# USE OF GRANULATED LIMESTONE AS FINE AGGREGATE

By

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DEDICATION

This document is dedicated to Dr. James Eades.

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The main objective of this study was to evaluate Portland Cement (PC) concrete using granulated limestone fines as a replacement for fine aggregate. Three types of fines (Ag82, dolomite and AgDry) and two different binders (sodium silicate and PC) were used in this investigation. Concrete made with Ottawa sand and FDOT road construction grade sand were compared to evaluate their potential for use in industrial applications in PC concrete. The effects of curing with and without fly ash, and at elevated temperatures were also evaluated in PC samples. Samples were tested for unconfined compressive strength, photographed under a petrographic microscope and photographed by SEM techniques.

A number of conclusions can be drawn from this study. Ag82 granulated with a PC binder showed the best potential for use as a replacement for fine aggregate in commercial PC concrete with an average 28-day unconfined compressive strength of 4200 psi. Sodium silicate was a much weaker binding agent than PC and possibly showed signs of solubility and alkali silica reactions in the samples. Concrete cured at elevated temperatures with fly

ash showed greater values of unconfined compressive strength than samples cured at elevated temperatures without fly ash, and samples cured at ambient temperatures with or without fly ash.

# CHAPTER 1
# INTRODUCTION

Across the United States it is estimated that 100 to 200 million short tons of byproduct fines (minus 200 mesh fines that are a byproduct of aggregate mining) are accumulated annually (University of Florida, 2002). This accumulation is directly related to the fact that two tons of stone must be mined for every ton that can be sold as a construction product. Fines constitute the majority of the waste product produced from mining and must be stored in settling ponds and screen piles.

As construction and infrastructure grow there is an increased demand on the crushed stone industry and therefore the rate of accumulation of byproduct fines continues to increase as well. Disposal and loss of sales costs associated with the byproduct fines weigh heavily on aggregate producers each year. This problem has prompted the International Center for Aggregate Research to create the Fines Expert Task Group to investigate and find uses for fine materials.

Identifying technically and economically feasible uses for these byproduct fines is important to the aggregate industry. Agricultural additives, soil conditioners and fill materials are presently the most common uses for fine materials. Physical or chemical modification of fines could transform them into useful products, rather than a byproduct for disposal or stockpiling.

In Florida stockpiling of byproduct fines is an increasing problem because of the growth of the aggregate mining industry. Since 1970, Florida's crushed stone industry has grown from approximately 40 to 90 million tons per year. Florida's Department of Transportation (FDOT) could benefit from a usage for its aggregate producer's byproduct fines. Much of the stone that is mined in Florida is used for concrete mix and the FDOT is interested in an effective and economic way to use these byproduct fines that are not presently used in concrete mix.

This study investigated and evaluated the performance of granulated limestone fines as aggregate in Portland cement (PC) concrete. Three types of fines were granulated with two binders to produce the fine aggregate for testing. Ottawa sand and a FDOT construction grade sand from the Grandin Sand Mine in Grandin, Florida, for PC and asphalt concretes were used as the standard fine aggregate for comparison.

Fly ash is commonly used in PC. Fly ash may be used as a partial substitute for the cement and/or as fine aggregate (i.e., replacing part or all of the sand). The effects of adding fly ash are to improve workability of the mix, increase early strength, and to decrease the cost of the concrete. This study evaluates the strength of concrete mixes, designed with and without fly ash, cured at room temperature or at 120 degrees Fahrenheit (°F).

Unconfined compression testing was done on 2-inch by 2-inch cubes of the different types of PC concrete. Variations in unconfined compressive strength were seen in the cubes that were consistent with the microscopic evaluations done on thin sections of the cubes. Strength was imparted to the cubes with a good grain-cement bond and/or with a strong, well-bonded fine aggregate. Weaknesses were seen in the cubes that had poor grain-cement bonding and/or weak fine aggregate binding properties. Fly ash made the final PC concrete stronger when cured at elevated temperatures, and weaker when cured at room temperature. PC concrete cubes were strongest when fly ash was added, and curing was done at elevated temperatures.

# CHAPTER 2
# FUNDAMENTALS

## Background

This chapter describes the various properties and aspects of concrete that have influenced this study. First, there is an overview of the quantity, characteristics and state of the byproduct fines that are found in Florida. Second, the properties of the byproduct fines that were used in the concrete cubes of this study are reviewed. Finally, a generalized summary of many of the materials that were used, properties that may have affected the products and techniques that were utilized in this study are listed and described.

## Byproduct Fines in Florida

The stone production industry of Florida has been steadily growing over the past 30 years, with smaller fluctuations occurring during periods of economic change. As the stone production industry grows, so does the accumulation of byproduct fines. The majority of these byproduct fines are generated during the physical alteration of larger stones to make smaller stone products.

Characterization of the amount and properties of the byproduct fines that are being produced and stockpiled is important. After this characterization has been completed, economic uses for the fines can be evaluated according to the actual characteristics of the fines that are being produced and stored in Florida. This process may enable stone producers to find a feasible and potentially a financially beneficial way to dispose of their byproduct fines.

The University of Florida published a paper in 2002 that characterized the byproduct fines produced and stored in Florida. This task was accomplished by contacting and studying stone producers throughout Florida. Producers of approximately 80% of the crushed stone generated annually in Florida were contacted. These producers were geographically well distributed throughout Florida, with the exception of the northwest. The paper's findings were verified and supplemented with historical research. The findings are summarized as follows:

First of all, the methods used to estimate the past and future production of fines allow for the magnitude of this storage problem to be quantified. There are estimated to be 38 million tons of byproduct fines stockpiled in the state of Florida. The findings of this study agree with past studies, but results indicate that changes in technology, over time, may have decreased the rate at which byproduct fines are produced. In spite of the potential reduction of production rate, future projections show that annual production is estimated to increase from 93 to 112 million tons per year between the years 2000 and 2010.

Second, the study characterized byproduct fines of Florida according to moisture content, acid insoluble contents, gradation and mineralogy. The fines were evaluated according to two classifications: dolomitic limestone/dolomite and limestone. These findings are summarized below.

**Moisture content.** All fines of this study were found to have high moisture content. The moisture content varied between samples but mean moisture content was calculated for the sample groups. The dolomitic limestone/dolomite and the limestone samples have a mean moisture content of 16.8 and 18.4%, respectively.

**Acid insoluble contents.** This study showed the limestone fines to have a higher acid insoluble content percentage (11.4%) when compared to the dolomitic limestone/dolomites (2.1%). Quartz was by far the largest acid insoluble constituent (clays, pyrite, rutile and geothite was also present).

**Gradation**. Particle size distribution varied between samples. This variation is a result of processing methods, and physical and chemical make-up of the fines at the various mines. Hydrometer and wet sieve analysis results were similar, indicating slightly more minus-325 material in the limestone as compared to the dolomitic limestone/dolomite. There were substantial amounts of clay in the fines that were studied. Clay can significantly change the properties of byproduct fines and should be properly considered when evaluating the fines for economic uses.

**Mineralogy**. Inherent differences in mineralogy were seen when comparing dolomitic limestone/dolomite with limestone. The mean dolomite concentration (by volume) for dolomitic limestone/dolomite and limestone was seen to be 91.3 and 5.7%, respectively. The mean calcite concentration for dolomitic limestone/dolomite and limestone was 8.4 and 80.1%, respectively. Less anticipated was the fact that calcite was more concentrated in the fine fraction (minus-325 mesh) for both classifications.

Finally, new and better products made with the byproducts and methods of marketing may greatly reduce the amount of byproduct fines that need to be disposed of or stored. In the past only 34.4% of byproduct fines were marketed and sold. In planning for the future, the companies that produce these byproduct fines feel a need for research and marketing assistance to aid in economically feasible methods of disposal of their byproduct fines according to the volume, location and characteristics of byproduct fines as they exist in Florida.

