiss CA	559	0	99	269	1814	1273	200
20% J-Rox 4 - granitic gneiss CA	526	0	132	269	1814	1261	200
25% J-Rox 4 - granitic gneiss CA	494	0	165	269	1814	1249	210

Mixture designs were created using the American Concrete Institute (ACI) 211.1, "Standard Practice for Selecting Proportions for Normal, Heavyweight, and Mass Concrete," design method for one cubic yard (ACI, 2002). The design was created to meet the requirements of having a slump between three and four inches, a maximum aggregate size of ¾ in, and a maximum cementitious material content of 658 pounds per cubic yard (pcy). The water cement ratio for all mixtures was set at 0.41, with the requirements from the client indicating it should be between 0.38 and 0.42. Using a water cement ratio of 0.41 allowed adequate workability of the concrete mixtures for preparing samples while still remaining suitably low enough to support strong odds of reasonable strength gain.

The batch quantities produced were 2.5 cubic feet (cf) for all control and J-Rox 4 mixtures. A quantity of 2.25 cf mixes was used for the J-Rox 3 concrete batches due to the amount of material available for use. Casting of the concrete specimens used for testing followed ASTM C192, "Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory," and were placed in the same moist room that mortar samples were placed in after demolding (ASTM, 2019). ASTM and AASHTO standards associated with the creation of concrete samples were followed as well. In order to easily remove the hardened concrete from the cylinder and beam molds, form release was applied to the molds prior to use.

3.6.1 Slump

ASTM C143, "Standard Test Method for Slump of Hydraulic-Cement Concrete," was used to perform slump testing on each concrete mixture (ASTM, 2020). Slump testing was performed in order to determine if J-Rox was able to perform the same increase in workability that fly ash provides for fresh concrete samples. The targeted slump for this project was 3 to 4 inches.

3.6.2 Air Content

While there was not a specific air content percentage required for these concrete mixtures, the test was still performed in accordance with ASTM C231, "Standard Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method" (ASTM, 2017). An air content of between 2 and 3% was considered appropriate since there was no use of an air-entraining admixture. The reason that air content not being required for this project is due to FDOT not requiring the need for air entrained concrete since the state typically does not experience freeze-thaw in their concrete.

3.6.3 Unit Weight

Fresh unit weight data was collected after completion of the air content test in accordance with ASTM C138, “Standard Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete” (ASTM, 2017). This is performed to ensure that the air content achieved through the pressure method is accurate and if the proportion of materials in the mixture were correctly proportioned.

3.7 Testing of Hardened Concrete

After the samples cured in the moist room for the required time span, mechanical and durability tests were performed to determine important characteristics about the performance of hardened concrete. Mechanical testing for this project included compressive strength, Modulus of Elasticity and Poisson’s Ratio, and shrinkage, while durability testing consisted of surface resistivity.

3.7.1 Compressive Strength

Compressive strength testing was performed on 4 in by 8 in cylinders in accordance with ASTM C39, “Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens” (ASTM, 2021). Data was collected at the test ages of 3, 7, 28, 56, and 90 days after the day the concrete was mixed and formed. The required minimum compressive strength at 28 days was a 5500 psi average of all samples.

3.7.2 Modulus of Elasticity and Poisson's Ratio

Modulus of elasticity and Poisson's ratio test specimens were 6 in by 12 in cylinders. This test was performed in accordance with ASTM C469, "Standard Test Method for Static MOE and Poisson's Ratio of Concrete Compression" (ASTM, 2014). This test was performed 28 days after the mixing date, with the compressive strength cylinders being broken prior to this test being run to ensure that ASTM C469's requirement of exceeding 40% of the ultimate strength for the load and displacement measurements was met.

3.7.3 Shrinkage

ASTM C157, "Standard Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete," was used to perform the unrestrained shrinkage test (ASTM, 2017). Three beams measuring 4 in by 4 in by 11 in were created for this test that had gauge studs molded into the center of each end during placement. The specimens were placed in a limewater bath after being demolded for thirty minutes and then tested to obtain an initial reading. They were then returned to the water bath where they remained for 28 days after the mixing date. At the 28 day mark they were measured again and then left to cure in a temperature- and humidity-controlled environmental chamber. The temperature of the chamber was controlled to be 73 degrees Fahrenheit, with a plus or minus (+/-) 3 °F tolerance, and have a relative humidity of 50%, with a plus or minus (+/-) 4% tolerance.

3.7.4 Surface Resistivity

The AASHTO T 358, "Standard Method of Test for Surface Resistivity Indication of Concrete's Ability to Resist Chloride Ion Penetration" was used for durability testing of samples (AASHTO, 2017). This test utilizes a non-destructive test method that measures the resistivity of 4 in by 8 in cylinders after they are removed from the moist curing room and were still in a wet state. This was done at 3, 7, 28, 56, and 90 days post mixing date prior to the compressive strength test being run.

CHAPTER 4 : TEST RESULTS

The following chapter provides a summary of the data that was collected through the testing program outlined in Chapter 3 of this thesis. As previously stated, control samples are labeled as either 100% OPC or 20% FA. J-Rox sample types were designated by three characteristics: the percent replacement that was used (15%, 20%, or 25%), the type of J-Rox that was used (J-Rox 1 through J-Rox 4), and the fineness level (fine-grind or coarse-grind) if applicable. Table 4.1 shows the sample designations that will be used to label data results in this chapter.

Table 4.1: Sample Designations

Sample	Designation
100% Cement Control	100 OPC
20% Fly Ash Control	20 FA
15% J-Rox Furnace fine-grind	15 JR1 FG
15% J-Rox Furnace coarse-grind	15 JR1 CG
25% J-Rox Furnace fine-grind	25 JR1 FG
25% J-Rox Furnace coarse-grind	25 JR1 CG
15% Low Silica fine-grind	15 JR2 FG
15% Low Silica coarse-grind	15 JR2 CG
25% Low Silica fine-grind	25 JR2 FG
25% Low Silica coarse-grind	25 JR2 CG
15% J-Rox Drum O fine-grind	15 JRO FG
25% J-Rox Drum O fine-grind	25 JRO FG
15% J-Rox Drum X fine-grind	15 JRX FG
25% J-Rox Drum X fine-grind	25 JRX FG
15% J-Rox Drum A coarse-grind	15 JRA CG
25% J-Rox Drum A coarse-grind	25 JRA CG
15% J-Rox Drum B coarse-grind	15 JRB CG
25% J-Rox Drum B coarse-grind	25 JRB CG
15% J-Rox 3	15 JR3
25% J-Rox 3	25 JR3
15% J-Rox 4 Water Quenched	15 JR4 WQ
25% J-Rox 4 Water Quenched	25 JR4 WQ
15% J-Rox 4 Air Cooled	15 JR4 AC
25% J-Rox 4 Air Cooled	25 JR4 AC

4.1 Testing of Paste Samples

The results for the initial and final set times of the paste samples that were tested using the procedures described in section 3.4 are provided in this section. Table 4.2 below shows the initial set time and final set time for each sample in minutes while Table 4.3 shows the change in distance over the setting period. The lines of the table that are highlighted in green are the samples that were selected to be used to produce concrete samples and different J-Rox samples are separated by thicker lines. This was the first test performed on each sample to determine its similarity to the setting time of samples that consist of only cement or use a replacement of fly ash to determine if further testing should be completed using that type of J-Rox for the production of concrete samples. The samples highlighted in green show those that were used for concrete testing. Final set time is the time it takes for the distance of the needle to reach zero added to the mix and mold time of the specimen. Initial set time was determined using the formula outlined in ASTM C191 which is as follows:

$$Initial\ Set\ Time = \left(\frac{(H-E)}{(C-D)}\right) * (C-25) + E$$

H: Time in minutes of first penetration less than 25 mm

E: Time in minutes of first penetration greater than 25 mm

C: Penetration reading at Time E

D: Penetration reading at Time H

Table 4.2: Initial and Final Set Times of Paste Samples

Sample	Initial Set Time (min)	Final Set Time (min)
100% Cement Control	194	364
20% Fly Ash Control	206	351
15% J-Rox 1 Furnace fine-grind	157	305
15% J-Rox 1 Furnace coarse-grind	188	321
25% J-Rox 1 Furnace fine-grind	253	366
25% J-Rox 1 Furnace coarse-grind	190	334
15% J-Rox 2 Low Silica fine-grind	253	366
15% J-Rox 2 Low Silica coarse-grind	161	366
25% J-Rox 2 Low Silica fine-grind	169	336
25% J-Rox 2 Low Silica coarse-grind	206	336
15% J-Rox 3	197	336
25% J-Rox 3	213	365
15% J-Rox 4 Water Quenched	213	365
25% J-Rox 4 Water Quenched	230	349
15% J-Rox 4 Air Cooled	201	304
25% J-Rox 4 Air Cooled	222	346

Table 4.3: Penetration Reading During Set Time

Time	100 OPC	20 FA	15 JR1 FG	15 JR1 CG	25 JR1 FG	25 JR1 CG	15 JR2 FG	15 JR2 CG	25 JR2 FG	25 JR2 CG	15 JR3	25 JR3	15 JR4 WQ	25 JR4 WQ	15 JR4 AC	25 JR4 AC
30	45	43	41.5	41	40	40.5	40	40	40	41	40	40.3	41	40.5	40.5	41
45	45	41	41.5	40.5	40	40	39.5	40	40	41	40	40.3	40	40.5	40.5	41
60	45	43	41	40.5	40	40	39.5	40	40	41	40	40.3	40	40.5	40	41
75	45	40	40.5	40.25	40	39.5	39.5	40	39.5	40.5	38.5	40	40	40.5	40	40
90	41	40	40	39.5	40	39	40	39.5	39.5	40	38.5	40	40	40.5	40	39.5
105	41	40	40	39.5	39	39	39.5	39.5	39.5	40	38.5	39	40	40.5	39.5	39.5
120	41	40	40	39.5	39	39.5	39	39	39.5	39.5	38.5	39	40	40	39.5	39.5
135	40	40	39.5	37.5	38.5	35	36.5	36.5	39	39.5	38.3	39	39.5	39.5	38.5	39.5
150	40	40	39.5	39	39	32.5	36	28	37	39	38.3	39	39	39.5	38.5	39
165	40	40	23	37	38.5	32.5	18.5	24.5	35	36	37.8	39	38.75	39.5	38.5	39
180	33	30	19	27	40	27	18.5	11	36.5	30.5	37.5	39	38.5	39	26	37
195	21	29	19	15	37	22	14	10	31	25	35.5	36.5	38.5	38.5	26	36
210	17	18	19	10	35	19.5	8.5	8	14.5	16	23.5	23.4	36	38	17.5	25
225	11	14	11	10	31.5	14	6	5.5	13.5	16	22	19	26	29.5	11	25
240	7	12	5.5	3	30	9	3.5	3	9.5	13.5	11	19	5	29	10.5	14.5
255	5	7	3	1.5	19.5	3	3	2	4.5	6	10.5	11	3	28.5	3	9
270	3.5	4	3	0.75	9	2.5	1.25	1.5	2	2.5	6.5	9.5	2	16.5	0.5	8
285	1	4	1	0.5	5	1	1.25	1	1.5	1	4	7.5	1.5	11.5	0.5	3
300	1	3.5	0	0.25	7.5	0.5	0.5	1	0.5	0.5	3	6.5	1	6.5	0	1
315	2	1	0	0	3	0.25	0.5	0.5	0.5	0.5	1	4.5	0.5	2.5	0	0.5
330	1	1	0	0	2	0	0.25	0	0.5	0	0.5	2.5	0.5	1	0	0
345	0.5	0	0	0	1	0	0.25	0	0	0	0.25	1.5	0	0	0	0
360	0	0	0	0	0	0	0	0	0	0	0	0.5	0	0	0	0
375	0	0	0	0	0	0	0	0	0	0	0	0.25	0	0	0	0
390	0	0	0	0	0	0	0	0	0	0	0	0.25	0	0	0	0
405	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

These results are also shown graphically in Figures 4.1 and 4.2 below. Figure 4.1 shows all samples that were tested while Figure 4.2 only compares the samples that were used to make concrete specimens. The types of J-Rox that were used to produce concrete have similar trends over time as the control samples as well as slight variations in initial and final set times. These were chosen in order to achieve the same period of workability that concrete made with fly ash provides.

The variables that affect the set time of the paste samples include the particle fineness, chemical composition, cement replacement rate, and the method used to prepare the sample (Mehta & Monterio, 2014). Particle fineness affects the ability of hydration in cementitious materials by providing more surface area for hydration sites the finer the particles are ground. More nucleation sites decrease the time needed to reach the initial and final set times. This explains why the finer ground samples reached set times much quicker than their coarse ground counterparts. Calcium, silica, and aluminum are three of the most important elements to form the crystalline structure created when cementitious materials are hydrated due to the various compounds they create. The greater the abundance of these elements, the quicker the formation of the structure and overall, and the faster the initial and final set times can be achieved. Factoring in the chemical compositions with the particle size shows that the amount of silica in the J-Rox sample affects the set time since results of the fine ground samples with lower silica contents took longer to reach initial and final set compared to those that had higher silica contents.

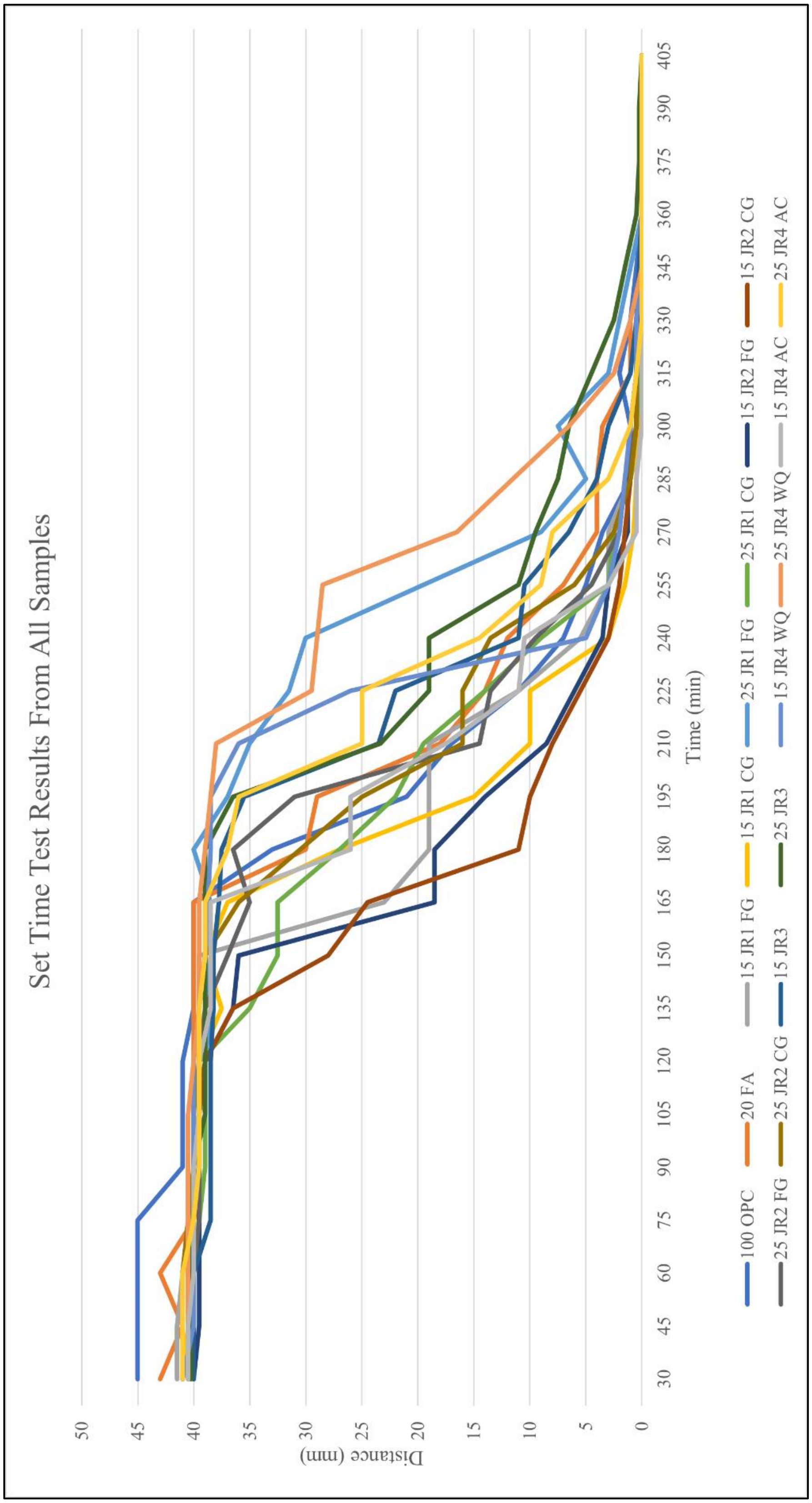

Figure 5: Set Time Results for All Samples

Comparing the test results for J-Rox samples that were used to produce concrete, the initial set time of all samples were very close to each other. The sample with the most similar initial set time to the controls was the 15 JR4 AC which was only seven minutes greater than the 100 OPC sample and five minutes greater than the 20 FA sample. Following that the next closest samples were the 15 JR3 and 25 JR3 which were both only 19 minutes greater than both controls. The 25 JR4 AC had the greatest difference in initial set time, taking 28 minutes longer than the 100 OPC and 16 minutes longer than the 20 FA to achieve initial set.

Comparing the final set times of the J-Rox samples to those of the controls, the results only showed slight differences. The 15% J-Rox 4 air cooled sample had the greatest difference in final set time than the other samples which were as close to being one minute apart from the 100 OPC and 14 minutes from the 20 FA samples. Comparing the air cooled to the water quenched, the final set time of the water quenched samples was much more similar to the controls but had a greater initial set time. The air cooled samples had a very similar chemical composition to the water quenched samples, so it is likely this difference comes from the particle size distributions. The air cooled sample shows a much more uniform distribution which is more similar to the distributions of the finer ground samples, whereas the water quenched has a left skewed distribution.