## Concrete

Concrete is likely to be a word of Latin origin that is based on hydraulic cement, a material that hardens with the addition of water (Neville and Brooks, 1987). Concrete is a composite material comprised of a binder, filler and water. The properties of the filler and binder directly affect the properties of the concrete. The filler is generally fine and course aggregate. Additives can be added to the filler or the binder to further alter the properties of the concrete.

## Portland Cement

This study deals with concrete that uses PC as the binder. PC is commonly used in cement and is a mixture of calcareous and argillaceous or other silica-, alumina- and iron oxide- bearing materials, which are burned at a clinkering (i.e., sintering) temperature (~2550°F), and ground (Davis, 1948). Basically, lime, silica, alumina, and iron oxide interact with one another in a kiln to form the major constituents of cement tricalcium silicate ($C_3S$), dicalcium silicate ($C_2S$), tricalcium aluminate ($C_3A$), and tetracalcium aluminoferrite ($C_4AF$). Table 1 outlines the main constituents of PC. These components are then ground into a powder to increase surface area in preparation for hydration with water and mixing with filler in a concrete mix.

When water and a filler are added to PC and allowed to cure, PC concrete is formed. Tricalcium silicate and dicalcium silicate are the most important compounds in the system and are responsible for the strength of hydrated cement paste. Hydration of the calcium occurs when water is added to the PC and a layer structure cement gel (C-S-H) is created (Idorn and Roy, 1985). The PC concrete should gain strength for the next 28 days.

Compressive strength tests show strength gain at a slower rate after the initial 28-day strengthening period (Lange, 1994). Immediately (first 7 days) after water is added to PC tricalcium silicate hydrates, and is responsible for the early strength of the concrete. Dicalcium silicate reacts at a slower rate, but is still an important constituent in the concrete. Tricalcium aluminate is considered undesirable in concrete, contributing very little to the strength of the concrete. Tetracalcium aluminoferrite is usually present in small quantities and comparatively, does not affect concrete strength.

Table 1: Main constituents of PC

| Name of Compound | Oxide Composition | Abbreviations | Hydration Reaction |
|---|---|---|---|
| *Tricalcium silicate | 3CaO.SiO2 | C3S | 2C3S + 6H => C3S2H3 + 3Ca(OH)2 |
| *Dicalcium silicate | 2CaO.SiO2 | C2S | 2C2S + 4H => C3S2H3 + Ca(OH)2 |
| Tricalcium aluminate | 3CaO.Al2O3 | C3A | C3A + 6H => C3AH6 |
| Tetracalcium aluminoferrite | 4CaO.Al2O3Fe2O3 | C4AF | |

*Most important compounds.
Note: CaO = C; SiO2 = S; Al2O3 = A; Fe2O3 = F; H = $H_2O$
(Neville and Brooks, 1987)

**Sand and Aggregate**

Sand and aggregate are generally the materials that are added to a PC concrete mix as filler. Size and shape of the sand and aggregate, added to concrete, can greatly affect the resulting strength and other properties of concrete. The inherent strength of the sand and aggregate affects the strength of the final PC concrete product as much as the bonding strength of the binder. PC concrete is generally only as strong as the coarse aggregate within, except in high strength and some lightweight concretes (Sarkar, 1990). The gradation of the finer particles also affects the workability or ease of placement, of concrete (Murdock and Brook, 1979). Finally, cleanliness of all materials will affect the strength and durability of the final product. The ability of the binder to create a strong bond can be negatively affected by the addition of unwanted minerals or chemicals from improperly cleaned filler.

The physical properties of the sand and aggregate can affect the strength of the concrete because they affect the porosity of the cement mix. Adequate porosity and surface area must be available in a concrete mix for the binder to fully surround and adhere to the filler. For example, oddly shaped particles can nestle and leave less room for the binder, and therefore weaken the strength of the concrete. In order to produce the same quality concrete as that made with nearly round or cubic aggregate, oddly shaped, rough, sharp or flat particles will require more fine particles to be added to the mix (Portland Cement Association, 1952).

## Water to Cement Ratio

The water to cement ratio (W/C) is based on the relative volumes of water and cement in a concrete mix and has a direct effect on the strength of PC concrete (Figure 1). A sufficient amount of water is essential for the strength of concrete. PC concrete with too little water may never reach its full strength potential because it is not fully reacted. PC concrete with too much water will have water trapped in the pore space that weakens the overall strength. Generally, there is a negative, straight-line relationship between the compressive strength and the porosity of concrete (Neville and Brooks, 1997). As seen in figure 1, as the W/C ratio increases the strength of PC concrete decreases. PC concrete that has been insufficiently compacted (hand compaction) will show a steep decline in compressive strength with a low W/C (Neville and Brooks, 1997) (Figure 1).

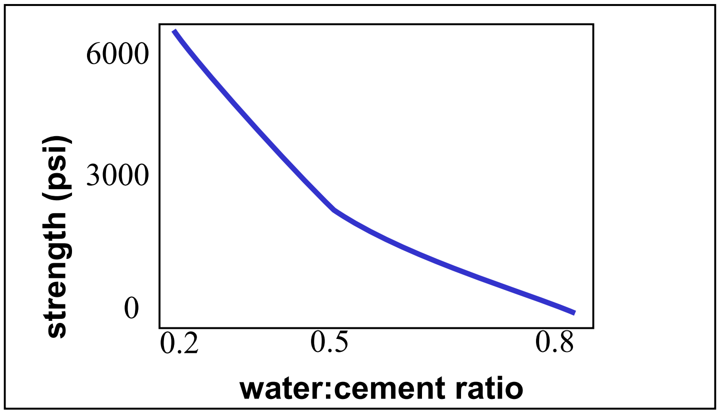


Figure 1: General relationship between strength and water to cement ratio.

**Fly ash**

Fly ash is a waste product of coal burning power plants. Fly ash consists of silica ($SiO_2$), alumina ($Al_2O_3$) and oxides of iron ($Fe_2O3$) and sometimes calcium ($CaO$) (Joshi and Lohtia, 1997). The particles are round and glassy and generally smaller than those of cement. There are two major types of fly ash based on chemical composition: Class F and Class C. Class F fly ash is created by burning anthracite or bituminous coal and is used to moderate heat gain during curing and to provide sulfide and sulfate resistance. Class C fly ash is from burning lignite or sub-bituminous coal and is used in situations where high early strength is important and for soil stabilization (IGS, 2003). The chemical and physical requirements of Class F and Class C fly ash are listed in Table 2.

.Generally, fly ash is used as a partial replacement for concrete because of its pozzolanic properties. Pozzolans are siliceous and aluminous materials that, although they alone have no cementitious properties, will, when found in very small particles and when mixed with water, form compounds with cementitious properties by reacting with calcium hydroxide (Joshi, 1997). The pozzolonic properties of fly ash are important because Florida allows 18 to 50% maximum replacement of PC by fly ash (Keck, 1997).

When added to a PC concrete mix fly ash has numerous effects on the strength and properties of concrete. Much of the effect that fly ash has on concrete is due to the increase in durability, strength and flexibility that is imparted on the concrete when fly ash is added (Owens, 1985). Generally, fly ash increases workability, decreases segregation and bleeding, retards setting time, decreases air entrainment, and lowers temperature and rate of hydration (Leshchinsky, 1991). Fly ash has a more dense matrix than PC, so when added to concrete mix a product is created that is less porous and permeable and therefore more resistant to chemically or biologically erosive materials (Anderson, 2002). Finally, fly ash inhibits the destructive alkali silica reactions in PC concrete. This process will be further explained in the following section.

When PC is replaced, in equal mass, by fly ash, the concrete is expected to have a lower compressive strength up to 28 days (Ellis, 1992), but as time passes, the pozzolanic properties begin to take effect and considerable strength improvement should be seen (Babu, 1996) (Figure 2). The reason for the late strength development is that the pozzolonic reactions take longer than the PC reactions. Finally, concrete compressive strength will decrease as the replacement percentages of the PC, with fly ash, increase (Hamernik, 1991).

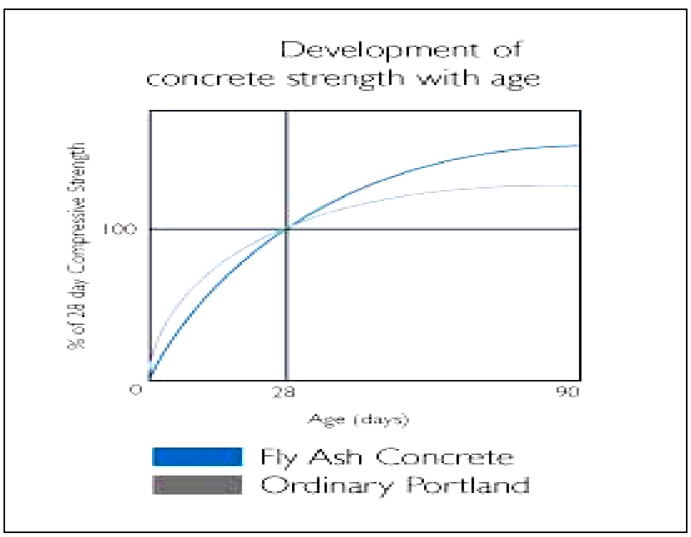


Figure 2: Fly ash versus ordinary PC
(Babu, 1996)

Table 2 Requirements for Class F and Class C fly ash in PC concrete

| ASTM C 618 – Class F and C | | | |
|---|---|---|---|
| **Chemical Requirements** | | **Class F** | **Class C** |
| $SiO_2 + Al_2O_3 + Fe_2O_3$ | Min. % | 70 | 50 |
| $SiO_3$ | Max. % | 5 | 5 |
| Moisture Content | Max. % | 3 | 3 |
| Loss on ignition (LOI) | Max. % | 6 | 6 |
| **Optional Chemical Requirements** | | **Class F** | **Class C** |
| Available alkalies | Max. % | 1.5 | 1.5 |
| **Physical Requirements** | | **Class F** | **Class C** |
| Fineness (+325 Mesh) | Max. % | 34 | 34 |
| Pozzolanic activity/cement (7 days) | Min. % | 75 | 75 |
| Pozzolanic activity/cement (28 days) | Min. % | 75 | 75 |
| Water requirement | Max. % | 105 | 105 |
| Autoclave expansion | Max. % | 0.8 | 0.8 |
| Uniform requirements[1]: density | Max. % | 5 | 5 |
| Uniform requirements[1]: Fineness | Max. % | 5 | 5 |
| **Optional Physical Requirements** | | **Class F** | **Class C** |
| Multiple factor (LOI x fineness) | | 255 | -- |
| Increase in drying shrinkage | Max. % | 0.03 | 0.03 |
| Uniformity requirements: Air entraining agent | Max. % | 20 | 20 |
| Cement/Alkali Reaction: Mortar expansion (14 days) | Max. % | 0.02 | -- |
| The density and fineness of individual samples shall not vary from the average established by the 10 preceding tests, or by all preceding tests if the number is less than 10, by more than the maximum percentages indicated. | | | |

## Alkali Silica Reaction

During the hydration of PC a very alkaline (pH of approximately 13.5) solution is formed (USDOT, 1997) of sodium and potassium oxides (alkalis). These alkalis are able to dissolve the siliceous rock in PC concrete. The reaction takes place when the silicon-oxygen bond is broken on the surfaces of siliceous aggregate. This alkali silica reaction (ASR) forms an alkali-silica gel, a “poorly defined colloidal isotropic coagulate, with exceptional swelling capability which may cause expansion and cracking in concrete” (Idord and Roy, 1985). The original siliceous aggregate is smaller than the resulting alkali-silica gel and is accommodated through formation of cracks in the binder and filler. The presence of alkali silica gel does not necessarily indicate significant changes to the

PC concrete structure, but if there is cracking there may be severe damage to the physical make-up and the strength of PC concrete. When cracks are formed they may permit more moisture to enter the internal structure in turn allowing for more of the siliceous rock to react with alkalis and therefore increasing damage to the PC concrete. The cracks may furthermore act as reservoirs for more accumulation of the alkali silica gel (Hollis, 1997). This ASR will continue as long as alkalis, reactive aggregate and water are available.

Many factors affect the extent of swelling and disruption caused by ASR in concrete. The nature and amounts of reactive aggregate is important in determining how much swelling will occur (Smith and Raba, 1986). For example, PC cement is considered to be relatively safe from ASR when the alkalis comprise less than 0.06% of the binder (USDOT, 1997). Finely ground siliceous materials can be more prone to this reaction due to the increased surface area. Finally, environmental factors such as an increase in temperature may increase the rate of the ASR, in turn causing an increased rate of expansion and potentially damage to the PC cement (Swamy and Al-Asali, 1986).

As reported in the previous section, fly ash has been found to inhibit ASR. This ASR inhibiting property is an important reason to use fly ash in PC concrete. Alkalies will preferentially bond with silica in fly ash, rather than in aggregate (Halstead, 1986) because the fly ash is silica-rich and react with the alkali-hydroxide that is formed when PC and water are mixed (Barringer, 1997).

## Temperature Effects on Concrete

Temperature can have a large effect on the strength of concrete. Extremely high temperatures are known to decrease the compressive strength of concrete. The rate of hydration, thermal stresses causes a tendency for drying shrinkage and cracking increases while permeability, strength and durability are decreased (Schindler and McCullough, 2002). Concretes cured at higher temperatures are seen to have higher initial strength, but lower strength in the long term (VTRC, 1998). This fact is important in warm climates, such as Florida, where an increase in curing temperature decreases the final strength of concrete (Jalali and Abyaneh, 1995).

Concrete additives should further affect the effect of temperature on concrete. For example, when set retarding admixtures and air-entrainers are added to the mix, concrete keeps approximately the same strength as if the curing temperature was not increased (Mittelacher, 1985 and Naik, 1985, respectively). Modest amounts of extra cement, added to the mix can also make-up for the loss of strength (Gaynor, 1985). Finally, the addition of fly ash to the mix will increase the final strength of the concrete at standard pressure (Owens, 1985), and under increased pressures (Ghosh, 1996). In PC concrete where the initial strength is lowered, but the final strength is increased with the addition of fly ash, temperature (or pressure) increases during curing should shorten the setting time and further increase the final strength of the PC concrete.

## Direct Observation Techniques

Microscopic examination of thin sections of PC concrete can be used to gain important insight into the strengths and weaknesses of the PC concrete sample. Petrographic and scanning electron microscopy (SEM) examination techniques can be used to evaluate microstructure and to investigate possible causes of deterioration of concrete, cement and aggregate (Stuzman, 2001). Petrographic examination has gained worldwide acceptance as a useful tool for finding alkali-silica reactions in concrete (DePuy, 1990). SEM may also provide useful information about the occurrence of alkali silica reactions in concrete. SEM uses direct observation of surface topography to examine concrete (Sakar andSamet, 1995) where micro-fractures and silica gels are seen as evidence of ASR.

CHAPTER 3

# TEST MATERIALS

In order to investigate economic feasibility a number of tests were performed on the byproduct fines from three locations in Florida. Due to their fine particle size and difficulty in handling, the fines were granulated with sodium silicate and PC as binders, into filler or aggregate material. The hope is that these granulated fines can be used as a replacement for conventional aggregate in PC concrete applications. The process used to granulate the byproduct limestone fines is described in chapter 4. Test ~~S~~amples were prepared as 2-inch by 2-inch concrete cubes made with granulated byproduct fines, water and PC, as specified by ASTM C 109 and described in Chapter 5. These 2-inch by 2-inch cubes then were evaluated to determine their strength characteristics and physical properties. The materials used in this study are described below.

A total of 8 different types of samples were compared in the first part of this study. The filler material of each of the cubes was varied for comparison. Six of the PC cement cubes were made with the three different types of byproduct limestone fines, granulated with two different binders. As a basis for comparison, Ottawa Sand (ASTM C 109) and construction sand, from a FDOT mine in Grandin, Florida, were used as the filler in the two remaining types of cubes.