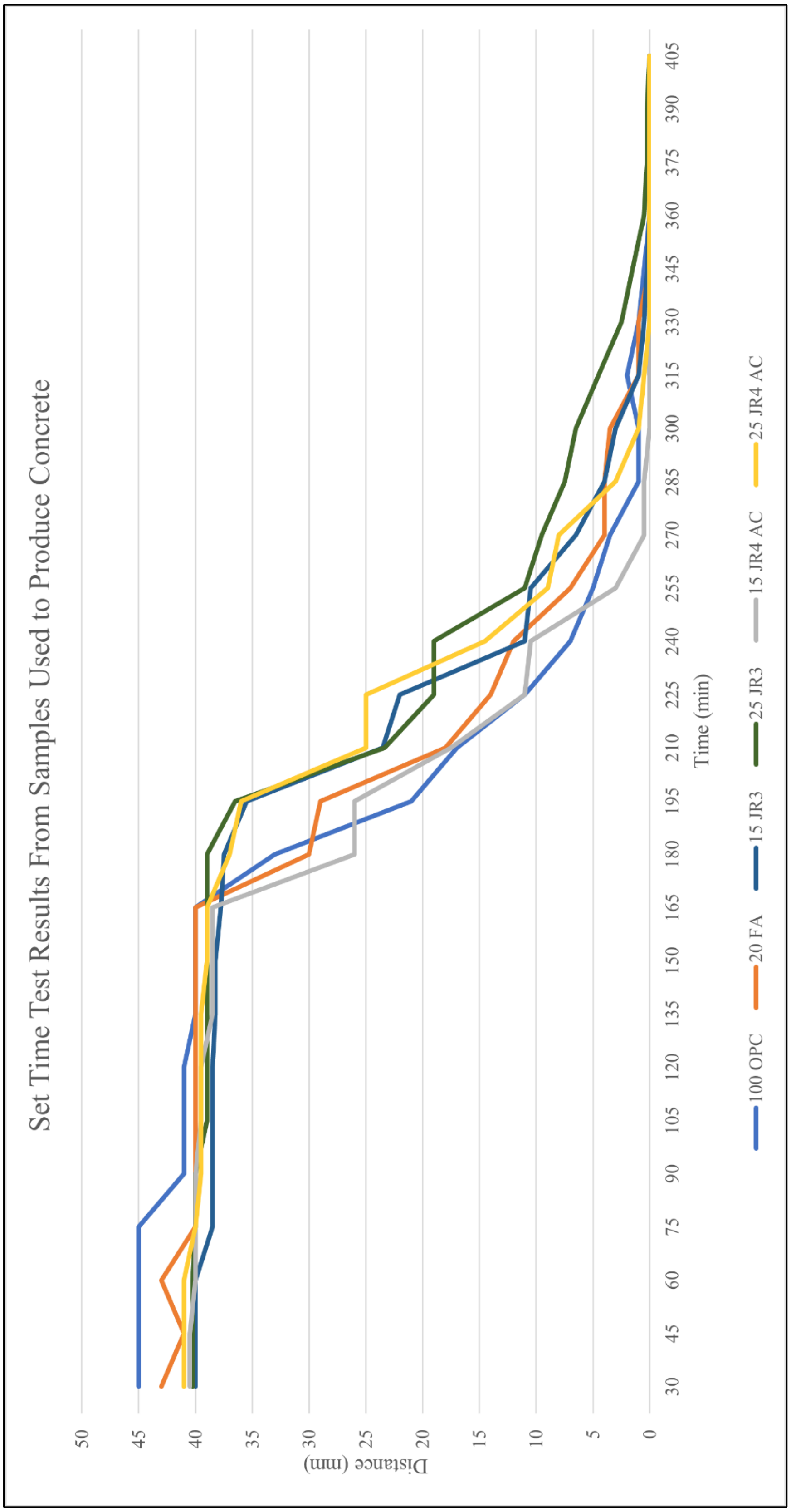

Figure 6: Set Time Results for Samples Used for Concrete

4.2 Testing of Mortar Samples

The results from the testing of mortar cubes for compressive strength were also used to determine the optimal available types of J-Rox to use for concrete testing. Table 4.4 shows the compressive strength results of each mortar sample at 3, 7, 28, and 90 days in psi. As stated previously in section 3.2.1.2, J-Rox Drum A, B, O, and X samples were only tested for mortar compressive strength. Concrete was not made with those samples because they showed similar compressive strength values to those of the low silica material (J-Rox 2), which had low compressive strength compared to control specimens. Again, J-Rox 3 and 4 showed similar results to the controls providing more reason to use those samples for concrete specimens.

When comparing the results among samples, it is evident that the amount of silica in the type of J-Rox shows a positive correlation with the overall strength gain of the sample. Comparing the overall chemistry to the of the J-Rox samples to that of fly ash shows that the main chemical compounds in J-Rox are SiO_2, P_2O_5, and CaO whereas in fly ash they are SiO_2, Fe_2O_3, and Al_2O_3. Those with higher silica contents perform more similarly to the control samples than those with lower silica contents such as J-Rox 2 and the A, B, O, and X drums. The J-Rox 1 and J-Rox 4 samples exhibited a greater 90 day mortar compressive strength than the control samples, while J-Rox 3 showed similar mortar compressive strengths to the fine ground drums (O and X). The finishing technique (fine or coarse grind) seems to not have as great of an effect on compressive strength of the mortar as it did on the setting time. However, the finer ground samples again proved to outperform than their coarse ground counterparts. Also, the air cooled J-

Rox 4 performed better than the water quenched adding to the conclusion that air cooling may be the preferable finishing method for forming the clinker.

Table 4.4: Mortar Cube Compressive Strength Results

Sample	Average Compressive Strength (psi)			
	3-day	7-day	28-day	90-day
100 OPC	2689	3070	4057	4705
20 FA	2301	2956	3393	4285
15 JR1 FG	4941	3563	3852	4941
15 JR1 CG	2843	3239	3957	4950
25 JR1 FG	5319	3077	3947	5319
25 JR1 CG	2509	3003	3679	5899
15 JR2 FG	1525	2785	3701	3939
15 JR2 CG	1239	2094	2477	2607
25 JR2 FG	1525	2431	3420	3245
25 JR2 CG	1331	2477	3258	3159
15 JRO FG	1582	2559	3399	3818
25 JRO FG	1004	2059	2404	3361
15 JRX FG	1486	2399	3166	3152
25 JRX FG	1116	2150	2889	3546
15 JRA CG	1193	2294	2988	3225
25 JRA CG	1000	1980	2580	2547
15 JRB CG	1408	2174	2769	2898
25 JRB CG	1078	1953	2231	2687
15 JR3	2340	2784	3189	3267
25 JR3	1984	2197	2850	3403
15 JR4 WQ	2896	3449	4434	4372
25 JR4 WQ	2963	2751	4022	4960
15 JR4 AC	2855	3068	3859	4997
25 JR4 AC	2784	2818	3715	5111

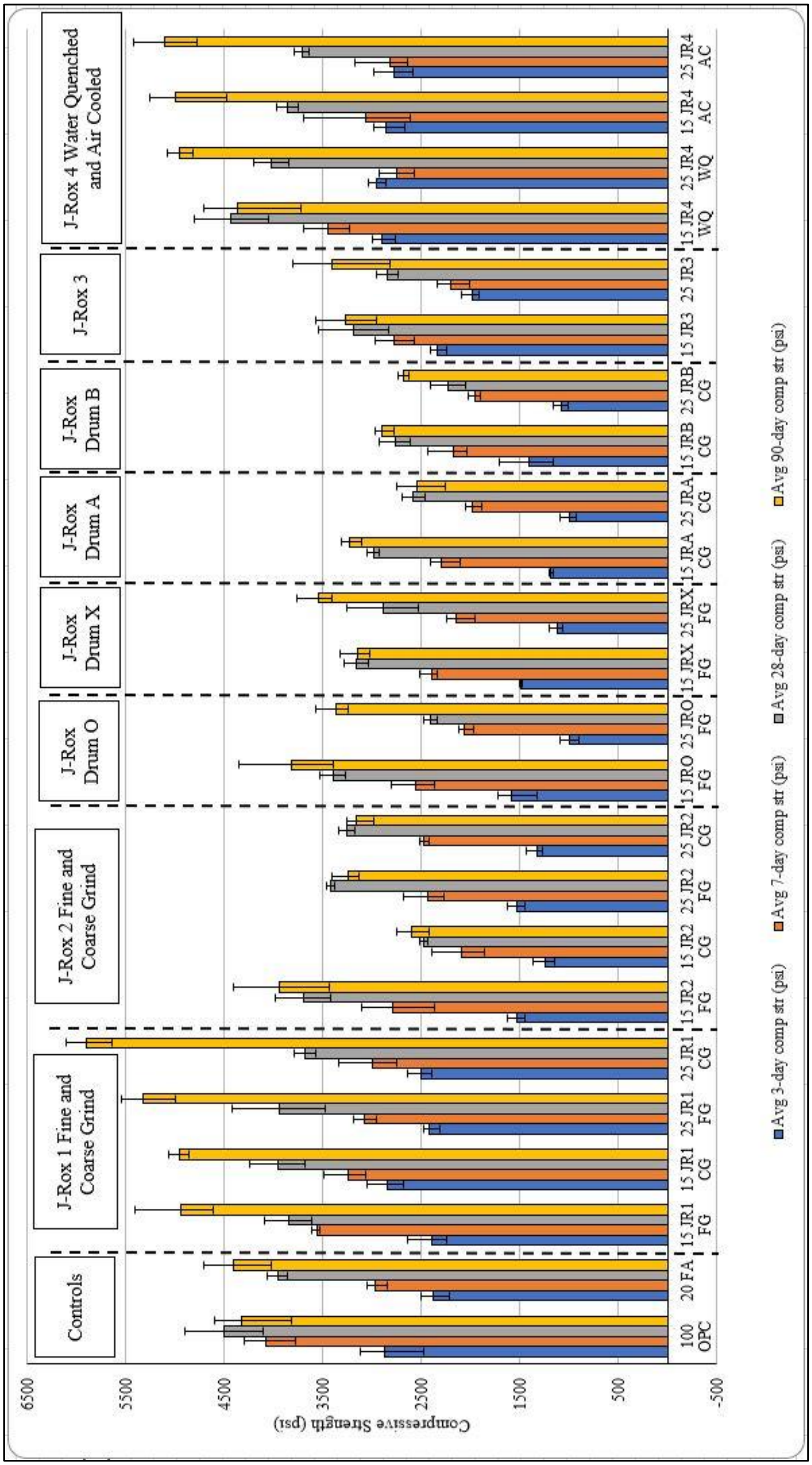

Figure 7: Mortar Cube Compressive Strength Results

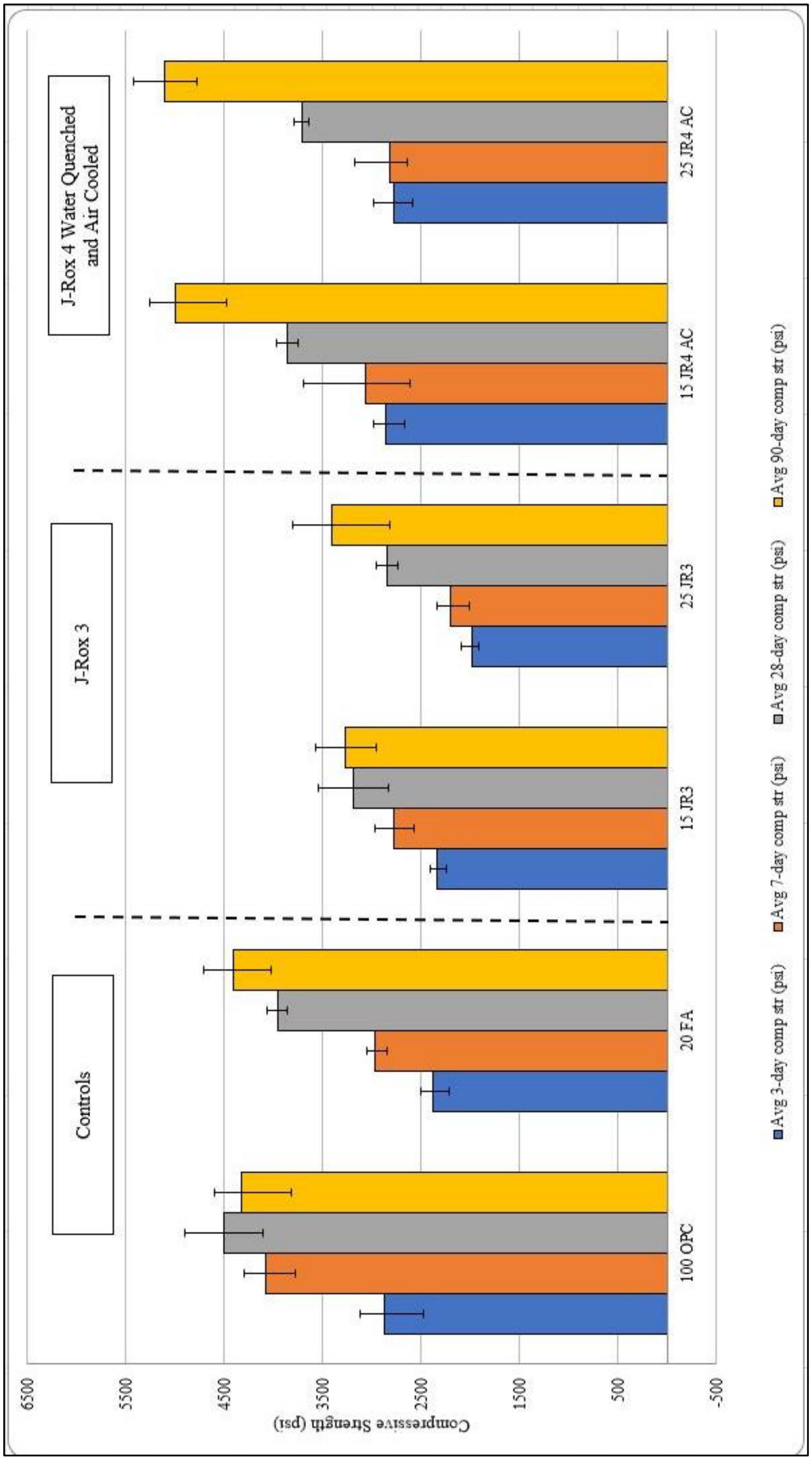

Figure 8: Mortar Compressive Strength of Samples Used to Make Concrete

4.3 Testing of Fresh Concrete Mixture Proportions

The following section presents a discussion of the results for the fresh property tests, as described in Section 3.6, for the concrete mixtures produced. The fresh properties tested were slump, air content, unit weight, and temperature; the results of which are summarized in Table 4.5 below. The minimum amount of cementitious material to be used in each mixture design was 640 lbs with a maximum w/cm ratio of 0.45. A w/cm ratio of 0.41 was used to keep the w/cm relatively low so as not to adversely affect concrete performance. In order to maintain the workability needed to produce test specimens that were properly consolidated and would provide accurate data, approximately 200 mL of a mid-range water reducer was added to each mixture.

There was no target air content for these mixtures, since FDOT does not require a specific air content since their pavement do not typically experience a significant number of freeze/thaw cycles. Therefore, the typical air content for non-air entrained concrete, between 2% and 3%, was the percentage typically obtained when measured. The mid-range water reducer allowed a targeted slump between 2 and 3 inches to be achieved, which although higher than standard for concrete pavement applications, provided the workability needed to adequately consolidate test specimens.

Table 4.5: Fresh Properties of Concrete Mixtures

Mixture	Slump (in)	Air Content	Unit Weight (pcf)	Temperature (°F)
100% Cement Control - granitic gneiss CA	5.25	4.50%	144.1	60
20% Fly Ash Control - granitic gneiss CA	2.5	2.50%	145.9	56
15% J-Rox 3 - granitic gneiss CA	1.5	2.50%	147.4	68
20% J-Rox 3 - granitic gneiss CA	2	2.50%	146.3	62
25% J-Rox 3 - granitic gneiss CA	3.75	2.60%	147.1	62
15% J-Rox 4 - granitic gneiss CA	1.75	2.40%	146.7	61
20% J-Rox 4 - granitic gneiss CA	1.75	2.40%	146.6	62
25% J-Rox 4 - granitic gneiss CA	0.75	2.50%	146.2	61

4.3.1 Slump

The results for slump are shown in Table 4.5. Although the slump values that were achieved were sometimes slightly less than the targeted value, the workability of the concrete was always sufficient for creating concrete samples that were properly consolidated that did not have voids or aggregate separation. The amount of WRA was very similar across all samples

4.3.2 Air Content

The results for air content tests performed on the freshly mixed concrete are shown in Table 4.5. For mixtures produced using granitic gneiss coarse aggregate, all air content measurements were within the specified range of between 2% and 3% for non-air entrained concrete except for the 100% OPC control mixture. Since that sample also had the largest slump, it can be assumed that the higher air content could likely be attributed to the WRA, and slightly less WRA should have been used for that mixture.

4.3.3 Unit Weight

The unit weight of each concrete sample is shown in Table 4.5. For samples made with the granitic gneiss coarse aggregate, unit weight values ranged from 144.1 pcf to 147.4 pcf. The lowest unit weight out of this range is also the sample with the highest air content. There is no real noticeable difference between the unit weights of the controls versus the unit weights of the samples made using J-Rox. The unit weight of the two samples made with the coastal limestone aggregate had unit weights of 137.4 pcf and 137.9 pcf which is due to the lower dry rodded unit weight value of the coastal aggregate compared to the granitic gneiss and the larger air content value.

4.4 Testing of Hardened Concrete

The following section provides the results from the tests performed on hardened concrete specimens which includes testing for the mechanical and durability properties. The mechanical properties will be discussed first which include compressive strength, shrinkage, MOE, and Poisson’s ratio. The durability property testing consisted of surface resistivity.

4.4.1 Mechanical Properties

Table 4.6 shows the mechanical properties of the concrete samples including compressive strength, MOE, and Poisson’s ratio. The following sections provide additional details about the results of each property.