A second set of samples were prepared and tested to further understand the properties that may be used to find an economically feasible use for byproduct fines in Florida. These 2-inch by 2-inch PC concrete cubes were made with Ottawa Sand (ASTM C 109). One half of the cubes were made with class F fly ash as a replacement for 30%

of the PC (ACI, 1993). The other set of cubes were made only with PC. Both types of concrete were cured at room temperature and at 120° F.

**Byproduct Limestone Fine**

Aglime from lot 82 (Ag82) are byproduct limestone fines from a large surface mine in Lee County, Florida. These limestone fines were demonstrated to be 83 and 17% calcite and quartz, respectively. Florida Rock Industries mined the limestone (DOT No. 12-008) from the Fort Myers Mine located 26° 32’ 27” NW latitude and 81° 47’ 24” NW longitude, off of I-75 near Fort Myers, Florida (Figure 3). The limestone is Pliocene to Pleistocene in age and are part of the Tamiami Formation and Fort Thompson Formations (Figure 4).

**Byproduct Dolomitic Limestone/ Dolomite Fine (Dolomite)**

These dolomitic limestone/ dolomite fines (the dolomite) were mined at a large Gulf Hammock Mine (DOT No. 34-106, Fig. 3) in Citrus County. The dolomite was demonstrated to be 57 and 43% calcite and dolomite, respectively. The surface mine is located at 29° 15’ 28” NW latitude and 82° 42’ 53” NW longitude, off of US 98. The dolomite is Eocene in age and is part of the Avon Park Formation (Figure 4).

**Limestone Fine - AgDry**

Aglime Dry (AgDry) are limestone fines from Columbia County, Florida (Figure 3). These fines were demonstrated to be 100% calcite. The fines were produced at the Anderson-Columbia City Mine (DOT No. 29-361) near Lake City. The large surface mine is specifically located at 30° 02’ 44” NW latitude and 82° 44’ 09” NW longitude, next to Limerock Industries Mine. The fines are Eocene in age and are part of the Ocala Limestone Formation.

## Ottawa Sand

Ottawa sand is the standard sand used in the testing of PC cements. Ottawa sand is almost entirely of naturally rounded grains of nearly pure quartz and is mined in Ottawa, Illinois. Table 3 contains the characteristics for Ottawa sand.

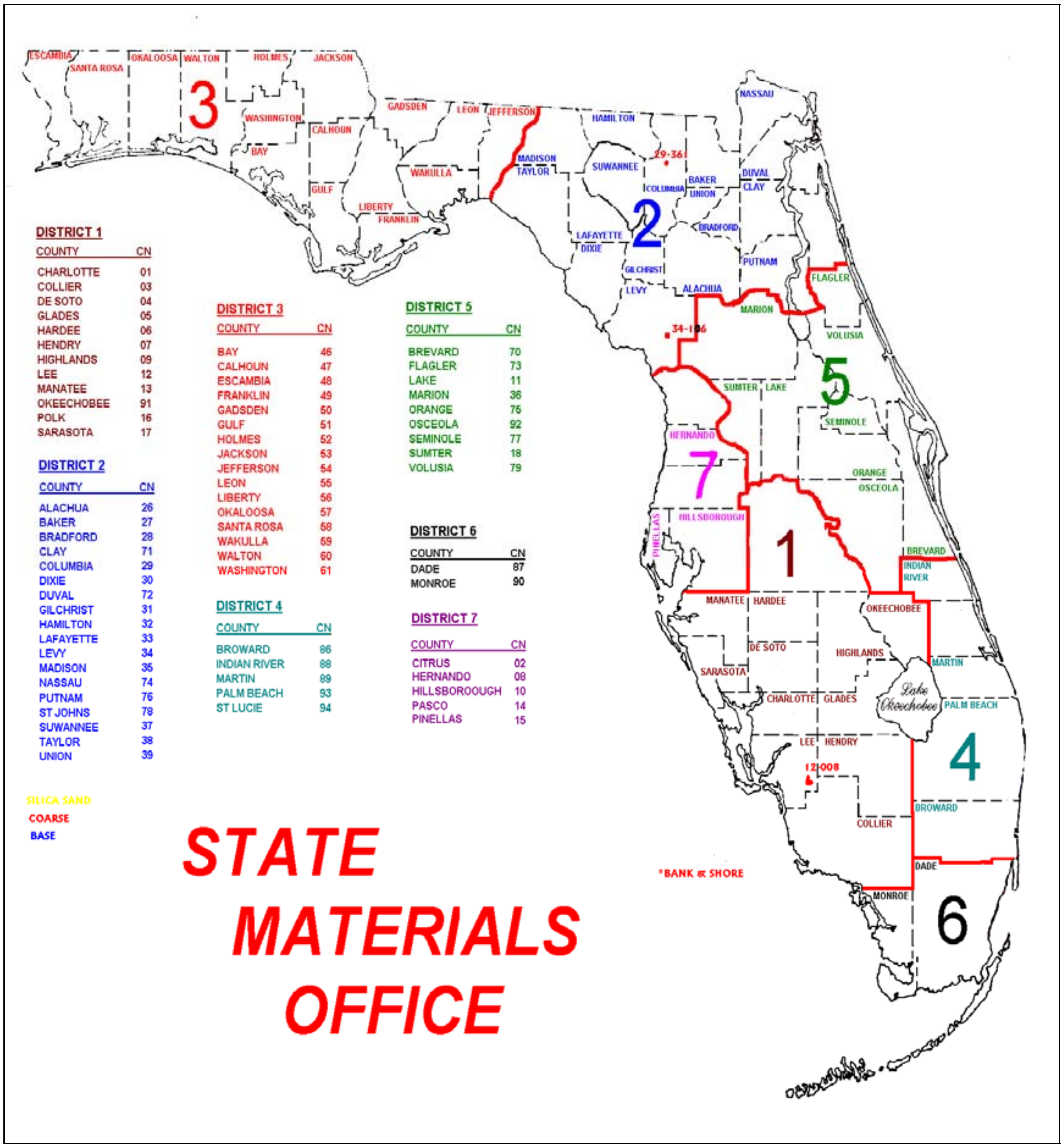


Figure 3: Location of limestone fines in Florida
Ag82 in Lee County.
Dolomite in Levy County.
AgDry in Columbia County.

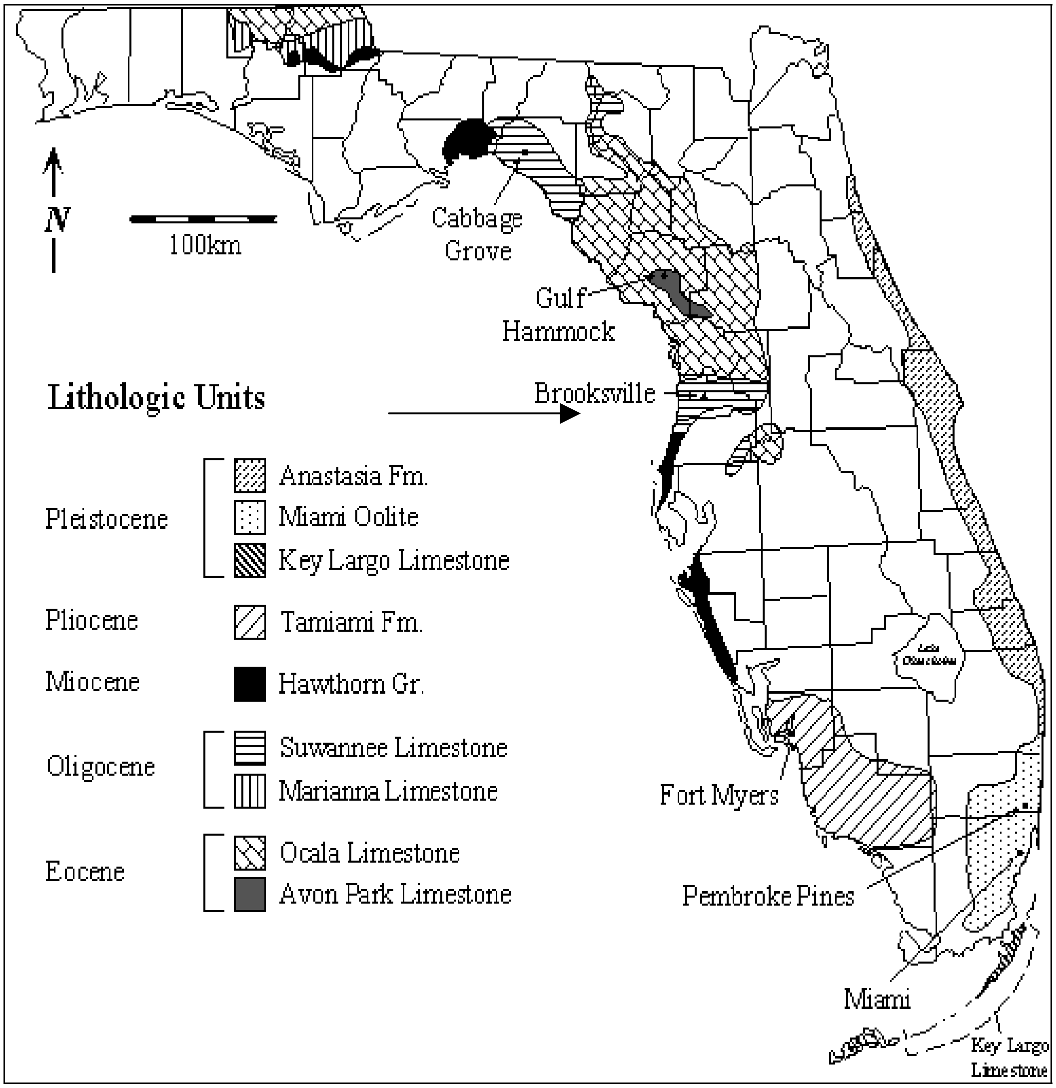


Figure 4: Exposed lithologic units of Florida

Table 3: Gradation standard sand requirements

| **Characteristics** | **Ottawa Sand** |
|---|---|
| Grading, % passing sieve: | |
| 1.18mm (No. 16) | 100 |
| 850µm (No. 20) | 100 |
| 600µm (No. 30) | 96 to 100 |
| 425µm (No. 40) | 65 to 75 |
| 300µm (No. 50) | 20 to 30 |
| 150µm (No. 010) | 0 to 4 |
| Source of sand | Ottawa, IL |

(ASTM C 778-92a)

## FDOT Sand

The DOT sand was produced at the Grandin Sand Mine in Grandin, Florida. The sand is standard DOT road construction grade sand. This sand is used in PC concrete and asphaltic concrete.

## Portland Cement

Type I/II PC was used for this report. Type I is "for use when the special properties specified for any other type are not required" (ASTM C 150-97). Type II is "for general use, more especially when moderate sulfate resistance or moderate heat of hydration is desired" (ASTM C 150-97). The requirements for the various types of Portland cement are found in ASTM C 150-97. Table 4 shows the chemical requirements characteristics of types I and II Portland cement, and the actual chemical make-up of the Type I/II Portland cement used for this project.

Table 4 Chemicals of PC

| | ASTM Requirements (max %) | | Actual (%) |
|---|---|---|---|
| **Compound** | **I and IA** | **II and IIA** | **Type I/II** |
| SiO2 | None | 20 | 22.05 |
| Al2O3 | None | 6 | 4.24 |
| Fe2O3 | None | 6 | 3.57 |
| CaO | N/A | N/A | 64.7 |
| MgO | 6 | 6 | 0.92 |
| SO3 | 3 | 3 | 2.75 |
| Na2O | N/A | N/A | 0.13 |
| K2O | N/A | N/A | 0.22 |
| TiO2 | N/A | N/A | 0.3 |
| P2O5 | N/A | N/A | 0.081 |
| Mn2O3 | N/A | N/A | 0.037 |
| SrO | N/A | N/A | 0.136 |
| Fe2O3 | N/A | N/A | 0.045 |
| Cr2O3 | N/A | N/A | 0.002 |
| Tricalcium Aluminate | None | 8 | 5.194 |
| Tricalcium Silicate | None | No | 50.949 |
| C3S | N/A | N/A | 54.353 |
| SS(C4AF+C2F) | N/A | N/A | 14.991 |
| C4AF | N/A | N/A | 10.875 |
| Ca3Sic | N/A | N/A | 58.743 |
| Equivalent Alkalies | 0.6 | 0.6 | 0.276 |

*All percentages found by FDOT by x-ray analysis.

(ASTM C 150)

# CHAPTER 4
# GRANULATION

The FDOT is interested in developing a method of converting byproduct limestone fines into a viable resource. Granulated byproduct limestone fines could potentially be used as filler in concrete applications or in flowable fill mixes in applications such as low strength backfills, slurry wall moisture barriers, vertical moisture barriers to maintain consistent moisture in swelling clays, foundation cushions, and pavement base and subbase layers (University of Florida, 2002). After a literary review, no research was found on the production concrete filler material from limestone fines.

This study focuses on evaluating the potential for use of granulated limestone fines as filler in concrete applications by strength testing and physical evaluation of 2-inch by 2-inch PC cement cubes. The Ag82, AgDry and dolomite fines were granulated into a material that were anticipated to be "hard, insoluble, and abrasion resistant" (Applied Chemical Technology, Inc., 1998). Applied Chemical Technology, Inc., Florence, Alabama granulated the three limestone fines using either sodium silicate or PC as the binding agents in a pilot-scale pan granulator.

An operating pug mill mixer/granulator was used to create the agglomerated product. Raw materials were added to a pugmill and water was evenly distributed over the powder mixture resulting granulation. The system was equilibrated so the byproduct fines were added to the system at a rate such that a constant product of granulated fines exits the system. The minimum amount of binder necessary and moisture level required for granule formation varies according to the raw material's physical and chemical characteristics, and therefore was calculated in beaker studies. All granulated materials were screened to 0.028 to 0.374 inches in diameter before being used as filler in the PC cement cubes.

## Portland Cement

Portland cement was used as one of the binders for the limestone fines. PC binder produced an average crush strength of 6.98 pounds for Ag82 and greater than 20 pounds for AgDry and Dolomite (Applied Chemical Technology, Inc., 1998). The different types of granules were tested in this study to test their strength in a PC cement mix. Table 5 shows the varying ingredient amounts and resulting moisture content and binder percentages for each of the limestone granules.

Sixty pounds of Ag82, 8 pounds of PC, and 0.79 gallons water were added to the pugmill to produce Ag82 filler granulated with the PC binder. After granulating for approximately 20 minutes until a desired size was reached, and drying at ambient conditions for 24 hours a final product was produced with a moisture content of 0.6% and approximately 11.7% PC binder.

Table 5 Product evaluation

| Limestone Type | Binder | Binder (%) | Crush Strength (lb) | Drying Temperature (°F) | Sodium (%) | Moisture (%) |
|---|---|---|---|---|---|---|
| Ag82 | Portland Cement (PC) | 11.7 | 2.36 | Ambient | N/A | 0.6 |
| Ag82 | Sodium Silicate (SS) | 6.5 | 6.98 | 276 | 0.44 | 1.9 |
| AgDry | Portland Cement (PC) | 16.1 | 20.50 | Ambient | N/A | 5.8 |
| AgDry | Sodium Silicate (SS) | 6.5 | 13.40 | 276 | 0.55 | 0.8 |
| Dolomite | Portland Cement (PC) | 16.7 | 21.15 | Ambient | N/A | 8 |
| Dolomite | Sodium Silicate (SS) | 5.3 | 7.55 | 276 | 0.55 | 1.1 |

(Applied Chemical Technology, Inc., 1998)

Sixty pounds of AgDry, 11.5 pounds of PC, and 1.45 gallons water were added to the pugmill to produce AgDry filler granulated with the PC binder. After granulating for approximately 20 minutes until a desired size was reached, and drying at ambient conditions for 24 hours a final product was produced with a moisture content of 5.8% and approximately 16.1% PC binder.