Table 4.6: Summary of Mechanical Properties

Mixture	Compressive Strength					MOE (psi)	Poisson's Ratio	Volumetric Shrinkage 28 Day Avg Microstrain
	3 Day	7 Day	28 Day	56 Day	90 Day			
100% Cement Control - granitic gneiss CA	5202	6381	7610	8066	8378	2,836,104	0.17	497
20% Fly Ash Control - granitic gneiss CA	4425	5387	6598	7496	8280	2,956,853	0.16	523
15% J-Rox 3 - granitic gneiss CA	5000	6345	8757	8264	8441	2,811,152	0.17	513
20% J-Rox 3 - granitic gneiss CA	4979	6038	7170	7863	8116	2,947,193	0.18	520
25% J-Rox 3 - granitic gneiss CA	4334	5510	7596	7934	8135	2,931,091	0.15	537
15% J-Rox 4 - granitic gneiss CA	4618	6242	7064	7934	7546	2,836,104	0.17	587
20% J-Rox 4 - granitic gneiss CA	4237	5763	6506	7553	7684	2,956,853	0.16	687
25% J-Rox 4 - granitic gneiss CA	3506	4803	6047	6653	7335	2,223,305	0.18	433

4.4.1.1 Compressive Strength

Three cylinders were used to test compressive strength at 3, 7, 28, 56, and 90 days of age. The averages of those three values for each mixture are presented in Table 4.6 above. Concrete made with both types of J-Rox showed similar compressive strengths to the granitic gneiss control mixtures at all test ages. An average compressive strength of

5500 psi at 28 days was the target value for compressive strength which was exceeded by all samples tested.

Concrete samples made with J-Rox 3 and J-Rox 4 exhibited greater compressive strength performances at replacement rates of 15% and generally had their lowest compressive strengths at the 25% replacement rate. Comparing the mixture that included a 20% replacement of fly ash with the 20% replacement of J-Rox, J-Rox specimens had higher compressive strengths at all test ages except for J-Rox 3 at 90 days. The following figure shows the compressive strengths of each sample graphically.

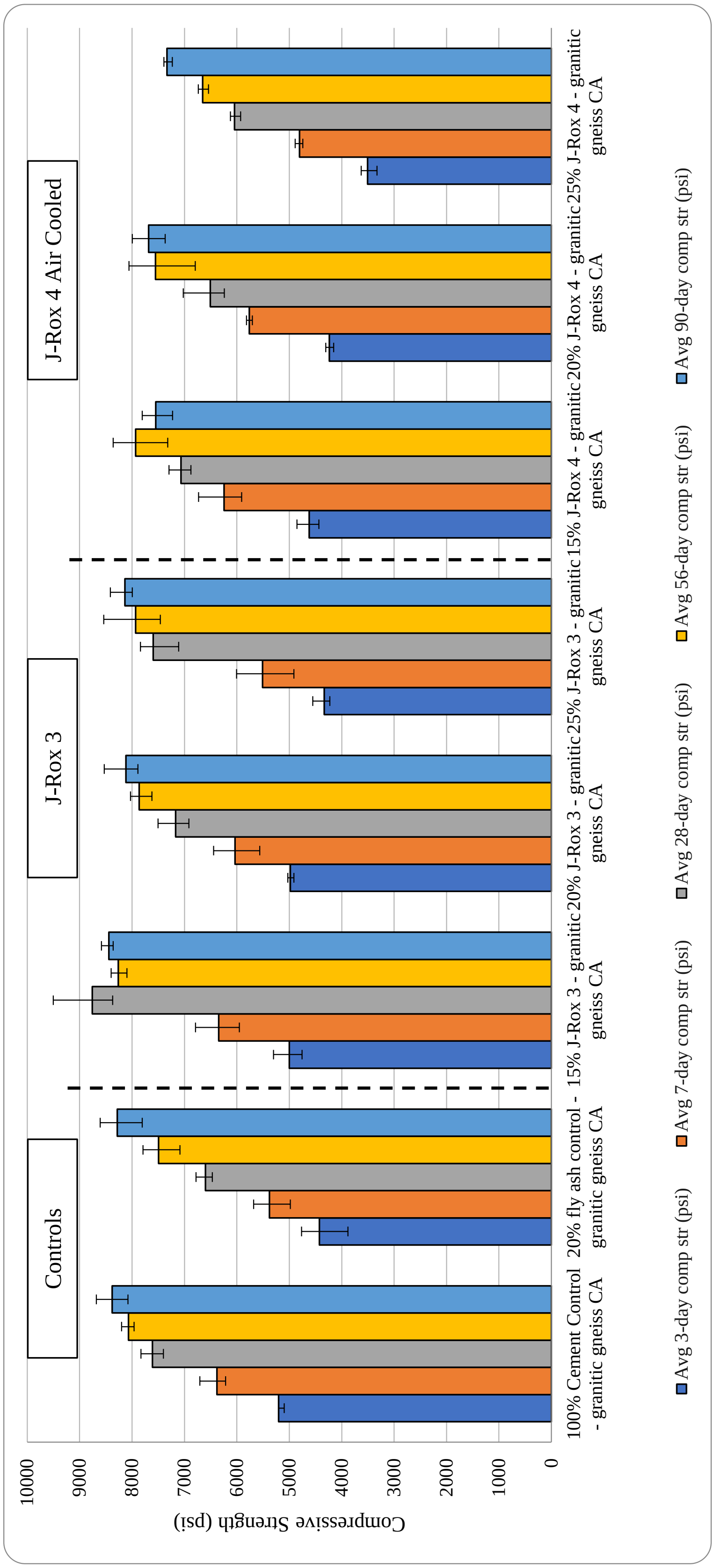

Figure 9: Concrete Compressive Strength Results

4.4.1.2 Modulus of Elasticity and Poisson's Ratio

The values for MOE and Poisson's ratio are shown in Table 4.6.

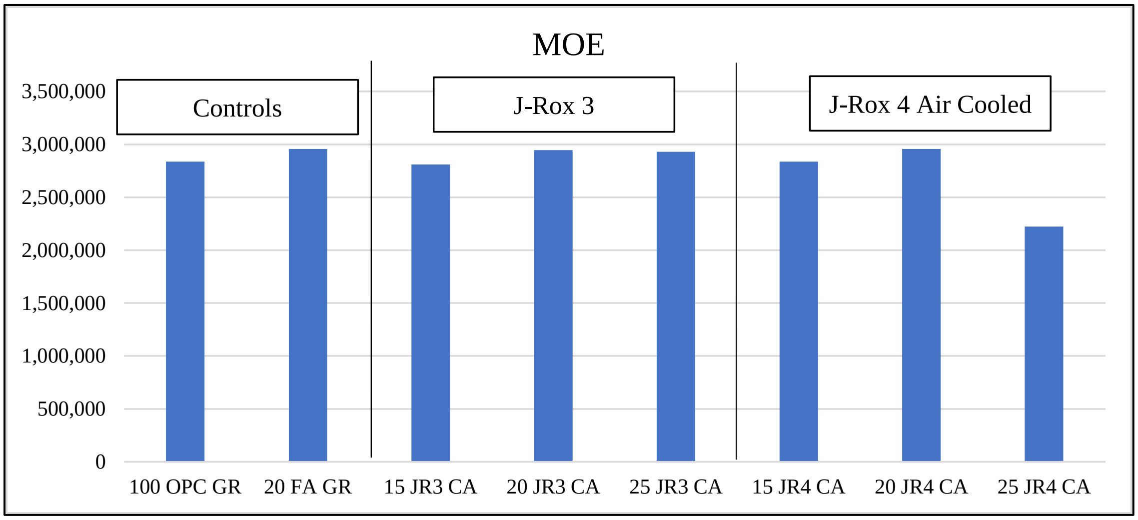

Figure 10: Modulus of Elasticity (MOE) Data

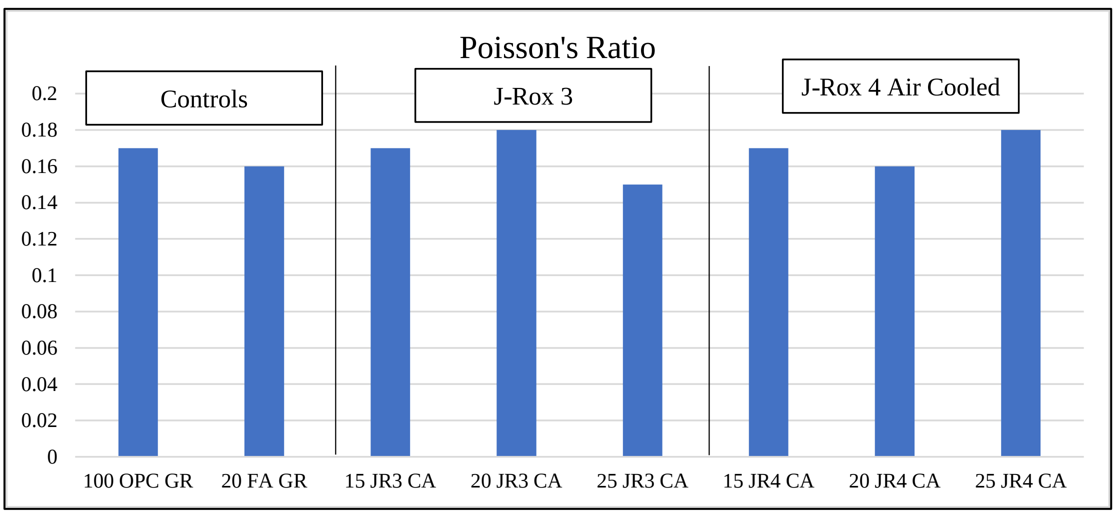

Figure 11: Poisson's Ratio Data

4.4.1.3 Shrinkage

The results for shrinkage testing are shown in Table 4.7. As specified in ASTM C157, shrinkage measurements were performed thirty minutes after demolding (test date 1), and then the specimens were placed in a water bath. The specimens were then removed from the water bath after 28 days and measured again. The beams were then air cured in an environmental chamber and were measured at 4, 7, 14, 28, and 56 days. When in the water bath the beams swell slightly resulting in a positive length change, but then experience negative length change for the remainder of testing due to being dried out in the environmental chamber.

The J-Rox 3 samples experienced the greatest positive length change during the water curing period compared to the other samples. The control samples and J-Rox 4 samples experienced very similar positive growth during the water curing. Negative length changes that occurred during the air curing process were very similar between all samples except for one beam from the 25% J-Rox 4 sample set which can be assumed to be an outlier.

Table 4.7: Shrinkage Microstrain at 28 Days

Sample		28 Day Change	Microstrain	Average
OPCGR Change	Beam 1	-0.000410	-410	440
	Beam 2	-0.000450	-450	
	Beam 3	-0.000460	-460	
FAGR Change	Beam 1	-0.000400	-400	415
	Beam 2	-0.000430	-430	
	Beam 3	-0.000420	-420	
15% J-Rox 3 Change	Beam 1	-0.000400	-400	417
	Beam 2	-0.000440	-440	
	Beam 3	-0.000410	-410	
20% J-Rox 3 Change	Beam 1	-0.000430	-430	423
	Beam 2	-0.000420	-420	
	Beam 3	-0.000420	-420	
25% J-Rox 3 Change	Beam 1	-0.000450	-450	396
	Beam 2	-0.000350	-350	
	Beam 3	-0.000390	-390	
15% J-Rox 4 Change	Beam 1	-0.000480	-480	463
	Beam 2	-0.000490	-490	
	Beam 3	-0.000420	-420	
20% J-Rox 4 Change	Beam 1	-0.000520	-520	510
	Beam 2	-0.000540	-540	
	Beam 3	-0.000470	-470	
25% J-Rox 4 Change	Beam 1	-0.000430	-430	450
	Beam 2	-0.000480	-480	
	Beam 3	-0.000440	-440	

The sample that showed the greatest shrinkage at 28 days was the 20% J-Rox 4, while the J-Rox 3 samples showed very similar results to the fly ash control. The microstrain for the 100% cement control mixture and all J-Rox 4 mixtures was greater than the 420 microstrain recommended as a limit for pavement concrete in AASHTO PP 84. A lower w/cm for the J-Rox 4 mixtures may have helped these mixtures achieve this target.

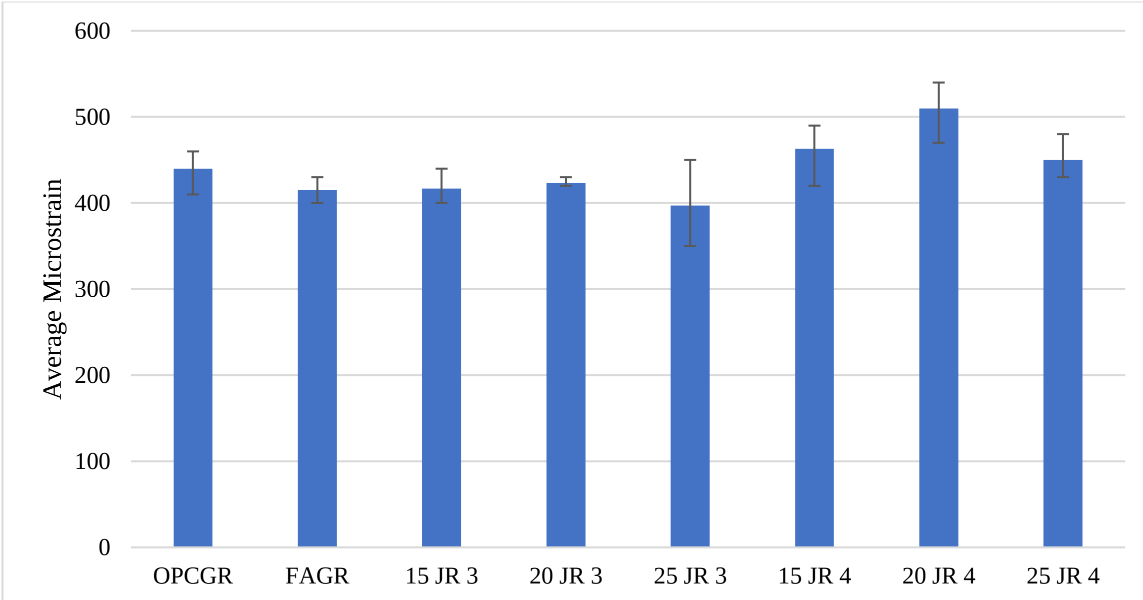

Figure 12: Average Microstrain at 28 Days

4.4.2 Durability Properties

Table 4.8: Surface Resistivity Results

Mixture	Average Resistivity (kΩ•cm)				
	3-day	7-day	28-day	56-day	90-day
100% Cement Control - granitic gneiss CA	4.63	5.40	7.82	8.99	9.53
20% Fly Ash Control - granitic gneiss CA	3.79	4.69	6.83	10.57	13.83
15% J-Rox 3 - granitic gneiss CA	3.87	4.96	6.69	8.63	9.30
20% J-Rox 3 - granitic gneiss CA	3.33	4.12	5.65	7.16	8.50
25% J-Rox 3 - granitic gneiss CA	3.40	4.39	5.72	7.75	8.90
15% J-Rox 4 -granitic gneiss CA	3.74	4.78	6.61	8.85	10.17
20% J-Rox 4 - granitic gneiss CA	3.47	4.55	6.78	9.64	12.70
25% J-Rox 4 - granitic gneiss CA	3.31	4.39	7.23	11.48	16.41

The only J-Rox mixtures that proved to have a similar resistivity to the fly ash control mixture were the J-Rox 4 mixtures, whereas the J-Rox 3 mixtures had very similar results to the cement control. Fly ash shows a large gain in resistivity during the 90-day testing period. The 20% and 25% J-Rox 4 samples showed similar late-age resistivity gain. This shows that the J-Rox 4 samples could be considered to offer similar benefits of reduced concrete permeability (and associated durability benefits) as fly ash due to an enhanced microstructure from the better bonds created from the interaction with the J-Rox particles and cement.

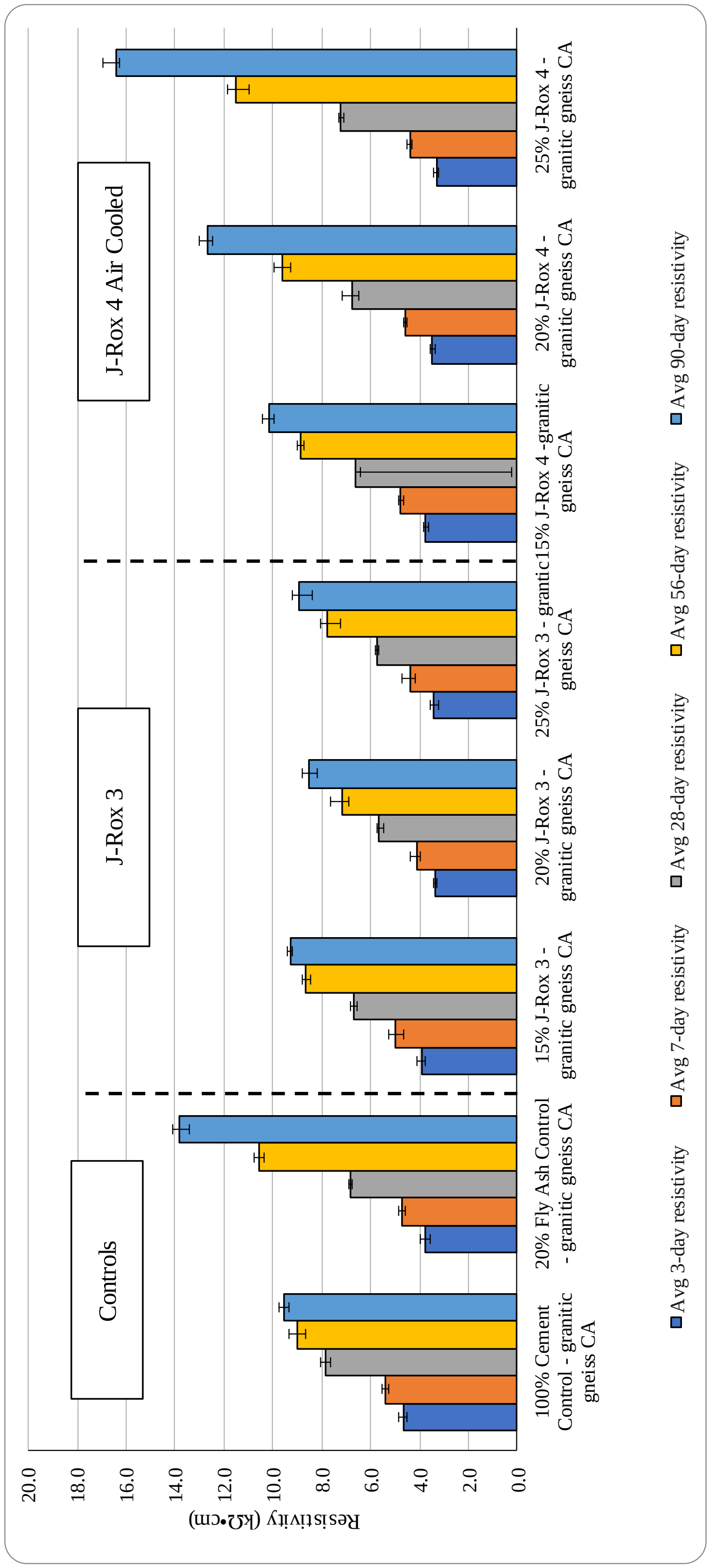

Figure 13: Surface Resistivity Results

4.3 Summary of Results

The original material provided, J-Rox 1, showed promising results during early testing. J-Rox 2 was then produced and provided for testing. J-Rox2 was a lower silica material and did not perform as well as the higher silica J-Rox 1; therefore, J-Rox 3 was produced in a manner that caused it to contain a higher silica content, which allowed it to perform more similarly to the first sample, J-Rox 1. J-Rox 4 was produced prior to the switch from gas power to electric power. The CO_2 was believed to be raising the amount of P_2O_5 to approximately 10% which reduces the amount of SiO_2 and CaO (Nurse, 2007). After paste and mortar testing, J-Rox 4 was determined to perform similarly to J-Rox 1 and J-Rox 3, and it was therefore used to make concrete samples.