Sixty pounds of dolomite, 12 pounds of PC, and 1.19 gallons water were added to the pugmill to produce dolomite filler granulated with the PC binder. After granulating for approximately 20 minutes until a desired size was reached, and drying at ambient conditions for 24 hours a final product was produced with a moisture content of 8.0% and approximately 16.7% PC binder.

**Sodium Silicate**

Sodium silicate (water glass) was also used as a binder for the limestone fines. On average, the crushing strength of the granules made with sodium silicate was 11.4 pounds for Ag82, AgDry and the Dolomite (Table 4).

Fifty pounds of Ag82 and 6 pounds of 58% sodium silicate solution were added to the pugmill to produce Ag82 filler granulated with the sodium silicate binder. No water was added to this mixture due to the 15.2% moisture content of the raw fines. After granulating for approximately 15 minutes, and drying at approximately 276°F, a final product was produced with a moisture content of 1.9% and approximately 6.5% sodium silicate binder. Multiple passes through a vibratory fluid-bed were necessary for complete drying.

Twenty-five pounds of the AgDry and 3 pounds of 58% sodium silicate solution were added to the pugmill to produce AgDry filler granulated with the sodium silicate binder. Approximately 0.66 gallons water was added to this mixture to add to the 0.2% moisture content of the raw fines. After granulating for approximately 20 minutes and drying at approximately 276 °F, a final product was produced with a moisture content of 0.8% and approximately 6.5% sodium silicate binder.

Seventy-three pounds of the dolomite and 7 pounds of 58% sodium silicate solution were added to the pugmill to produce dolomite filler granulated with the sodium silicate binder. No water was added to this mixture due to the 17.5% moisture content of the raw fines. After granulating for approximately 20 minutes and drying at approximately 276°F, a final product was produced with a moisture content of 1.1% and approximately 5.3% sodium silicate binder.

CHAPTER 5
METHODOLOGY

## Preparation of 2-inch by 2-inch Specimens

This study compares the compressive strength of eight types of PC concrete. These PC concretes are made with varying filler, listed as follows:

1. Ottawa Sand (OS)
2. DOT Sand (DOT sand)
3. Dolomite with sodium silicate binder (Dol SS)
4. Dolomite with PC binder (Dol PC)
5. Ag82 with sodium silicate binder (Ag82 SS)
6. Ag82 with PC binder (Ag82 PC)
7. AgDry with sodium silicate binder (AgDry SS)
8. AgDry with PC binder (AgDry PC)

Following ASTM C 109 procedures, each type of PC mortar was made into a 2-inch by 2-inch cube in preparation compressive strength testing. The procedures begin by measuring out the correct amount of cement, sand, water, and fly ash. ASTM C 109 specifies 740 grams of cement for every 9 cubes. The volume of filler aggregate is also specified for Ottawa sand in ASTM C 109. This volume of filler aggregate was adopted for the various fillers, tested in this study.

The three types of granulated fines were screened for 15 minutes. The minus plus-4 and minus 40 sieve sizes were discarded. The granulated fines in the minus-4 and plus-40 mesh size were used as fine aggregate for this experiment.

The water required for Ottawa sand is specified is ASTM C 109. The water used for the different fine aggregates varied according to volume of voids per 0.264 gallons (1000 milliliters (mL)) for the different materials (Table 6). The procedure for determining this amount was done by filling a cylindrical 1000 mL tube with filler aggregate and filling the tube to 1000 mL with water. The volume of water needed to fill the tube to 1000 mL, with the filler aggregate in place, was used as the volume of water for the mortar mix of the corresponding aggregate and referred to as the volume of voids. This procedure was developed with Dr. Eades from the University of Florida Department of Geological Science as recommended by technicians with Florida Crushed Stone Company, Brooksville, Florida.

The PC cement, filler aggregate and water were then mechanically mixed following ASTM C 305-94 procedures. Generalized procedures begin by first adding the water to the bowl. Second, the cement was added and mixed on low speed for 30 seconds and medium speed for 30 seconds. The mix then stood for 1.5 minutes, while the mixture was scraped from the sides of the bowl in the first 15 seconds. After scraping and standing, the batch was mixed on medium speed for 1 minute.

Table 6 Amount of water added to cubes

| Type of Sand | Amount of Water added to mix to make 9 cubes in gallons (and in mL) | Water to Cement Ratio |
|---|---|---|
| Ottawa Sand | 0.095 (359) | 0.485 |
| DOT Sand | 0.090 (340) | 0.459 |
| Dol SS | 0.163 (616) | 0.832 |
| Dol PC | 0.146 (553) | 0.747 |
| Ag82 SS | 0.139 (525) | 0.709 |
| Ag82 PC | 0.135 (512) | 0.692 |
| AgDry SS | 0.151 (570) | 0.770 |
| AgDry PC | 0.133 (504) | 0.681 |

As specified by ASTM C109, after completing the mixing stages no more than 2 minutes and 30 seconds passed before beginning to mold the specimens. As recommended by technicians with Florida Crushed Stone Company, Brooksville, FL, a lubricant (WD-40) was sprayed on all surfaces of the mold that would be in contact with the PC mix to ensure the cubes could be easily removed from the molds. Cubes were molded one at a time, as specified in ASTM C 109, by first adding approximately one inch of mortar to the mold and tamping 32 times in approximately 10 seconds. Tamper specifications are outlined in ASTM C 109. The remaining portion of the cube was then filled and tamped in the same pattern. This procedure was repeated for each cube and the excess mortar was removed with a trowel to ensure a flat top surface of the cube.

Finally, the cubes were placed in a moist cabinet or moist room for the next 24 hours, as required by ASTM C511-96. For this experiment, a cooler with standing water was used and assumed to meet the specifications of ASTM C511-96. After 24 hours the cubes were removed from the moist area in order to begin curing.

**Curing the Cubes**

Curing involved completely submersing the cubes in lime-saturated water (ASTM C 109). Lime-saturated water was created by filling flat, covered, tanks with water, and allowing free access to a source of lime (CaC(OH)). The water was replaced as it became murky, and replenished as it evaporated.

Some of the samples were cured at a temperature of 120° F. Elevated temperatures for curing were created by storing the tank of lime-saturated water in an oven, wrapped in a plastic bag. The temperature and water level were closely monitored to be sure they remained nearly constant.

Cubes were cured for 3, 7, 14, or 28 days in the lime-saturated water. At the end of its specified curing time each cube was carefully removed and placed in a plastic bag to keep it moist, until it was time to test that cube.

## Unconfined Compressive Strength Testing

A number of steps were taken to try to minimize errors in the final results. To reduce errors in specimen preparation cubes were made and broken in triplicate. In addition, the cubes for the test program were made and tested in a random order to minimize any bias that might have occurred. A number of "practice" cubes were created, cured and broken to eliminate a "learning curve" which could affect the results.

Loads were applied according to ASTM C 109. The cubes were broken on a Tinius Olsen Testing Machine (Figures 5, 6 and 7) that allows the speed at which the load is applied to vary. This tactic enables the operator to adjust the load period. The first period of loading, (time necessary to complete loading to one half of the maximum load) may occur in any amount of time. The second period of loading (remaining period of time necessary to load the specimen to its maximum load) must occur in not lest than 20 seconds nor more than 80 seconds after the first period of loading is finished. The correct timing of loading was necessary and was successfully executed through expertise and experience of the operator. After testing, each cube was removed from the machine. Each sample was then digitally photographed alone, and beside the other samples with the same composition and curing period.

## Thin Section Preparation

A thin section of concrete must be created to have a useful tool for petrographic and SEM analysis. The generalized techniques used to make a thin section are as follows (Ahmed, 1994; Stuzman, 2001):

1. the pore solution is replaced with ethanol,
2. the ethanol is replaced with epoxy,
3. the epoxy is cured

4. the sample is cut using a diamond blade or a wafering saw that is lubricated with porpylene glycol,

5. the sample is polished with successively finer particle size diamond polishing pastes from 9 μm to 0.25 μm and a lap wheel to remove evidence of cutting and grinding, and

6. the sample is coated with carbon or metal.