Both J-Rox 3 and J-Rox 4 concrete samples showed similar performance to the 100% OPC and 20% fly ash control samples in compressive strength, MOE, Poisson’s ratio, and shrinkage. As previously stated, set time and mortar compressive strengths are also similar. The only notable difference in results between the control samples and the J-Rox 4 concrete are the surface resistivity values, although the mixtures with a higher replacement rate of J-Rox 4 (20% and 25%) exhibited 90-day resistivity values that were close to or exceeded those of the fly ash control mixture.

CHAPTER 5 : DISCUSSION OF RESULTS

5.1: Introduction

This chapter provides a summary of how the results of the paste, mortar, and concrete testing of the various J-Rox samples compare to those of the controls; particularly the comparison to fly ash replacement control due to the future potential use of using J-Rox over fly ash in concrete paving applications. Fly ash provides a variety of benefits in concrete including late-age strength gain, increased workability with longer initial set time, and greater durability. The test methods performed during this research study were selected so as to best gather data that would allow for well-rounded comparative analysis between the effects of using J-Rox over fly ash when producing concrete.

This chapter will go on to discuss in greater depth the comparison between the creation of the J-Rox samples, the comparisons between their chemical structure, and which of the samples was over all deemed most effective. This sample will then be compared in depth to the control samples to clarify how similarly J-Rox is able to perform to fly ash.

5.2: Analysis of Results

5.2.1 Comparison Among J-Rox Samples

The first data analysis that was necessary in this study was to determine the best method for producing J-Rox samples to achieve the necessary chemical composition and particle distribution that would provide the most effective material and also align with the production of the other materials produced at Novaphos. The production methods of Novaphos used to involve both kiln and furnace use, however their decision to produce material solely with the furnace was the first change in direction of how the material was produced. The addition of chemical compounds into the production process, such as silica also determined the performance of the J-Rox sample and how its chemical make-up was able to resemble that of SCMs that are in use. Grinding of the material and cooling processes were the final two items in production that affected the performance of the material, with finer ground samples performing better than coarse, and air cooled performing better than water quenched.

The analysis of these processes was necessary during the overall time period of testing J-Rox samples so that each batch of material produced was developed to perform better than the last and more like the target of having the same results as other SCMs. One of the main qualities of the J-Rox samples that affected the results was the chemical composition which relied heavily on the production method. For example, the higher content silica samples produced better results than the low silica samples which was something that could be addressed by adjusting the feedstock material and certain production processes. This was also the case with the grinding process since the fine ground samples were continuously performing better than the coarse ground samples. Those conclusions were used to create the J-Rox 3 sample which results were used to determine whether the air cooled, or water quenched method would perform better with the J-Rox 4 sample. In the end the J-Rox 3 and J-Rox 4 air cooled proved to be the most promising samples out of all J-Rox materials produced for use in concrete applications.

5.2.2: Comparison of Controls to J-Rox

The differences in chemical composition between the J-Rox samples that presented the set time and mortar compressive strengths most similar to the fly ash control are shown in the following Table.

Table 5.1: Chemical Composition Comparison Between Fly Ash and J-Rox Samples

Percent Weight (%)				
Parameter	**Cement**	**Fly Ash**	**J-Rox 3**	**J-Rox 4 AC**
F	0	0	1	1
SiO2	20.1	52.7	63.4	58.9
P_2O_5	0	0.21	2.5	11.4
CaO	63.6	2.1	24	20.6
MgO	1.4	1.1	2.1	1.7
Al_2O_3	4.8	26.7	0.9	0.8
Fe_2O_3	3.5	11.12	1.5	1.7
Na	0	0.34	0.3	0.3
K	0	2.24	0.1	0.1
SO_4	0	0	1.7	0.7
C	0	0	1.1	1.6
S	0	0	0.7	0.3

The table above compares the control materials that were used for this project. It is noted that these control materials are specific to this project due to the location in which this project was performed, and is why a comparison of a general sample to these materials was provided in Chapter 3. Amounts of Fe_2O_3, and Al_2O_3 are higher in in fly ash than they are in the samples of J-Rox whereas SiO_2, CaO, and P_2O_5 are higher in both J-Rox types. Despite these differences in chemical make-up, the performance of the mixtures produced using J-Rox 3 and J-Rox 4 was either better than or similar to the results of the control samples. After the J-Rox 4 sample was produced, the high amount of P_2O_5 was a concern for how the material would perform especially in compressive strength at 28 days. If this was the case, the production of J-Rox would be changed to a method that produced less CO_2 in an attempt to reduce the level of P_2O_5. As the results show, this was not the result and the samples made with J-Rox 4 actually had only slightly lower compressive strengths which still averaged higher than the 100% OPC control. The 25% J-Rox 4 also had a longer initial set time than the fly ash control allowing for a longer period of workability but had a similar final set time to both control samples.

The greater amount of CaO may also appear to be a potential problem in the use of J-Rox as a SCM due to the fact that greater amounts of CaO cause longer hydration times and can cause the oxygen ions to not form as densely as they normally would be due to the Ca^{2+} ion being so large (Mehta & Monteiro, 2014). It is likely that the addition silica during the production process is able to solve some of these issues due to the silica being able to absorb the extra CaO.

5.3: Comparison to FDOT Specifications

The following Table is published in FDOT's Standard Specifications for Road and Bridge Construction. The concrete produced in this study was to meet the 28 day compressive strength, w/cm ratio, and slump of a Class IV which was achieved in the OPC control and 25% J-Rox 3 mixes, whereas the rest all lacked in reaching a 3 inch slump. However, targeted slump is a value that can be reached through the addition of admixtures causing it to be easily adjusted in future testing to meet the requirement.

Table 5.2: FDOT Concrete Requirements by Class (FDOT, 2022)

Table 346-3 Master Proportion Table			
Class of Concrete	28-day Specified Minimum Compressive Strength (f'c) (psi)	Maximum Water to Cementitious Materials Ratio (pounds per pounds)	Target Slump Value (inches)
I (1)	3,000	0.53	3 (2)
I (Pavement)	3,000	0.50	1.5 or 3 (3)
II (1)	3,400	0.53	3 (2)
II (Bridge Deck)	4,500	0.44	3 (2)
III (4)	5,000	0.44	3 (2)
III (Seal)	3,000	0.53	8
IV	5,500	0.41(4)	3 (2)
IV (Drilled Shaft)	4,000	0.41	8.5
V (Special)	6,000	0.37(4)	3 (2)
V	6,500	0.37(4)	3 (2
VI	8,500	0.37(4)	3 (2)
VII	10,000	0.37(4)	3 (2)

lotes:
1) For precast three-sided culverts, box culverts, endwalls, inlets, manholes and junction boxes, the target slump value and air ontent will not apply. The maximum allowable slump is 6 inches, except as noted in (2). The Contractor is permitted to use oncrete meeting the requirements of ASTM C478 (4,000 psi) in lieu of the specified Class I or Class II concrete for precast ndwalls, inlets, manholes and junction boxes.
2) Increased slump and slip form concrete as defined in 346-3.1.
3) Meet the requirements of Section 350.
4) When silica fume or metakaolin is required, the maximum water to cementitious material ratio will be 0.35. When ultrafine ly ash is used, the maximum water to cementitious material ratio will be 0.30.

The controls and J-Rox concrete samples met the necessary 28 day minimum compressive strength much sooner in the testing period than 28 days. At the three day compressive strength test only one sample, 25% J-Rox 4, did not exceed 4,000 psi, which meets the requirement for concrete up to Class II concrete and bridge decks. The 15% and 20% J-Rox 3 and 15% J-Rox 4 samples all exceeded 6,000 psi at 7 days, but all samples exceeded 6,000 psi by 28 days meaning that it is acceptable for use up to Class V (Special). The lowest compressive strength at the 28 day mark was 6,047 psi (25% J-Rox 4) and the greatest was 8,757 psi (15% J-Rox 3). A simple approach to meet these strength requirements would be to lower the w/cm of mixutures, using additional water reducing admixture to assist with obtaining the desired workability.

5.4 Influence of P_2O_5 on Performance

The following scatter plots show the relationship between the amount of P_2O_5 (percent weight of P_2O_5) in the mixture and compressive strength at 28 and 90 days, resistivity at 28 and 90 days, and modulus of elasticity at 28 days. From these plots, a clear trend between the percent weight of P_2O_5 in the mixtures and the concrete performance variable being compared cannot be seen. While the compressive strength at 28 days shows a trend that the greater the amount of P_2O_5 does cause the overall strength to be somewhat lower, this could be due to the fact that the J-Rox mixtures (with higher P_2O_5 contents) exhibited later age strength gain due to the delayed reactivity of the material. This can be noted in the fact that the slope relating the P_2O_5 content and the 90 day compressive strength is lower.

As can be seen in the plots of resistivity vs. P_2O_5 content and modulus of elasticity vs. P_2O_5 content, a correlation between the percent P_2O_5 by weight and these variables also exists. However, this relationship is likely also related to the later-age reactivity of the J-Rox, and is not necessarily due to the presence of P_2O_5 specifically. resistivity, and test date does not appear to exist. The 25% J-Rox 4 sample is the only value that shows a large variation between MOE and P_2O_5 percent weight which gives the possibility that it is an outlier in this analysis.

No notable issues with concrete performance due to the higher P_2O_5 content were observed for the mixtures and tests performed as part of this study.

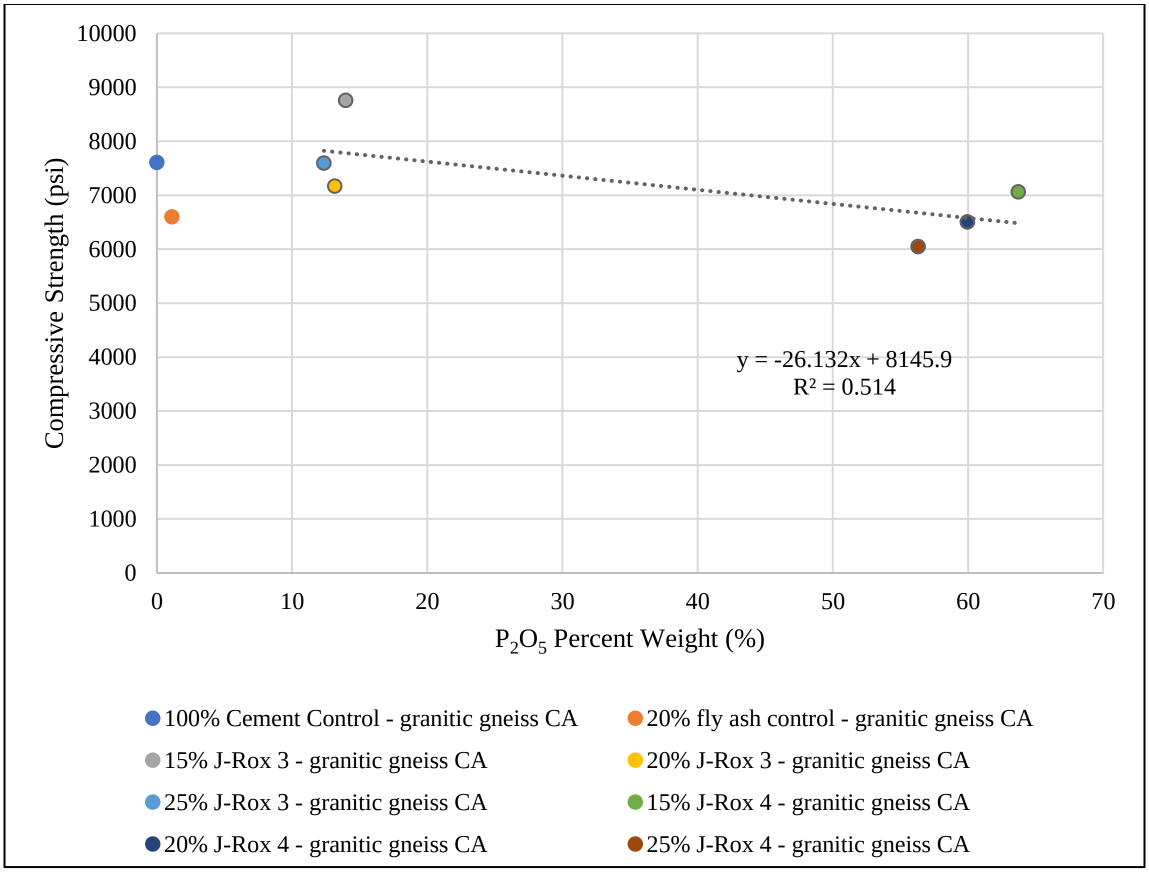

Figure 14: P2O5 Percent Weight vs. 28 Day Compressive Strength

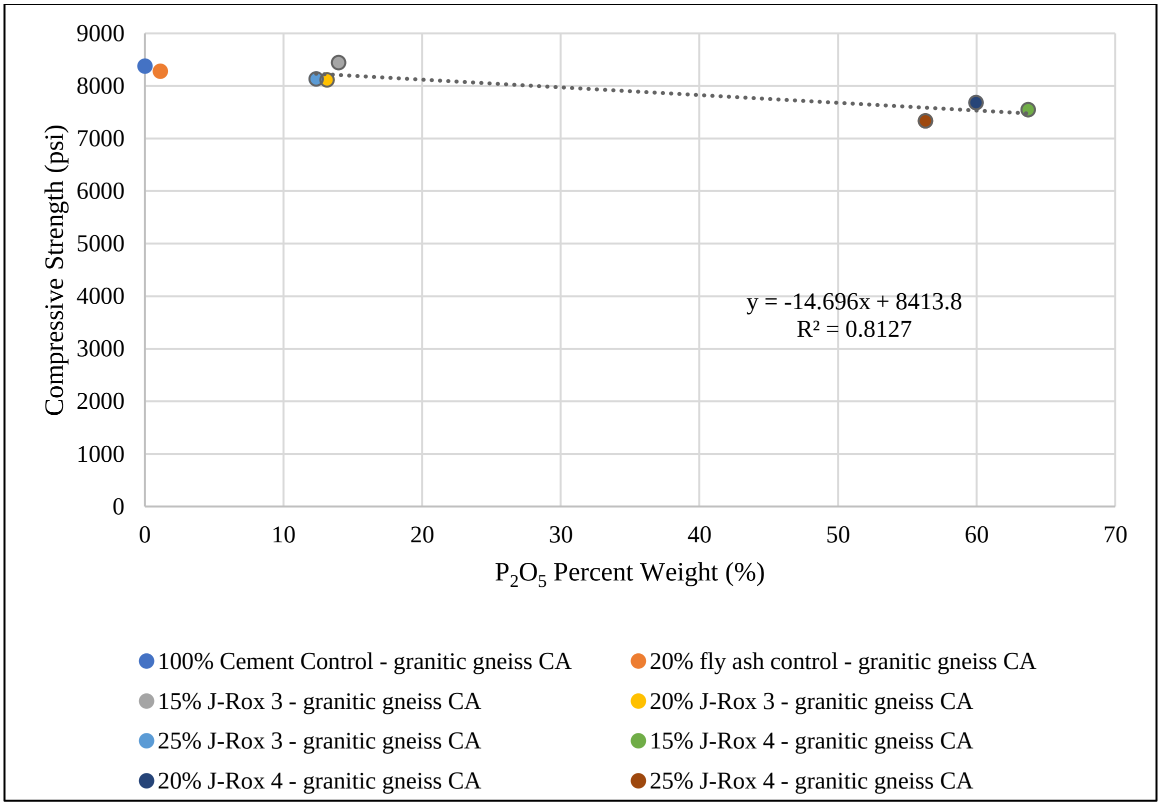

Figure 15: P2O5 Percent Weight vs. 90 Day Compressive Strength

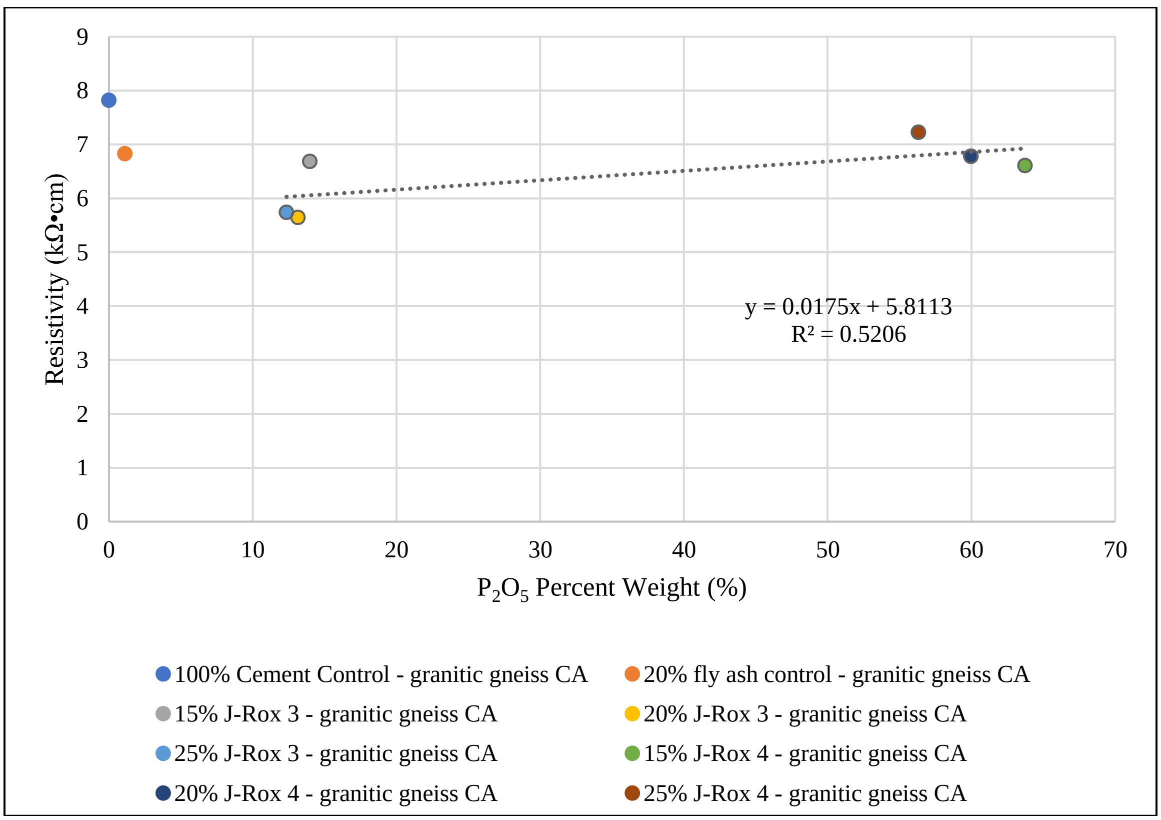

Figure 16: P2O5 Percent Weight vs. 28 Day Resistivity

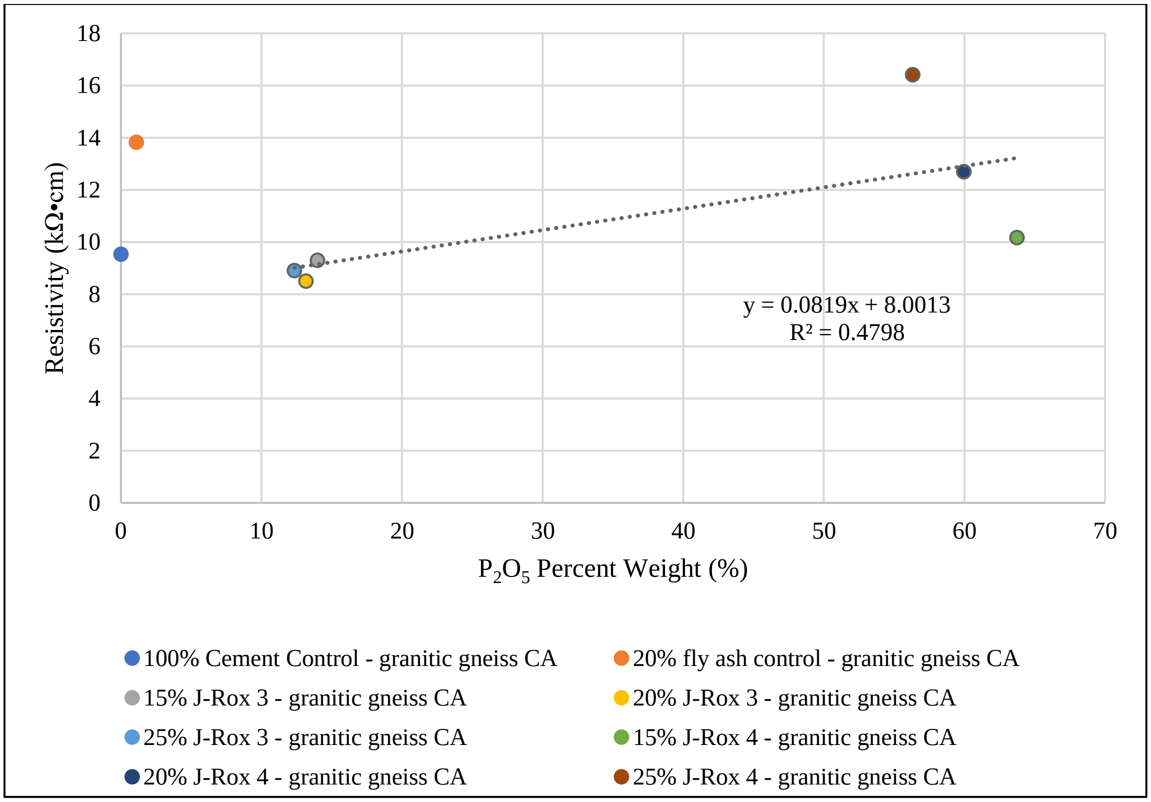

Figure 17: P2O5 Percent Weight vs. 90 Day Resistivity

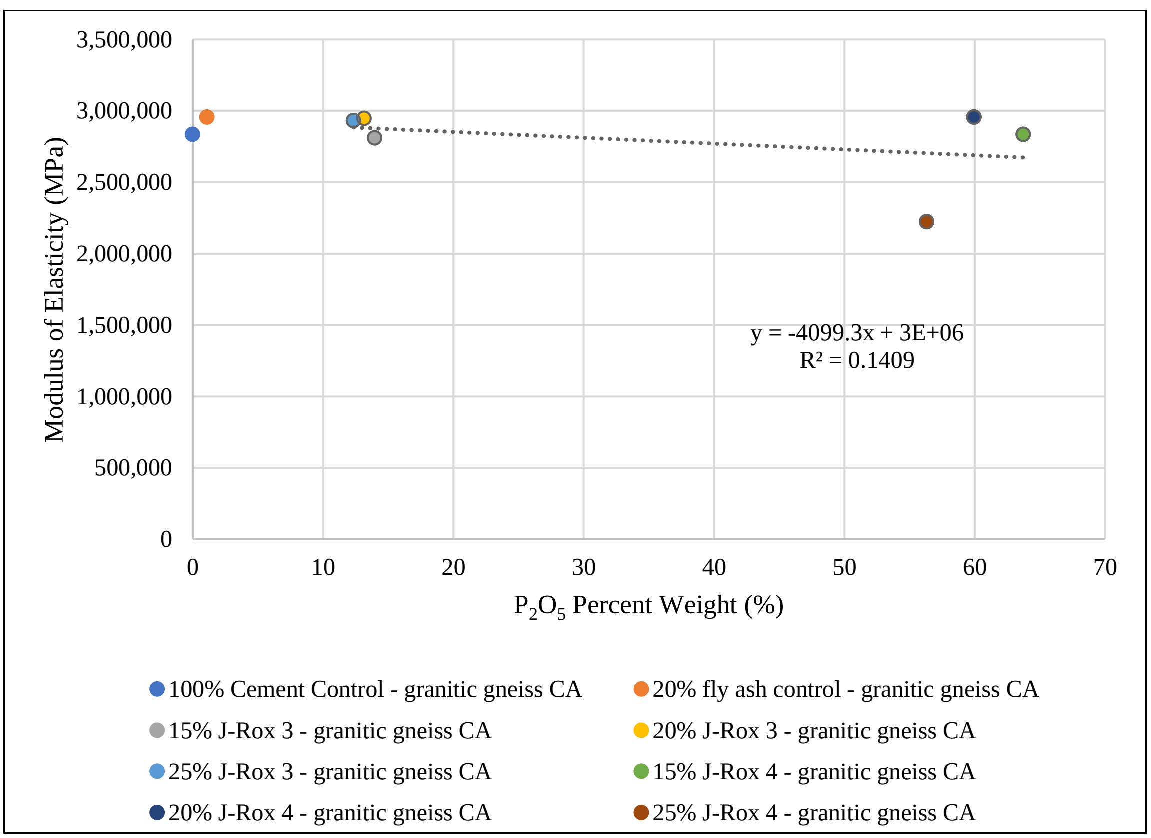

Figure 18: P2O5 Percent Weight vs. MOE

CHAPTER 6 : CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORK

This thesis presents the results found from laboratory testing of a by-product of producing phosphoric acid, J-Rox, which is targeted to be used as a replacement for portland cement when producing concrete, similar to other supplementary cementitious materials such as fly ash. An analysis of the test results provided insight into the most effective production methods that resulted in J-Rox composition and particle characteristics that allow it to achieve concrete and mortar properties similar to those of mixtures containing fly ash. The purpose of this chapter is to summarize the conclusions gathered from the analysis of the test results, as well as provide recommendations for future and ongoing projects related to the use of J-Rox as a SCM in concrete applications.

6.1: Conclusions

Laboratory testing of the paste, mortar, and concrete samples, along with the analysis of production methods to provide feedback to the producers to produce the most beneficial product provided valuable information regarding the most favorable composition of J-Rox to use in concrete applications to achieve results similar to those when a SCM is used in conjunction with ordinary portland cement in paste, mortar, and concrete samples. This study is limited to the use of only one version of a Type I/II OPC and Class F fly ash, and it is understood that the chemistry of these materials varies by supplier, type of source materials, production process, and time. Therefore, it is possible that the results found in this study may differ from those performed with a similar mixtures that use different cements and fly ashes.

Overall, J-Rox showed promise for use as a supplementary cementitious material in concrete applications. The SiO_2 content of the J-Rox appears to have the greatest effect on the results of mechanical property and durability performance testing. The presence of P_2O_5 does not seem to significantly affect the mechanical properties or durability performance of the concrete mixtures at the levels present in the J-Rox provided for this study. Specific, key findings from the laboratory testing are as follows:

J-Rox Characteristics

- The early versions of J-Rox had a chemical makeup that is similar to a Class F fly ash.
- Later versions, particularly J-Rox 4 had a chemical makeup similar to that of a Class C fly ash due to its higher level of calcium.

Fresh properties

- The higher the percentage of J-Rox in the concrete sample, the greater the amount of water or water reducer needed to reach the necessary slump. This is consistent with J-Rox being a ground material, which is likely more angular than fly ash (which consists of spherical particles that improve workability).
- Initial set time is affected by the fineness of the J-Rox particles, with the finer ground samples having a later initial set time than coarse ground.
- Final set time of samples with higher silica contents are closer to those of the control samples.
- P_2O_5 content of the J-Rox did not appear to affect fresh properties of mortar or concrete.

Mechanical properties and durability performance

- Overall, the J-Rox samples that were used to produce concrete in this research study had results for compressive strength, MOE, and initial and final set time that were very similar to the fly ash controls. However, it is understood that as a coproduct, the J-Rox composition will be controlled by the process required to produce phosphoric acid, and the composition of J-Rox produced in the future should be monitored or controlled in order to continue to achieve similar performance.

- J-Rox 4 outperformed the controls and J-Rox 3 samples in mortar compressive strength testing, but the opposite trend was observed during concrete testing. This is likely caused by variability in the water content used in the mortar testing as it was changed slightly during batching to meet the flow tests, but held constant during concrete production and testing to maintain w/cm. It also could have occurred due to variability in environmental conditions when the samples were made.
- J-Rox 3 concrete mixtures showed relatively low resistivity throughout the testing period. However, J-Rox 4 concrete mixtures showed improved later-age resistivity, similar to concrete mixtures containing fly ash.
- Overall, MOE results were very similar to those of the control samples with the exception being the 25% J-Rox 4 mixture, which may have been an outlier. Poisson's ratio results for all J-Rox varied minimally, with all mixtures within +/- 0.02 of the cement or fly ash control mixtures.
- Shrinkage values for J-Rox 3 samples had similar values to that of the fly ash control mixture, which were close to the target of 420 $\mu\varepsilon$ recommended by AASHTO PP 84, whereas J-Rox 4 mixtures exhibited higher shrinkage. The cause for this is not readily evident, but it is understood that the shrinkage of both

the control mixtures and the J-Rox mixtures was likely highly dependent on the w/cm ratio used. Future testing could use a lower w/cm and reduce the shrinkage potential of both the J-Rox and control mixtures.

- The amount of P_2O_5 in J-Rox does not appear to have a significant impact on the mechanical and durability properties of J-Rox concrete samples made with a percent weight of P_2O_5 of up to 11%.

Comparison to FDOT

- The compressive strengths of the J-Rox concrete specimens produced during this project met the 28 day strengths required by FDOT for up to Class V concrete applications.
- The resistivity of concrete mixtures produced during this study was lower than necessary for the durability needed according to the special circumstances implemented by FDOT in January of 2017, based on AASHTO T 358 for use in aggressive environments and for anything above a Class III. However, it is noted that the resistivity of all mixtures (control and J-Rox) could be improved by reducing the w/cm of the mixtures.

6.2: Recommendations for Future Work

The conclusions made from this study of using J-Rox as a replacement for other supplementary cementitious materials in concrete paving applications should continue to be researched through 1) repeating the study once the phosphorous production operations are finalized and the J-Rox product composition and grinding has reached steady-state, and 2) use on a pilot project. The producer could use the data collected in this study to inform changes or improvements to the production process and quality of the material which should then be used for additional laboratory testing.

To ensure durability performance of concrete elements produced with J-Rox, importance should be placed on increasing the resistivity of the concrete mixtures moving forward. Due to the climate and environmental conditions of Florida including heavy exposure to salt water and heavy storms that can easily damage infrastructure it is important to create concrete that is durable against these aggressive conditions. The resistivity of the specimens did not appear to be adversely affected by use of the J-Rox. Therefore, conventional mixture design and proportioning strategies could be used to increase the resistivities of both J-Rox and control mixtures. Use of a lower w/cm ratio in future concrete mixtures should help lower the resistivity of both control and J-Rox mixtures and will potentially allow a better comparison of the J-Rox mixture performance to that of conventional mixtures especially if the future samples have a similar chemical composition to that of the J-Rox 4.

According to the FDOT Standard Specifications for Road and Bridge Construction, Table 6.1 lists the tests that should be completed when testing a SCM to deem if it is adequate to use in practice. All tests except the chloride diffusion were performed as part of this study; therefore, it may be beneficial to add ASTM C1556 (chloride diffusion testing) to a future phase of work.

Table 6.1: FDOT Concrete Testing Requirements for New SCM Use (FDOT, 2022)

Table 929-1 Concrete Testing Requirements		
Test Description	Standard Test Method	Test Age
Surface Resistivity	AASHTO T 358	28 days
Compressive Strength	ASTM C39	28 days
Chloride Diffusion	ASTM C1556 or NT Build 443	6 months, 12 months (1)
Length Change	ASTM C157	28 days(2)
(1) Upon completion of all 28 day and 6-month testing, the SCM producer may present the data to the SMO for acceptance. The12 month data shall be provided to the SMO upon completion. (2) Follow the Air Storage procedure.		

As stated previously, the last version of J-Rox created (J-Rox 4) showed a similar chemical composition to Class C fly ash. When comparing Class C to Class F, typically concrete produced using a Class C fly ash exhibits lower durability performance benefits than concrete produced using a Class F fly ash. That being said, if the future generations of J-Rox continue to have a chemical makeup similar to that of a Class C fly ash due to relatively high levels of calcium it would be recommended to perform tests to evaluate the expansion potential of the concrete and compare how durability is affected.

Future work could also include testing additional concrete mixtures that use other sources and/or types of cement and fly ash with different chemical compositions and particle size characteristics to better understand how J-Rox interacts with different cementitious materials. The use of portland limestone cement would also be an interesting addition to the testing program as future markets turn toward using that material more due to availability and sustainability benefits.