Figure 5: Tinius Olsen testing machine

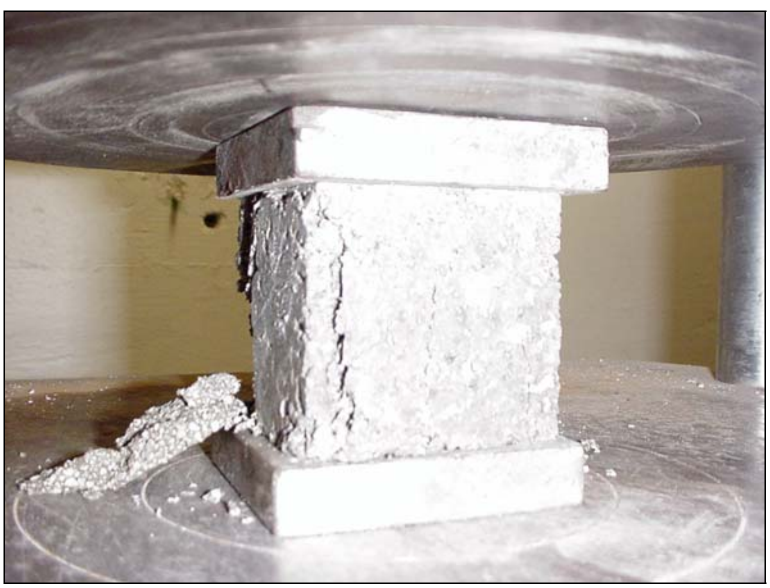

Figure 6: Cube in testing machine

Figure 7: Typical cube, in the process of breaking

Right side shows the hourglass shape that was produced when all of the cubes were broken.

# CHAPTER 6
# RESULTS

## Granulated Samples

Unconfined compressive strength was measured for the 8 types of PC concrete cubes made with various fillers (as listed in Chapter 5). Table 7 lists the measured strength for the 8 types of PC concrete cubes according to the run (run in triplicate) and the number of days of curing before testing was run (3,7,14 or 28 days). The cubes produced a wide range of strength exhibiting values between 500 and 7000 pounds per square inch (psi) of unconfined compressive strength. The data for anomalously high or low values were considered to be invalid and were removed from the resulting data set and are seen as blanks in table 7. The data from the three runs, for each of the 8 types of PC concrete were averaged to produce a single value of compressive strength (Table 8).

The unconfined compressive strengths as a function of curing time of the 8 types of PC concrete cubes with varying fine aggregate can easily be compared (Figure 7). The cubes prepared with FDOT sand showed the greatest strength values followed by the cubes prepared with Ottawa sand. The three next strongest concrete mixes were Ag82, AgDry, and Dolomite (strongest to weakest), all granulated with PC. The weakest concretes were AgDry, Dolomite, and Ag82, (strongest to weakest) pelletized with sodium silicate. Figure 7 displays a log derived trend line for the results for each of the 8 types of PC concrete cubes to aid in the visual comparison of the different unconfined compressive strengths over time.

Figure 8 shows a graph of water content ratio versus unconfined compressive strength. This graph generally shows a decrease in compressive strength with an increase in water: cement ratio.

**Heated and Room Temperature Samples; With and Without Fly Ash**

This portion of the study evaluates the effects of adding of fly ash to cement mix and elevating curing temperatures on the cement cubes made with and without fly ash. The use of fly ash as a partial replacement for the PC in a cement mix can create a less expensive and stronger resulting product. Samples were prepared with and without fly ash to compare the relative strength of PC concrete with and without fly ash.

Temperature during curing affects concrete strength. Samples were cured at room temperature and at an elevated temperature of 120° F to reproduce the effects of curing in hot versus temperate climates. The effects of adding fly ash and curing at different temperatures can then be cross evaluated to see the relative compressive strength changes from adding, or not adding, fly ash to PC cement that is cured at room temperature and at elevated temperatures

All of the concrete cubes of this portion of were made with Ottawa sand as the filler. One half of the samples were made with class F fly ash as a replacement for 30% of the PC and the other half of the samples were made only with PC. One half of the samples made with, and one half of the samples made without fly ash were cured at room temperature for 3, 7, 14 and 28 days. The remaining half of the samples made with, and the remaining half of the samples made without fly ash were cured at an elevated temperature of 120° F for 3, 7, 14 and 28 days. Each set of samples were run in triplicate to reduce resulting preparation variable and randomize errors created during data collection.

Table 7 Unconfined compressive strength of each cube in psi

| Run Number | Days Cured | OS | DOT sand | Dol PC | Ag82 PC | AgDry PC | Dol SS | Ag82 SS | AgDry SS |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 3 | 3413 | 3600 | 1738 | 2475 | 2500 | 575 | 913 | 980 |
| | 7 | 3538 | 5838 | 3075 | 3375 | 3175 | 1213 | 1200 | 1025 |
| | 14 | 4200 | 7288 | 2763 | 4088 | 2913 | 1313 | 1498 | 1513 |
| | 28 | 4763 | | 2788 | 4050 | 3800 | 1525 | 1313 | |
| | | | | | | | | | |
| 2 | 3 | 3450 | 3688 | 1550 | 3263 | 2063 | 600 | 1038 | 890 |
| | 7 | 3488 | 5888 | 2450 | 3450 | 3313 | 988 | 888 | 1163 |
| | 14 | 3713 | 7038 | | 3950 | 2738 | 1138 | | 1450 |
| | 28 | 4400 | 7063 | 3238 | 4100 | 2813 | 1230 | 838 | 1550 |
| | | | | | | | | | |
| 3 | 3 | 3475 | 4450 | 1913 | 2563 | 1975 | 585 | 900 | 995 |
| | 7 | 3338 | 5688 | 2775 | 4088 | 3075 | 988 | 800 | 1075 |
| | 14 | 4125 | 7700 | 3113 | 4013 | 3188 | 1263 | 1163 | 1275 |
| | 28 | 4188 | 7250 | 2925 | 4450 | 2888 | 1540 | 763 | 1450 |

(Values that were considered to be outlying data were removed. Samples were run in triplicate (see run number))

Table 8: Average unconfined compressive strength (psi) found for each type of cube

| | Day | OS | DOT sand | Dol PC | Ag82 PC | AgDry PC | Dol SS | Ag82 SS | AgDry SS |
|---|---|---|---|---|---|---|---|---|---|
| **Average of Runs** | 3 | 3446 | 3446 | 1733 | 2767 | 2179 | 587 | 950 | 955 |
| | 7 | 3454 | 5804 | 2767 | 3638 | 3188 | 1063 | 963 | 1088 |
| | 14 | 4013 | 7342 | 2938 | 4017 | 3188 | 1238 | 1330 | 1413 |
| | 28 | 4450 | 7071 | 2983 | 4200 | 3167 | 1432 | 971 | 1429 |
| **Standard Deviation** | | 485 | 1778 | 589 | 637 | 501 | 362 | 185 | 237 |
| | **Average** | 3841 | 5916 | 2605 | 3656 | 2931 | 1080 | 1054 | 1221 |