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APPENDIX A: SUPPLEMENTAL INFORMATION FOR CHAPTER 3

Variables			Specific Gravities	
Max Aggregate Size:	0.75	in	Cement	3.15
Water/Air Content (Table 6.3.3):	270	lbs/CY	Fly Ash	2.29
Air (Table 6.3.3):	1%		J-Rox	2.2
Water/Cement Ratio:	0.41		Coarse Agg	2.63
Coarse Agg Content (Table 6.3.6):	0.7		Fine Agg	2.62
Cement Content:	658	lbs/CY	Water	1.00

Water:	269.78

2.5 Cubic Foot Mixtures

100% OPC Mixture:	Batch Weights:		Absolute Volumes:		Batch Quantities	
Cement	658	lbs/CY	3.35	CF	36.56	lbs
Fly Ash	0	lbs/CY	0.00	CF	0.00	lbs
J-Rox	0	lbs/CY	0.00	CF	0.00	lbs
Coarse Aggregate	1814	lbs/CY	11.06	CF	100.80	lbs
Water	269.78	lbs/CY	4.32	CF	14.99	lbs
Air	0	lbs/CY	0.27	CF	0.00	lbs
Fine Aggregate	1308	lbs/CY	8.00	CF	72.69	lbs
Total Weight/CY:	4051	lbs/CY	27.00	CF	225.03	lbs
Batch Unit Weight:	150.02		lbs/CF			

20% Fly Ash Mixture:	Batch Weights:		Absolute Volumes:		Batch Quantities	lbs
Cement	526	lbs/CY	2.68	CF	29.24	lbs
Fly Ash	132	lbs/CY	0.92	CF	7.31	lbs
J-Rox	0	lbs/CY	0.00	CF	0.00	lbs
Coarse Aggregate	1814	lbs/CY	11.06	CF	100.80	lbs
Water	269.78	lbs/CY	4.32	CF	14.99	lbs
Air	0	lbs/CY	0.27	CF	0.00	lbs
Fine Aggregate	1267	lbs/CY	7.75	CF	70.41	lbs
Total Weight/CY:	4009	lbs/CY	27.00	CF	371.25	lbs
Batch Unit Weight:	148.50		lbs/CF			

2.25 Cubic Foot Mixtures

15% J-Rox Mixtures:	**Batch Weights:**		**Absolute Volumes:**		**Batch Quantities**	
Cement	559	lbs/CY	2.85	CF	51.79	lbs
Fly Ash	0	lbs/CY	0.00	CF	0.00	lbs
J-Rox	99	lbs/CY	0.72	CF	9.14	lbs
Coarse Aggregate	1814	lbs/CY	11.06	CF	168.00	lbs
Water	269.78	lbs/CY	4.32	CF	24.98	lbs
Air	0	lbs/CY	0.27	CF	0.00	lbs
Fine Aggregate	1273	lbs/CY	7.79	CF	117.87	lbs
Total Weight/CY:	4015	lbs/CY	27.00	CF	371.77	lbs
Batch Unit Weight:	148.71		lbs/CF			

20% J-Rox Mixtures:	**Batch Weights:**		**Absolute Volumes:**		**Batch Quantities**	
Cement	526	lbs/CY	2.68	CF	48.74	lbs
Fly Ash	0	lbs/CY	0.00	CF	0.00	lbs
J-Rox	132	lbs/CY	0.96	CF	12.19	lbs
Coarse Aggregate	1814	lbs/CY	11.06	CF	168.00	lbs
Water	269.78	lbs/CY	4.32	CF	24.98	lbs
Air	0	lbs/CY	0.27	CF	0.00	lbs
Fine Aggregate	1261	lbs/CY	7.71	CF	116.77	lbs
Total Weight/CY:	4003	lbs/CY	27.00	CF	370.68	lbs
Batch Unit Weight:	148.27		lbs/CF			

25% J-Rox Mixtures:	**Batch Weights:**		**Absolute Volumes:**		**Batch Quantities**	
Cement	494	lbs/CY	2.51	CF	45.69	lbs
Fly Ash	0	lbs/CY	0.00	CF	0.00	lbs
J-Rox	165	lbs/CY	1.20	CF	15.23	lbs
Coarse Aggregate	1814	lbs/CY	11.06	CF	168.00	lbs
Water	269.78	lbs/CY	4.32	CF	24.98	lbs
Air	0	lbs/CY	0.27	CF	0.00	lbs
Fine Aggregate	1249	lbs/CY	7.64	CF	115.68	lbs
Total Weight/CY:	3992	lbs/CY	27.00	CF	369.59	lbs
Batch Unit Weight:	147.83		lbs/CF			

APPENDIX B: SUPPLLEMENTAL INFORMATION FOR CHAPTER 4

Holcim NSF Certified to NSF/ANSI 61 & 372

Material: Portland Cement
Type: I-II (MH)

Material Certification Report

Test Period: 18-Sep-2019 to 19-Sep-2019
Date Issued: 14-Oct-2019

Certification

This cement meets the specifications of ASTM C150 and AASHTO M85 for Type I-II (MH) cement.

General Information

Supplier:	Holcim (US) Inc. d/b/a LafargeHolcim US	Source Location:	Holly Hill Plant Silo: 18
Address:	8700 West Bryn Mawr Ave Chicago, IL 60631		2173 Gardner Blvd Holly Hill, SC 29059
Contact:		Contact:	Scott Poaps / (803) 496-2995

The following is based on average test data during the test period. The data is typical of product shipped from this source; individual shipments may vary.

Test Data on ASTM Standard Requirements

Chemical Item	Limit [1]	Result	Physical Item	Limit [1]	Result
SiO_2 (%)	-	20.1	Air Content (%)	12 max	7
Al_2O_3 (%)	6.0 max	4.8	Blaine Fineness (m^2/kg)	260-430	404
Fe_2O_3 (%)	6.0 max	3.5			
CaO (%)	-	63.6	Autoclave Expansion (%) (C151)	0.80 max	-0.04
MgO (%)	6.0 max	1.4	Compressive Strength MPa (psi)		
SO_3 (%) [2]	3.0 max	3.2	3 day	10.0 (1450) min	31.0 (4500)
Loss on Ignition (%) [5]	3.5 max	1.9	7 day	17.0 (2470) min	38.4 (5570)
Insoluble Residue (%)	1.50 max	0.36	28 day (previous month's data)	-	46.9 (6800)
CO_2 (%)	-	1.0			
$CaCO_3$ in Limestone (%)	70 min	88	Initial Vicat (minutes)	45-375	126
Potential Phase Compositions [3]:					
C_3S (%)	-	56	Mortar Bar Expansion (%) (C1038)	0.020 max	0.003
C_2S (%)	-	15			
C_3A (%)	8 max	7			
C_4AF (%)	-	11			
C_3S + 4.75C_3A (%)	100 max	88			

Test Data on ASTM Optional Requirements

Chemical Item	Limit [1]	Result	Physical Item	Limit [1]	Result
Equivalent Alkalies (%)	-	0.48	Heat of Hydration kJ/kg (cal/g) (ASTM C1702) 3 Days [4]	-	267 (64)

Notes (*1-9)

1 - Dashes in the Limit / Result columns mean Not Applicable.
2 - It is permissible to exceed the specification limit provided that ASTM C1038 Mortar Bar Expansion does not exceed 0.020% at 14 days.
3 - Adjusted per Annex A1.6 of ASTM C150 and AASHTO M85.
4 - Test results represent the most recent value and is provided for information only.
5 - Limit = 3.0 when limestone is not an ingredient in the final cement product
9/18/2019
Grind 261

Additional Data

Item	Limestone	Inorganic Processing Addition	Base Cement Phase Composition	Result
Amount (%)	2.6	0.7	C_3S (%)	58
SiO_2 (%)	2.7	30.3	C_2S (%)	15
Al_2O_3 (%)	0.7	11.0	C_3A (%)	7
Fe_2O_3 (%)	0.8	0.7	C_4AF (%)	11
CaO (%)	50.1	41.4		
SO_3 (%)	0.6	2.0		

Printed: 10/14/2019 3:10:27 PM

Version: 180412

Scott Poaps, Quality Manager

Figure 19: Cement Mill Certification

SGS TEC SERVICES
Testing • Engineering • Consulting

June 4, 2020

Ms. Tara Cavalline
UNC Charlotte
9201 University City Blvd.
Charlotte, NC 28223-0001

Phone: 704-687-2305
Fax: 704-687-6653
Email: tcavalline@uncc.edu

Subject: **Report of Results for Product Testing**
Product Name: Roxboro DS 11/23-12/11 (2015)
TEC Services Project #: 20-1612
TEC Laboratory #: 20-461

Dear Ms. Cavalline:

SGS TEC Services is an AASTHO R18, ANS/ISO/IEC 17025:2005 and Army Corp of Engineers accredited laboratory. SGS TEC Services is pleased to present this report of our test results on the submitted material designated as "Roxboro DS 11/23-12/11 (2015)". Our services were performed in accordance with the terms and conditions of our Service Agreement TEC-PRO-20-1612. The test results presented only pertain to the samples tested.

The Roxboro material was delivered to our Lawrenceville, GA facility on April 14, 2020. At the request of UNC Charlotte, testing was performed on the material per ASTM C618-19 *Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete*. The chemical analysis test results are reported in attached Table 1.

SGS TEC Services appreciates the opportunity to provide our professional services for this important project. If you have any questions regarding this report, or if we can be of further assistance please contact us at 770-995-8000.

Sincerely,

SGS TEC SERVICES

Dean T. Roosa
Project Manager

Shawn P. McCormick
Laboratory Principal

ISO 17025 ACCREDITED LABORATORY
US Army Corps of Engineers

SGS TEC SERVICES
235 Buford Drive | Lawrenceville GA 30046
770-995-8000 | www.tecservices.com

AASHTO R18
ASTM INTERNATIONAL

A.1: Fly Ash Chemical Analysis Page 1

Report of Results for Product Testing *June 4, 2020*
Product Name: Roxboro DS 11/23-12/11 (2015)
TEC Services Project #: 20-1612
TEC Laboratory #: 20-461

Table 1 – Results of the Chemical Analysis

Oxides	Results Weight (%)
Silicon Dioxide (SiO_2)	**52.7**
Aluminum Oxide (Al_2O_3)	**26.7**
Iron Oxide (Fe_2O_3)	**11.12**
Sum ($SiO_2 + Al_2O_3 + Fe_2O_3$)	**90.5**
Calcium Oxide (CaO)	**2.1**
Magnesium Oxide (MgO)	**1.1**
Sodium Oxide (Na_2O)	**0.34**
Potassium Oxide (K_2O)	**2.24**
Equivalent Alkalies (Na_2O+0.658 K_2O)	**1.81**
Titanium Dioxide (TiO_2)	**1.42**
Manganic Oxide (Mn_2O_3)	**0.026**
Phosphorus Pentoxide (P_2O_5)	**0.21**
Sulfur Trioxide (SO_3)	**0.75**
Loss on Ignition	**1.9**
Moisture Content	**0.38**

A.2: Fly Ash Chemical Analysis Page 2

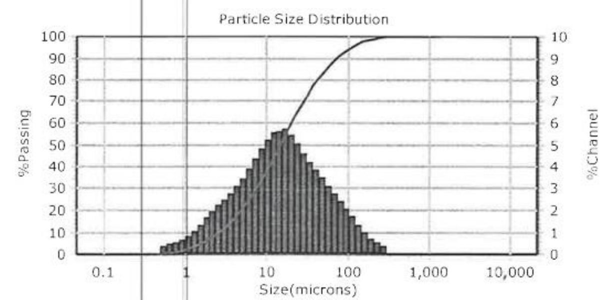
Microtrac Total Solutions in Particle Characterization

- Particle Size Analysis -

C:\Microtrac\FLEX 11.1.0.4\Databases\novaphos2020.MDB

-Tabular Data -

Size(um)	%Chan	%Pass	Size(um)	%Chan	%Pass
1408	0.00	100.00	3.27	2.47	13.35
1184	0.00	100.00	2.750	2.23	10.88
995.6	0.00	100.00	2.312	1.95	8.65
837.2	0.00	100.00	1.945	1.64	6.70
704.0	0.00	100.00	1.635	1.32	5.06
592.0	0.00	100.00	1.375	1.02	3.74
497.8	0.00	100.00	1.156	0.79	2.72
418.6	0.00	100.00	0.972	0.64	1.93
352.0	0.00	100.00	0.818	0.53	1.29
296.0	0.37	100.00	0.688	0.43	0.76
248.9	0.49	99.63	0.578	0.33	0.33
209.3	0.68	99.14	0.486	0.00	0.00
176.0	0.96	98.46	0.409	0.00	0.00
148.0	1.31	97.50	0.344	0.00	0.00
124.5	1.69	96.19	0.2890	0.00	0.00
104.7	2.05	94.50			
88.00	2.39	92.45			
74.00	2.74	90.06			
62.23	3.11	87.32			
52.33	3.48	84.21			
44.00	3.82	80.73			
37.00	4.17	76.91			
31.11	4.56	72.74			
26.16	5.01	68.18			
22.00	5.45	63.17			
18.50	5.73	57.72			
15.56	5.58	51.99			
13.08	5.57	46.41			
11.00	5.22	40.84			
9.25	4.79	35.62			
7.78	4.34	30.83			
6.54	3.89	26.49			
5.50	3.45	22.60			
4.62	3.06	19.15			
3.89	2.74	16.09			



- Measurement Info -

Title
Jroc fine
Identifiers
6-8-20
10:40am
Database Record 88
Run Number Avg Of 3
Date 6/8/2020
Time 10:44 AM
Acquired Date 6/8/2020
Acquired Time 10:44 AM
Serial Number S6309
Sample Cell ID 3151
Calculated Data
Above Residual 0
Below Residual 0
Loading Factor 0.0143
Transmission 0.973
RMS Residual 0.530%
User Defined Calculations
Name Value
Recalculation Status
Live-Meas : : Original :

-SOP Info-

BALL MILL FEED(*)
Timing
Setzero Time 30 (sec)
Run Time 30 (sec)
Number of Runs 3
Multi-Run Delay 0 (min)
Delay First Meas. Disabled
Analysis
JDC Ball Mill Feed
Refractive Index 1.60
Transparency Transp
Shape Irregular
WATER
Refractive Index 1.33
Options:
Filter Enabled
Analysis Gain 0
Analysis Mode S3000/3500
Perspective
Progression Standard
Distribution Volume
Upper Edge(um) 1408
Lower Edge(um) 0.243
Residuals Enabled

Summary	
Data	Value
MV(um):	28.98
MN(um):	0.991
MA(um):	6.45
CS:	9.31E-01
SD:	23.95
Mz:	23.43
σι:	28.36
Ski:	0.655
Kg:	1.584

Percentiles	
%Tile	Size(um)
10.00	2.574
20.00	4.84
30.00	7.54
40.00	10.71
50.00	14.64
60.00	19.86
70.00	27.99
80.00	42.52
90.00	73.71
95.00	109.8

Peaks Summary		
Dia(um)	Vol%	Width
14.64	100	47.9

6/8/2020 10:46 AM

FLEX
11.1.0.4

- Notes -

10 buckets/x

Fine JRox - Drum X

Sample System(Wet)
Number of Rinses 3
Flow Rate (%) 55%
Deaeration Cycles 4

A.3: J-Rox Drum X Particle Size Analysis

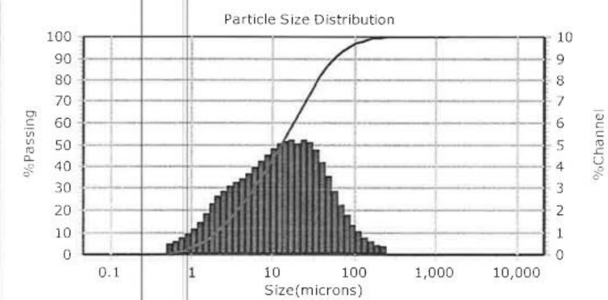
Microtrac
Total Solutions in Particle Characterization

- Particle Size Analysis -

C:\Microtrac\FLEX 11.1.0.4\Databases\novaphos2020.MDB

-Tabular Data -

Size(um)	%Chan	%Pass	Size(um)	%Chan	%Pass
1408	0.00	100.00	3.27	3.07	17.86
1184	0.00	100.00	2.750	2.87	14.79
995.6	0.00	100.00	2.312	2.61	11.92
837.2	0.00	100.00	1.945	2.26	9.31
704.0	0.00	100.00	1.635	1.84	7.05
592.0	0.00	100.00	1.375	1.44	5.21
497.8	0.00	100.00	1.156	1.12	3.77
418.6	0.00	100.00	0.972	0.90	2.65
352.0	0.00	100.00	0.818	0.73	1.75
296.0	0.00	100.00	0.688	0.59	1.02
248.9	0.32	100.00	0.578	0.43	0.43
209.3	0.42	99.68	0.486	0.00	0.00
176.0	0.55	99.26	0.409	0.00	0.00
148.0	0.74	98.71	0.344	0.00	0.00
124.5	1.01	97.97	0.2890	0.00	0.00
104.7	1.33	96.96			
88.00	1.75	95.63			
74.00	2.25	93.88			
62.23	2.85	91.63			
52.33	3.53	88.78			
44.00	4.20	85.25			
37.00	4.73	81.05			
31.11	5.06	76.32			
26.16	5.18	71.26			
22.00	5.02	66.08			
18.50	5.19	61.06			
15.56	5.14	55.87			
13.08	5.02	50.73			
11.00	4.81	45.71			
9.25	4.53	40.90			
7.78	4.22	36.37			
6.54	3.93	32.15			
5.50	3.67	28.22			
4.62	3.44	24.55			
3.89	3.25	21.11			



Summary	
Data	Value
MV(um):	23.50
MN(um):	0.972
MA(um):	5.35
CS:	1.121
SD:	19.37
Mz:	19.13
σi:	21.95
Ski:	0.606
Kg:	1.328

Percentiles	
%Tile	Size(um)
10.00	2.040
20.00	3.67
30.00	5.96
40.00	8.95
50.00	12.76
60.00	17.86
70.00	25.08
80.00	35.55
90.00	56.11
95.00	82.28

Peaks Summary		
Dia(um)	Vol%	Width
12.76	100	38.73

6/8/2020 10:36 AM

FLEX
11.1.0.4

- Measurement Info -

Title	
Jroc fine	
Identifiers	
6-8-20	
10:30am	
Database Record	84
Run Number	Avg Of 3
Date	6/8/2020
Time	10:35 AM
Acquired Date	6/8/2020
Acquired Time	10:35 AM
Serial Number	S6309
Sample Cell ID	3151
Calculated Data	
Above Residual	0
Below Residual	0
Loading Factor	0.0143
Transmission	0.970
RMS Residual	0.573%
User Defined Calculations	
Name	Value
Recalculation Status	
Live-Meas : : Original :	

-SOP Info-

BALL MILL FEED(*)	
Timing	
Setzero Time	30 (sec)
Run Time	30 (sec)
Number of Runs	3
Multi-Run Delay	0 (min)
Delay First Meas.	Disabled
Analysis	
JDC Ball Mill Feed	
Refractive Index	1.60
Transparency	Transp
Shape	Irregular
WATER	
Refractive Index	1.33
Options:	
Filter	Enabled
Analysis Gain	0
Analysis Mode	S3000/3500
Perspective	
Progression	Standard
Distribution	Volume
Upper Edge(um)	1408
Lower Edge(um)	0.243
Residuals	Enabled

- Notes -

10 buckets/0

Fine JRox - Drum O

Sample System	(Wet)
Number of Rinses	3
Flow Rate (%)	55%
Deaeration Cycles	4

A.4: J-Rox Drum O Particle Size Analysis

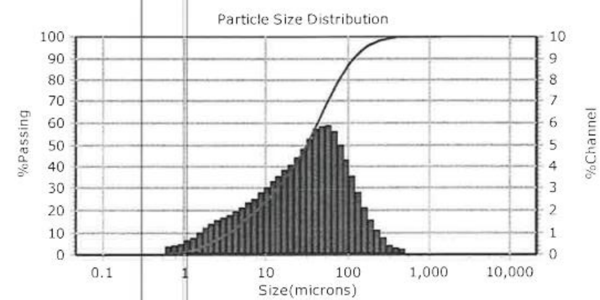
Microtrac — Total Solutions in Particle Characterization

- Particle Size Analysis -

C:\Microtrac\FLEX 11.1.0.4\Databases\novaphos2020.MDB

-Tabular Data -

Size(um)	%Chan	%Pass	Size(um)	%Chan	%Pass
1408	0.00	100.00	3.27	1.67	9.31
1184	0.00	100.00	2.750	1.54	7.64
995.6	0.00	100.00	2.312	1.39	6.10
837.2	0.00	100.00	1.945	1.18	4.71
704.0	0.00	100.00	1.635	0.96	3.53
592.0	0.00	100.00	1.375	0.76	2.57
497.8	0.22	100.00	1.156	0.60	1.81
418.6	0.29	99.78	0.972	0.48	1.21
352.0	0.41	99.49	0.818	0.40	0.73
296.0	0.73	99.08	0.688	0.33	0.33
248.9	1.06	98.35	0.578	0.00	0.00
209.3	1.52	97.29	0.486	0.00	0.00
176.0	2.10	95.77	0.409	0.00	0.00
148.0	2.79	93.67	0.344	0.00	0.00
124.5	3.53	90.88	0.2890	0.00	0.00
104.7	4.29	87.35			
88.00	4.99	83.06			
74.00	5.59	78.07			
62.23	5.90	72.48			
52.33	5.85	66.58			
44.00	5.71	60.73			
37.00	5.26	55.02			
31.11	4.79	49.76			
26.16	4.39	44.97			
22.00	4.07	40.58			
18.50	3.81	36.51			
15.56	3.56	32.70			
13.08	3.30	29.14			
11.00	3.04	25.84			
9.25	2.78	22.80			
7.78	2.54	20.02			
6.54	2.32	17.48			
5.50	2.12	15.16			
4.62	1.94	13.04			
3.89	1.79	11.10			



Summary

Data	Value
MV(um):	50.20
MN(um):	1.120
MA(um):	9.32
CS:	6.44E-01
SD:	42.67
Mz:	42.82
σι:	45.91
Ski:	0.520
Kg:	1.173

Percentiles

%Tile	Size(um)
10.00	3.51
20.00	7.77
30.00	13.66
40.00	21.48
50.00	31.37
60.00	43.06
70.00	57.84
80.00	78.95
90.00	118.8
95.00	164.2

Peaks Summary

Dia(um)	Vol%	Width
31.37	100	85.35

6/8/2020 1:08 PM

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11.1.0.4

- Measurement Info -

Title	
Jroc coarse	
Identifiers	
6-8-20	
1:00pm	
Database Record	100
Run Number	Avg Of 3
Date	6/8/2020
Time	1:05 PM
Acquired Date	6/8/2020
Acquired Time	1:05 PM
Serial Number	S6309
Sample Cell ID	3151
Calculated Data	
Above Residual	0
Below Residual	0
Loading Factor	0.0300
Transmission	0.958
RMS Residual	0.597%
User Defined Calculations	
Name	Value
Recalculation	Status
Live-Meas : : Original :	

-SOP Info-

BALL MILL FEED(*)	
Timing	
Setzero Time	30 (sec)
Run Time	30 (sec)
Number of Runs	3
Multi-Run Delay	0 (min)
Delay First Meas.	Disabled
Analysis	
JDC Ball Mill Feed	
Refractive Index	1.60
Transparency	Transp
Shape	Irregular
WATER	
Refractive Index	1.33
Options:	
Filter	Enabled
Analysis Gain	0
Analysis Mode	S3000/3500
Perspective	
Progression	Standard
Distribution	Volume
Upper Edge(um)	1408
Lower Edge(um)	0.243
Residuals	Enabled

- Notes -

10 buckets/A

Coarse J-Rox - Drum A

Sample System	(Wet)
Number of Rinses	3
Flow Rate (%)	55%
Deaeration Cycles	4

A.5: J-Rox Drum A Particle Size Analysis

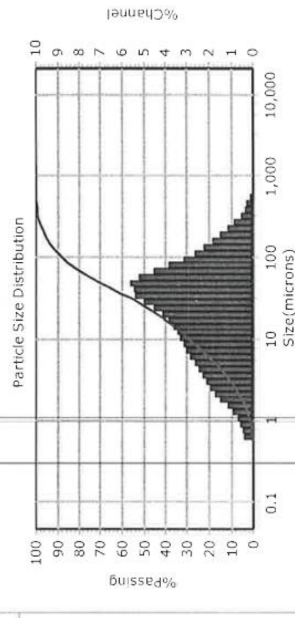
Microtrac — Total Solutions in Particle Characterization

- Particle Size Analysis -

C:\Microtrac\FLEX 11.1.0.4\Databases\novaphos2020.MDB

-Tabular Data -

Size(um)	%Chan	%Pass	Size(um)	%Chan	%Pass
1408	0.00	100.00	3.27	2.13	11.42
1184	0.00	100.00	2.750	1.96	9.29
995.6	0.00	100.00	2.312	1.74	7.33
837.2	0.00	100.00	1.945	1.46	5.59
704.0	0.00	100.00	1.635	1.16	4.13
592.0	0.11	100.00	1.375	0.90	2.97
497.8	0.26	99.89	1.156	0.69	2.07
418.6	0.35	99.63	0.972	0.55	1.38
352.0	0.49	99.28	0.818	0.45	0.83
296.0	0.83	98.79	0.688	0.38	0.38
248.9	1.14	97.96	0.578	0.00	0.00
209.3	1.48	96.82	0.486	0.00	0.00
176.0	1.83	95.34	0.409	0.00	0.00
148.0	2.21	93.51	0.344	0.00	0.00
124.5	2.65	91.30	0.2890	0.00	0.00
104.7	3.17	88.65			
88.00	3.80	85.48			
74.00	4.52	81.68			
62.23	5.18	77.16			
52.33	5.59	71.98			
44.00	5.45	66.39			
37.00	5.37	60.94			
31.11	4.95	55.57			
26.16	4.52	50.62			
22.00	4.14	46.10			
18.50	3.85	41.96			
15.56	3.62	38.11			
13.08	3.44	34.49			
11.00	3.29	31.05			
9.25	3.15	27.76			
7.78	3.00	24.61			
6.54	2.83	21.61			
5.50	2.63	18.78			
4.62	2.45	16.15			
3.89	2.28	13.70			



Summary

Data	Value
MV(um):	46.60
MN(um):	1.137
MA(um):	8.03
CS:	7.47E-01
SD:	38.71
Mz:	37.38
σi:	44.83
Ski:	0.588
Kg:	1.383

Percentiles

%Tile	Size(um)
10.00	2.918
20.00	5.94
30.00	10.42
40.00	16.96
50.00	25.57
60.00	35.92
70.00	49.20
80.00	69.19
90.00	113.9
95.00	170.0

Peaks Summary

Dia(um)	Vol%	Width
25.57	100	77.41

6/8/2020 1:15 PM

FLEX 11.1.0.4

- Measurement Info -

Title	
Jroc coarse	
Identifiers	
6-8-20	
1:10pm	
Database Record	104
Run Number	Avg Of 3
Date	6/8/2020
Time	1:13 PM
Acquired Date	6/8/2020
Acquired Time	1:13 PM
Serial Number	S6309
Sample Cell ID	3151
Calculated Data	
Above Residual	0
Below Residual	0
Loading Factor	0.0242
Transmission	0.961
RMS Residual	0.525%
User Defined Calculations	
Name	Value
Recalculation Status	
Live-Meas : : Original :	

-SOP Info-

BALL MILL FEED(*)	
Timing	
Setzero Time	30 (sec)
Run Time	30 (sec)
Number of Runs	3
Multi-Run Delay	0 (min)
Delay First Meas.	Disabled
Analysis	
JDC Ball Mill Feed	
Refractive Index	1.60
Transparency	Transp
Shape	Irregular
WATER	
Refractive Index	1.33
Options:	
Filter	Enabled
Analysis Gain	0
Analysis Mode	S3000/3500
Perspective	
Progression	Standard
Distribution	Volume
Upper Edge(um)	1408
Lower Edge(um)	0.243
Residuals	Enabled

- Notes -

10 buckets/A

Coarse J Rox - Drum B

Sample System(Wet)
Number of Rinses 3
Flow Rate (%)55%
Deaeration Cycles 4

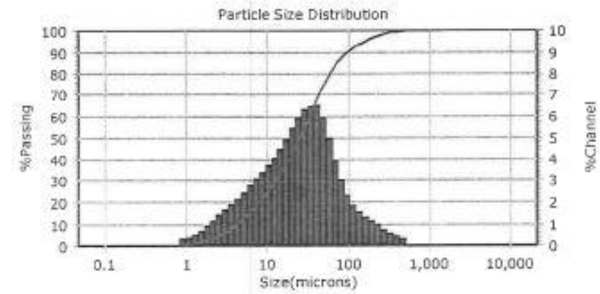
A.6: J-Rox Drum B Particle Size Analysis

Microtrac — Particle Size Analysis

C:\Microtrac\FLEX 11.1.0.4\Databases\novaphos2020.MDB

-Tabular Data -

Size(um)	%Chan	%Pass	Size(um)	%Chan	%Pass
1408	0.00	100.00	3.27	1.65	7.13
1184	0.00	100.00	2.750	1.40	5.48
995.6	0.00	100.00	2.312	1.15	4.08
837.2	0.00	100.00	1.945	0.92	2.93
704.0	0.00	100.00	1.635	0.71	2.01
592.0	0.00	100.00	1.375	0.54	1.30
497.8	0.34	100.00	1.156	0.42	0.76
418.6	0.44	99.66	0.972	0.34	0.34
352.0	0.58	99.22	0.818	0.00	0.00
296.0	0.76	98.64	0.688	0.00	0.00
248.9	0.96	97.88	0.578	0.00	0.00
209.3	1.14	96.92	0.486	0.00	0.00
176.0	1.32	95.78	0.409	0.00	0.00
148.0	1.54	94.46	0.344	0.00	0.00
124.5	1.85	92.92	0.2890	0.00	0.00
104.7	2.34	91.07			
88.00	3.03	88.73			
74.00	3.96	85.70			
62.23	5.00	81.74			
52.33	5.93	76.74			
44.00	6.53	70.81			
37.00	6.43	64.28			
31.11	6.36	57.85			
26.16	5.93	51.49			
22.00	5.43	45.56			
18.50	4.93	40.13			
15.56	4.46	35.20			
13.08	4.05	30.74			
11.00	3.71	26.69			
9.25	3.40	22.98			
7.78	3.10	19.58			
6.54	2.78	16.48			
5.50	2.48	13.70			
4.62	2.18	11.22			
3.89	1.91	9.04			



Summary

Data	Value
MV(um):	43.52
MN(um):	1.665
MA(um):	10.57
CS:	5.67E-01
SD:	30.99
Mz:	33.26
σι:	39.12
Ski:	0.554
Kg:	1.620

Percentiles

%Tile	Size(um)
10.00	4.21
20.00	7.95
30.00	12.69
40.00	18.42
50.00	25.08
60.00	32.98
70.00	43.06
80.00	58.37
90.00	96.16
95.00	158.5

Peaks Summary

Dia(um)	Vol%	Width
25.08	100	61.99

3/26/2021 9:21 AM

FLEX
11.1.0.4

- Measurement Info -

Title
JR Bottom
Identifiers
-
3/6/21
Database Record 580
Run Number Avg Of 3
Date 3/26/2021
Time 9:19 AM
Acquired Date 3/26/2021
Acquired Time 9:19 AM
Serial Number S6309
Sample Cell ID 3151
Calculated Data
Above Residual 0
Below Residual 0
Loading Factor 0.0261
Transmission 0.964
RMS Residual 0.431%
User Defined Calculations
Name Value
Recalculation Status
Live-Meas : : Original :

-SOP Info-

BALL MILL FEED(*)
Timing
Setzero Time 30 (sec)
Run Time 30 (sec)
Number of Runs 3
Multi-Run Delay 0 (min)
Delay First Meas. Disabled
Analysis
JDC Ball Mill Feed
Refractive Index 1.60
Transparency Transp
Shape Irregular
WATER
Refractive Index 1.33
Options:
Filter Enabled
Analysis Gain 0
Analysis Mode S3000/3500
Perspective
Progression Standard
Distribution Volume
Upper Edge(um) 1408
Lower Edge(um) 0.243
Residuals Enabled

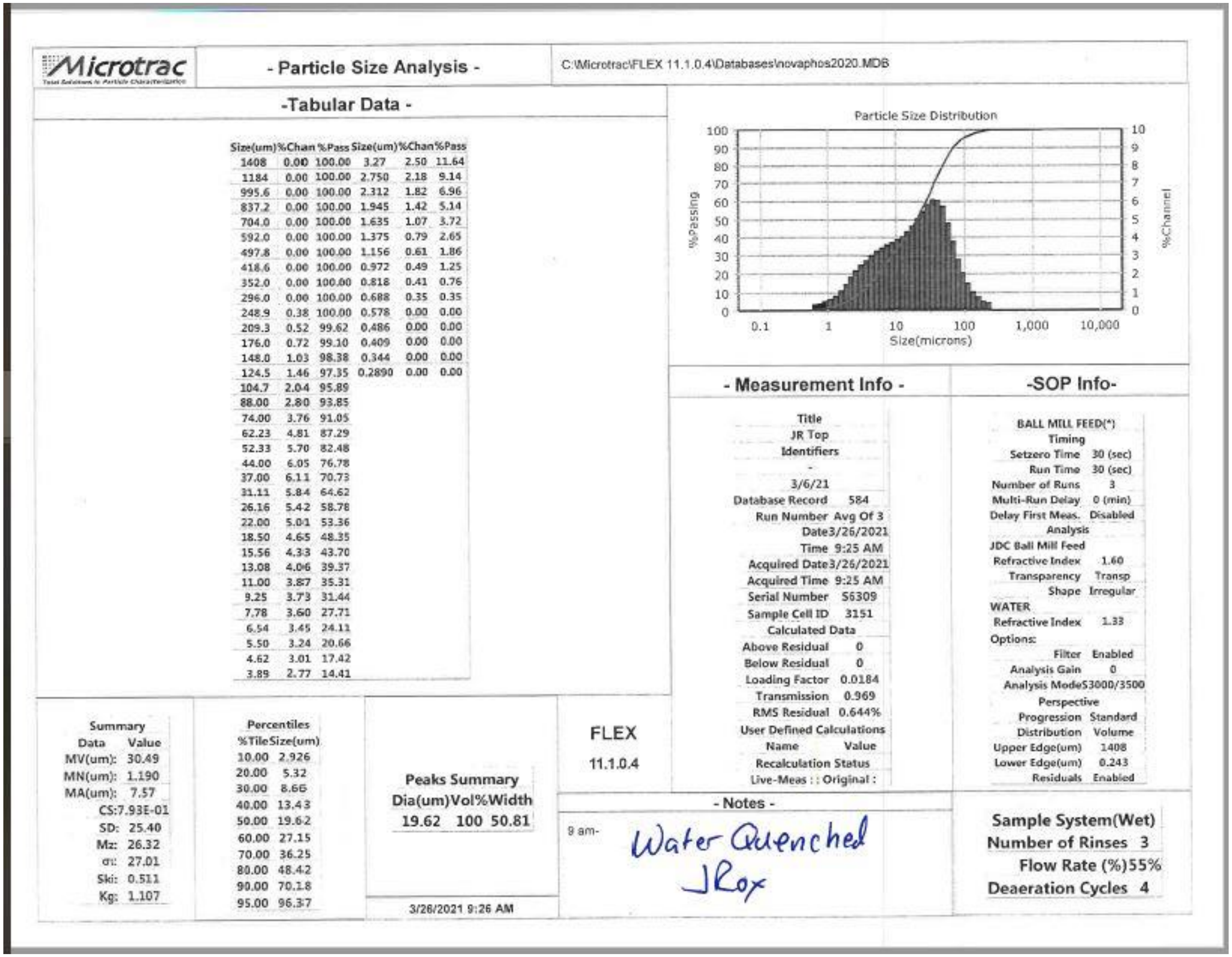
- Notes -

9 am-

Air Cooled JRox

Sample System(Wet)
Number of Rinses 3
Flow Rate (%) 55%
Deaeration Cycles 4

A. 7: J-Rox 4 Air Cooled Particle Size Distribution


Microtrac - Particle Size Analysis - C:\Microtrac\FLEX 11.1.0.4\Databases\novaphos2020.MDB

-Tabular Data -

Size(um)	%Chan	%Pass	Size(um)	%Chan	%Pass
1408	0.00	100.00	3.27	2.50	11.64
1184	0.00	100.00	2.750	2.18	9.14
995.6	0.00	100.00	2.312	1.82	6.96
837.2	0.00	100.00	1.945	1.42	5.14
704.0	0.00	100.00	1.635	1.07	3.72
592.0	0.00	100.00	1.375	0.79	2.65
497.8	0.00	100.00	1.156	0.61	1.86
418.6	0.00	100.00	0.972	0.49	1.25
352.0	0.00	100.00	0.818	0.41	0.76
296.0	0.00	100.00	0.688	0.35	0.35
248.9	0.38	100.00	0.578	0.00	0.00
209.3	0.52	99.62	0.486	0.00	0.00
176.0	0.72	99.10	0.409	0.00	0.00
148.0	1.03	98.38	0.344	0.00	0.00
124.5	1.46	97.35	0.2890	0.00	0.00
104.7	2.04	95.89			
88.00	2.80	93.85			
74.00	3.76	91.05			
62.23	4.81	87.29			
52.33	5.70	82.48			
44.00	6.05	76.78			
37.00	6.11	70.73			
31.11	5.84	64.62			
26.16	5.42	58.78			
22.00	5.01	53.36			
18.50	4.65	48.35			
15.56	4.33	43.70			
13.08	4.06	39.37			
11.00	3.87	35.31			
9.25	3.73	31.44			
7.78	3.60	27.71			
6.54	3.45	24.11			
5.50	3.24	20.66			
4.62	3.01	17.42			
3.89	2.77	14.41			

Summary

Data	Value
MV(um):	30.49
MN(um):	1.190
MA(um):	7.57
CS:	7.93E-01
SD:	25.40
Mz:	26.32
σi:	27.01
Ski:	0.511
Kg:	1.107

Percentiles

%Tile	Size(um)
10.00	2.926
20.00	5.32
30.00	8.66
40.00	13.43
50.00	19.62
60.00	27.15
70.00	36.25
80.00	48.42
90.00	70.18
95.00	96.37

Peaks Summary

Dia(um)	Vol%	Width
19.62	100	50.81

3/26/2021 9:26 AM

FLEX 11.1.0.4

- Measurement Info -

Title
JR Top
Identifiers
-
3/6/21
Database Record 584
Run Number Avg Of 3
Date 3/26/2021
Time 9:25 AM
Acquired Date 3/26/2021
Acquired Time 9:25 AM
Serial Number S6309
Sample Cell ID 3151
Calculated Data
Above Residual 0
Below Residual 0
Loading Factor 0.0184
Transmission 0.969
RMS Residual 0.644%
User Defined Calculations
Name Value
Recalculation Status
Live-Meas : : Original :

-SOP Info-

BALL MILL FEED(*)
Timing
Setzero Time 30 (sec)
Run Time 30 (sec)
Number of Runs 3
Multi-Run Delay 0 (min)
Delay First Meas. Disabled
Analysis
JDC Ball Mill Feed
Refractive Index 1.60
Transparency Transp
Shape Irregular
WATER
Refractive Index 1.33
Options:
Filter Enabled
Analysis Gain 0
Analysis Mode S3000/3500
Perspective
Progression Standard
Distribution Volume
Upper Edge(um) 1408
Lower Edge(um) 0.243
Residuals Enabled

- Notes -

9 am- Water Quenched JRox

Sample System(Wet)
Number of Rinses 3
Flow Rate (%)55%
Deaeration Cycles 4

A. 8: J-Rox 4 Water Quenched Particle Size Distribution

SGS TEC SERVICES
Testing • Engineering • Consulting

September 30, 2020

Ms. Mark Vignovic
Novaphos LLC
3200 CR 630 West
Fort Meade, FL 33841

Phone: (863) 285-8607
Fax: (863) 285-8504
Email: mvignovic@novaphos.com

Subject: **Report of Results for Product Testing**
Product Name: High Silica 9/18/19, Low Silica 2/6/20 and Drum X 6/8/20
TEC Services Project #: 20-1644
TEC Laboratory #: 20-933

Dear Mr. Vignovic:

SGS TEC Services is an AASTHO R18, ANS/ISO/IEC 17025:2005 and Army Corp of Engineers accredited laboratory. SGS TEC Services is pleased to present this report of our test results on the submitted material designated as "High Silica 9/18/19, Low Silica 2/6/20 and Drum X 6/8/20". Our services were performed in accordance with the terms and conditions of our Service Agreement TEC-PRO-20-1644. The test results presented only pertain to the samples tested.

The material was delivered to our Lawrenceville, GA facility on August 10, 2020. At the request of Novaphos Development LLC, testing was performed on the material per ASTM C618-19 *Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete*. The chemical analysis test results are reported in attached Table 1.

SGS TEC Services appreciates the opportunity to provide our professional services for this important project. If you have any questions regarding this report, or if we can be of further assistance please contact us at 770-995-8000.

Sincerely,

SGS TEC SERVICES

Dean T. Roosa
Project Manager

Shawn P. McCormick
Laboratory Principal

ISO 17025 ACCREDITED LABORATORY
US Army Corps of Engineers

SGS TEC SERVICES
235 Buford Drive | Lawrenceville GA 30046
770-995-8000 | www.tecservices.com

AASHTO R18
ASTM INTERNATIONAL

A.9: Chemical Analysis Comparison of J-Rox 1, J-Rox 2, and J-Rox Drum X Page 1

Report of Results for Product Testing *September 30, 2020*
Product Name: High Silica 9/18/19, Low Silica 2/6/20 and Drum X 6/8/20
TEC Services Project #: 20-1644
TEC Laboratory #: 20-993

Table 1 – Results of the Chemical Analysis

Oxides	Results Weight (%) High Silica 9/18/19	Results Weight (%) Low Silica 2/6/20	Results Weight (% Drum X 6/8/20)
Silicon Dioxide (SiO_2)	67.4	29.5	50.9
Aluminum Oxide (Al_2O_3)	1.3	1.5	1.4
Iron Oxide (Fe_2O_3)	2.82	2.24	2.61
Sum (SiO_2 + Al_2O_3 + Fe_2O_3)	71.6	33.2	54.9
Calcium Oxide (CaO)	23.3	43.2	31.4
Magnesium Oxide (MgO)	2.2	3.9	3.1
Sodium Oxide (Na_2O)	0.36	0.60	0.46
Potassium Oxide (K_2O)	0.30	0.21	0.27
Equivalent Alkalies (Na_2O+0.658 K_2O)	0.56	0.74	0.64
Titanium Dioxide (TiO_2)	0.08	0.08	0.08
Phosphorus Pentoxide (P_2O_5)	1.12	11.47	6.52
Sulfur Trioxide (SO_3)	0.65	1.43	0.78
Loss on Ignition	2.2	6.4	2.4
Moisture Content	0.20	0.28	0.22
Total Available Alkali			
Sodium Oxide (Na_2O)	0.14	0.36	0.19
Potassium Oxide (K_2O)	0.12	0.10	0.10
Equivalent Alkalies (Na_2O+0.658 K_2O)	0.22	0.43	0.26

Page **2** of **2**

A.10: Chemical Analysis Comparison of J-Rox 1, J-Rox 2, and J-Rox Drum X Page 2

Sample Time	Sample Date	% F	SCM (Si, Fe, Al)	% SiO_2	% P_2O_5	% CaO	% MgO	% Al_2O_3	% Fe_2O_3	% Na	% K	% SO_4	As (ppm)	Cd (ppm)	Cr (ppm)	Ni (ppm)	Pb (ppm)	Zn (ppm)	U (ppm)	% C (avg)	% S. (avg)
3:45 PM	3/5/21	0.89	63.6	60.8	10.9	20.2	1.53	0.65	2.1	0.3	0.1	0.63	2.25	0.13	63	101	1.9	11	33.51	2.0	0.26
4:15 PM	3/5/21	0.85	64.7	62.0	9.1	20.6	1.61	0.84	1.95	0.33	0.12	0.72	1.15	0.11	59	97	2.28	16	43.34	1.0	0.28
2:00 PM	3/11/21	0.79	63.5	60.0	12.6	20.1	1.44	0.46	3.02	0.32	0.07	0.36	2.18	0.33	73	102	1.31	15	24.57	1.3	0.13
2:30 PM	3/11/21	0.79	63.4	59.5	13.0	20.1	1.45	0.44	3.44	0.38	0.06	0.41	2.43	0.25	77	97	1.01	7	23.61	1.7	0.16
3:00 PM	3/11/21	0.89	61.3	57.9	13.0	21.0	1.53	0.5	2.96	0.32	0.05	0.56	1.88	0.2	65	94	1.22	9	21.57	2.4	0.23
3:30 PM	3/11/21	0.85	61.7	58.6	12.6	21.0	1.57	0.55	2.55	0.33	0.07	0.56	1.64	0.18	60	103	1.14	12	20.62	1.6	0.20
4:00 PM	3/11/21	0.84	61.3	58.0	12.2	21.6	1.64	0.7	2.58	0.38	0.09	0.67	1.55	0.18	60	105	1.81	11	31.58	1.5	0.25
4:30 PM	3/11/21	0.65	61.9	58.9	11.3	21.6	1.64	0.82	2.12	0.37	0.11	0.79	1.56	0.16	50	101	1.3	12	38.11	1.2	0.27
4:50 PM	3/11/21	0.59	61.9	59.0	11.9	22.3	1.68	0.78	2.2	0.36	0.09	0.74	1.79	0.29	61	104	0.48	11	45.8	1.2	0.26
5:15 PM	3/11/21	0.85	61.4	58.5	10.7	21.0	1.64	0.85	2.04	0.38	0.1	0.82	2.33	0.3	60	102	1	13	50.44	1.4	0.28
5:30 PM	3/11/21	0.74	61.5	58.6	10.8	21.2	1.65	0.94	1.96	0.38	0.11	0.84	1.7	0.26	55	102	1.02	13	48	1.0	0.27
5:45 PM	3/11/21	0.79	62.1	59.3	10.5	21.9	1.7	0.98	1.86	0.38	0.12	0.85	1.47	0.25	54	104	1.02	13	50.4	0.7	0.27
6:00 PM	3/11/21	0.80	60.3	57.7	10.1	20.5	1.56	0.85	1.78	0.36	0.11	0.85	1.52	0.25	53	102	1.3	12	56.11	1.2	0.34
6:15 PM	3/11/21	0.84	61.7	58.9	11.0	21.6	1.67	0.84	1.96	0.37	0.11	0.87	1.74	0.28	58	106	0.48	11	55.29	1.0	0.32
1:30 PM	3/12/21	0.74	62.5	59.7	9.8	18.0	1.32	0.55	2.19	0.27	0.09	0.45	2.15	0.24	80	95	0.72	14	10.64	0.9	0.21
1:45 PM	3/12/21	0.79	63.1	60.1	10.4	21.1	1.61	0.8	2.2	0.31	0.1	0.69	1.35	0.24	72	100	0.44	19	29.73	0.8	0.27
2:00 PM	3/12/21	0.84	62.0	59.3	10.7	20.5	1.57	0.77	1.94	0.3	0.11	0.72	1.45	0.15	67	98	3.19	112	27.55	1.3	0.29
2:15 PM	3/12/21	0.85	62.1	59.7	9.3	18.9	1.43	0.7	1.7	0.28	0.09	0.71	1.45	0.14	63	95	1.39	16	23.89	1.1	0.33
2:30 PM	3/12/21	1.05	62.0	59.0	11.5	21.5	1.61	0.67	2.3	0.28	0.07	0.73	2.32	0.16	77	104	1.46	13	26.96	1.5	0.29
3:30 PM	3/12/21	0.64	61.8	58.7	11.8	22.1	1.67	0.82	2.27	0.32	0.11	82	0.6	0.16	55	98	10.56	233	24.29	0.8	0.29
3:45 PM	3/12/21	0.74	61.2	58.5	11.4	22.6	1.75	0.92	1.76	0.34	0.11	0.86	0.62	0.2	75	106	0.94	12	26.1	0.7	0.27
4:00 PM	3/12/21	0.70	62.7	59.5	10.2	22.1	1.72	0.97	2.21	0.34	0.13	0.97	0.12	0.16	76	106	0.952	12	26.29	0.8	0.34
4:15 PM	3/12/21	0.79	62.6	59.6	9.9	22.2	1.72	0.98	2.09	0.34	0.16	1	0.48	0.17	70	95	1.17	17	29.94	0.5	0.30
4:30 PM	3/12/21	0.85	62.3	59.2	10.3	21.9	1.67	0.96	2.14	0.34	0.16	0.93	0.34	0.15	72	96	1.35	118	23.12	0.5	0.33
4:45 PM	3/12/21	0.89	62.0	58.5	10.3	22.2	1.72	1.01	2.44	0.34	0.14	0.97	0.38	0.19	83	113	1.14	9	25.82	1.0	0.31
5:00 PM	3/12/21	0.94	62.5	59.5	10.1	21.8	1.69	0.99	2.05	0.36	0.13	0.99	0.41	0.16	69	102	1.23	12	25.53	0.9	0.34
5:15 PM	3/12/21	0.74	61.8	58.7	10.6	22.4	1.7	0.97	2.07	0.34	0.12	1.04	0.33	0.14	66	100	1.16	19	23.47	1.4	0.38
5:30 PM	3/12/21	0.79	61.1	57.9	11.2	22.4	1.73	0.96	2.3	0.35	0.12	0.98	1.13	0.31	62	103	1.04	10	46.07	1.3	0.35
5:45 PM	3/12/21	0.80	62.6	59.7	10.6	22.1	1.65	0.93	1.96	0.34	0.11	0.91	1.06	0.23	54	96	1.13	29	56.19	1.2	0.33
6:00 PM	3/12/21	0.79	62.2	59.1	10.8	22.0	1.65	0.88	2.2	0.32	0.1	0.82	1.97	0.28	61	93	1.18	8	49.43	1.1	0.29
12:00 PM	3/15/21	0.74	62.1	59.3	12.6	22.1	1.64	0.64	2.2	0.43	0.14	0.63	1.96	0.17	63	93	0.31	8	31.6	1.0	0.20
12:15 PM	3/15/21	0.69	62.4	59.5	12.5	21.2	1.53	0.6	2.28	0.38	0.08	0.51	1.18	0.15	69	100	0.58	4	29.97	0.7	0.20
12:30 PM	3/15/21	0.74	63.3	60.7	13.4	21.6	1.53	0.61	2.03	0.29	0.1	0.36	1.55	0.14	61	100	0.41	7	32.92	0.4	0.11
12:45 PM	3/15/21	0.80	63.2	60.4	11.9	21.8	1.62	0.68	2.16	0.28	0.08	0.67	0.88	0.14	64	99	0.35	27	29.31	0.4	0.23
1:00 PM	3/15/21	0.59	62.5	59.2	11.9	21.7	1.69	0.73	2.55	0.3	0.08	0.9	0.81	0.18	81	108	0.65	11	29.27	1.1	0.28
1:15 PM	3/15/21	0.60	62.5	59.5	11.6	21.7	1.66	0.73	2.34	0.28	0.07	0.82	0.6	0.15	70	103	1.18	14	32.54	0.9	0.29
1:30 PM	3/15/21	0.64	61.9	58.7	11.3	21.1	1.63	0.74	2.46	0.32	0.09	0.85	0.37	0.14	79	106	1.36	40	34.33	0.7	0.29
1:45 PM	3/15/21	0.70	62.9	59.9	10.4	21.6	1.72	0.85	2.11	0.36	0.11	1.17	0.8	0.13	69	108	1.24	263	41.81	0.8	0.35
2:00 PM	3/15/21	0.69	62.2	59.6	10.6	21.5	1.63	0.86	1.73	0.31	0.1	0.96	0.53	0.11	55	99	0.72	13	33.46	0.7	0.35
2:15 PM	3/15/21	0.84	62.3	59.5	10.5	21.3	1.65	0.87	1.93	0.32	0.1	1.05	0.82	0.12	58	98	0.8	15	30.08	0.8	0.36
2:30 PM	3/15/21	0.74	62.0	59.0	11.1	22.1	1.73	0.84	2.14	0.3	0.08	1.02	1.02	0.13	69	109	0.97	21	39.11	0.6	0.33
	Average	**0.78**	**62.2**	**59.3**	**11.1**	**21.4**	**1.62**	**0.79**	**2.20**	**0.33**	**0.10**	**2.77**	**1.27**	**0.19**	**66**	**101**	**1.34**	**30**	**34**	**1.0**	**0.28**
	Water Quenched	**0.7**	**61.7**	**59.4**	**11.3**	**20.9**	**1.59**	**0.71**	**1.8**	**0.65**	**0.11**	**1.36**	**1.35**	**0.13**	**54**	**106**	**1.14**	**18**	**44**	**1.2**	**0.27**

A.11: Chemical Analysis of J-Rox 4

Parameter	Units	December 2019 - March 2020				June 8, 2020			November 20, 2020		March 26, 2021	
		High Silica	SGS	Low Silica	SGS	Fine (X&O)	Coarse (A&B)	SGS Drum X	J-Rox 3	Ground Sample Retains*	Air Cooled	Water Quenched
F	%	1.2		2.5		1.4	1.2		1.0	0.9	1.0	0.8
SiO2	%	61.8	67.4	30.3	29.5	51.8	54.9	50.9	63.4	57.2	58.9	57.2
P2O5	%	4.2	1.1	11.8		6.4	5.5	6.5	2.5	5.3	11.4	11.8
CaO	%	24.1	23.3	42.6	43.2	30.9	28.7	31.4	24.0	25.6	20.6	20.3
MgO	%	2.1	2.2	4.4	3.9	3.0	2.7	3.1	2.1	2.3	1.7	1.6
Al2O3	%	0.8	1.3	2.1	1.5	1.3	1.2	1.4	0.9	1.0	0.8	0.6
Fe2O3	%	1.5	2.8	1.8	2.2	3.1	2.0	2.8	1.5	2.2	1.7	1.6
Na	%	0.2	0.4	0.6	0.6	0.4	0.4	0.5	0.3	0.3	0.3	0.3
K	%	0.1	0.3	0.2	0.2	0.1	0.1	0.3	0.1	0.1	0.1	0.1
SO4	%	2.4		2.5		1.9	1.8		1.7	1.6	0.7	0.6
C	%	2.4		3.0		1.9	1.8		1.1	2.5	1.6	3.2
S	%	0.6		1.0		0.7	0.6		0.7	0.6	0.3	0.3
As	ppm	1.1		2.1		3.1	4.6		0.6	1.1	1.3	1.7
Cd	ppm	0.2		0.2		0.42	0.3		0.1	0.3	0.2	0.2
Cr	ppm	74		251		92	83		88.0	92	49	48
Ni	ppm	171		251		220	184		159.0	169	111	108
Pb	ppm	0.8		0.4		0.8	0.2		0.0	0.5	1.2	1.2
Zn	ppm	125		70		13	12		7.0	20	24	13
U	ppm	99		129		218	171		41.0	139	31	44
									* ball mill had trace amounts of previous ground J-Rox			

A. 12: Chemical Compositions of all J-Rox Samples

APPENDIX C: SUPPLLEMENTAL INFORMATION FOR CHAPTER 5

Table 0.1: FDOT Resistivity Specifications (Cavalline et al., 2018)

State/Standard	Resistivity Specification		
	Concrete Type	Requirement (kΩ-cm)	Age
Florida DOT special circumstances. Implemented AASHTO T 358 in January 2017	Ternary blend - extremely aggressive environment	> 29	28 days
	Ternary blend - moderately aggressive environment	17-29	28 days
	Ternary blend - slightly aggressive environment	< 17	28 days
	Structural Concretes: Class IV, V, V (special), VI with use of silica fume, ultrafine fly ash, or metakaolin	≥ 29	28 days
	Ultra-high performance repair material for vertical surfaces	≥ 22	28 days
	Special fillers for cathodic protection	Can be 15 or less	28 days
	Special fillers for non-cathodic protection	≥ 22	28 days