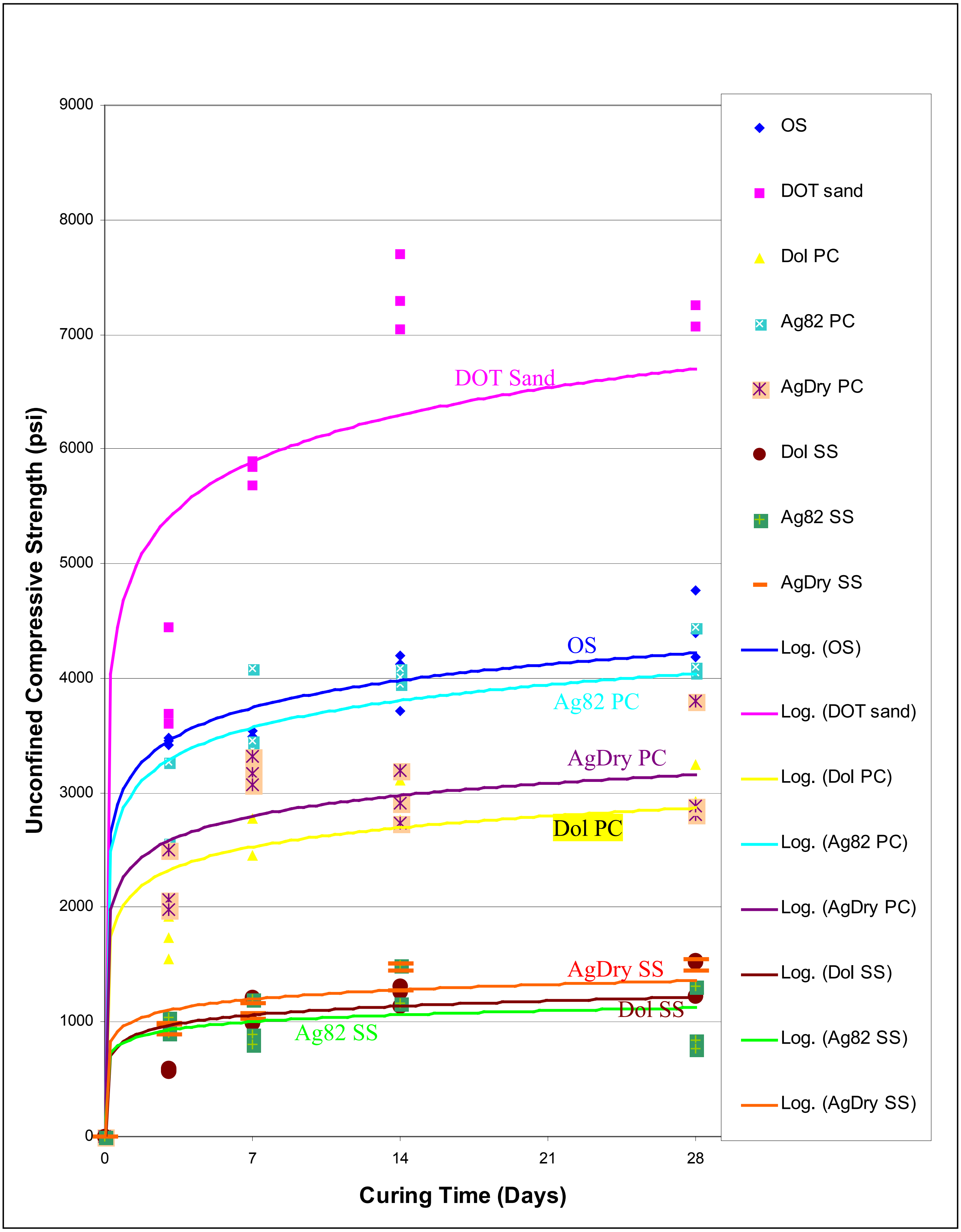


Figure 8: Unconfined compressive strength vs. curing time for PC concrete samples

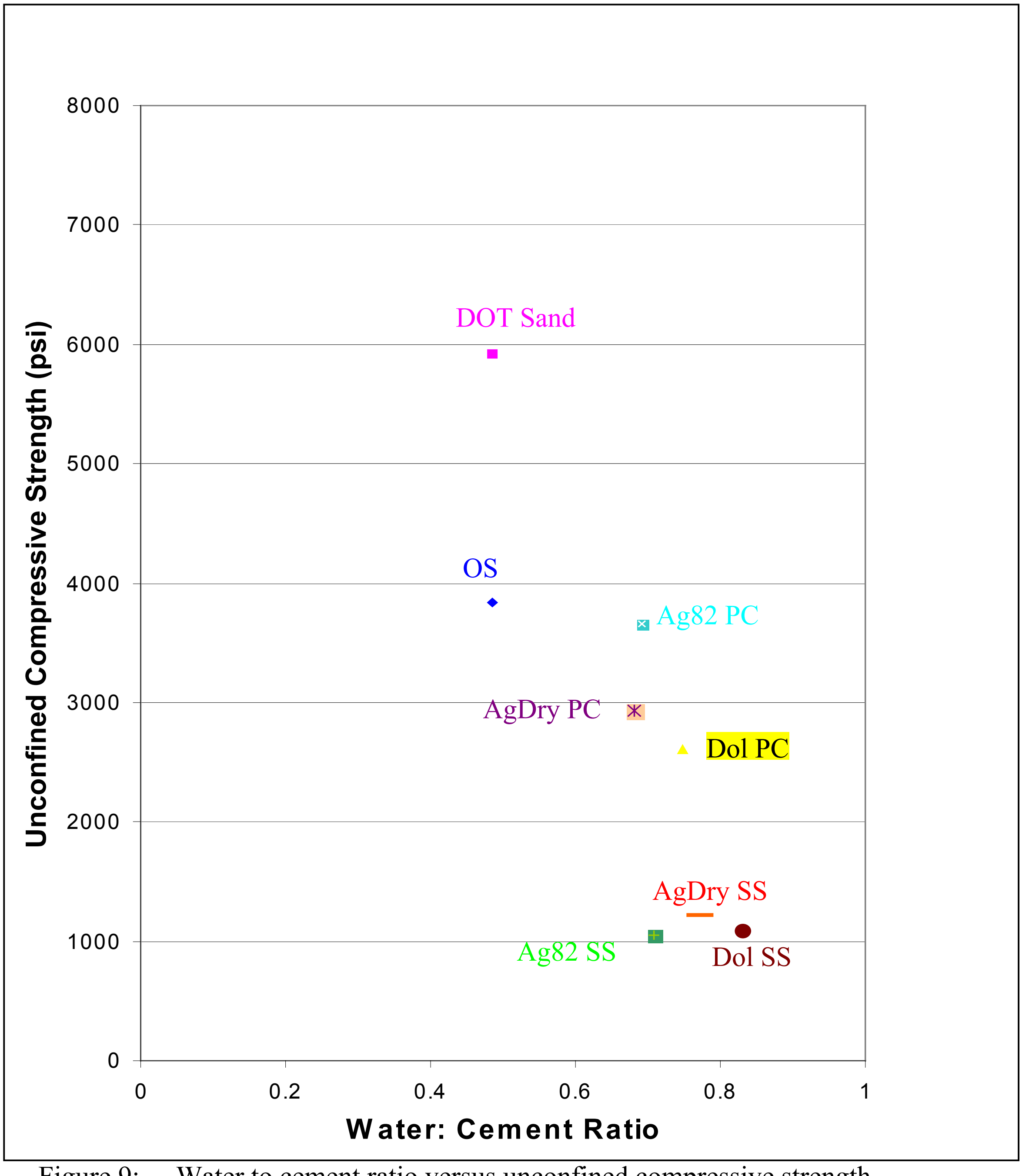


Figure 9: Water to cement ratio versus unconfined compressive strength

All of the concrete cubes of this portion of were made with Ottawa sand as the filler. One half of the samples were made with class F fly ash as a replacement for 30% of the PC and the other half of the samples were made only with PC. One half of the samples made with, and one half of the samples made without fly ash were cured at room temperature for 3, 7, 14 and 28 days. The remaining half of the samples made with, and the remaining half of the samples made without fly ash were cured at an elevated temperature of 120° F for 3, 7, 14 and 28 days. Each set of samples were run in triplicate to reduce resulting preparation variable and randomize errors created during data collection.

A value of unconfined compressive strength was found for each sample, with and without fly ash, cured at room temperature and at 120° F (Table 9). Cubes that produced extremely high or low values of strength were removed from the data set. The data were averaged to produce one value of unconfined compressive strength for each type of sample and curing period (Table 10). Unconfined compressive strength increased over curing time for all of the different cubes and testing periods, except the concrete with fly ash that was cured at high temperatures. This sample, made with fly ash and cured at a high temperature showed a decrease in unconfined compressive strength from 14 to 28 days.

A number of comparisons between samples can be made. These comparisons are broken down as follows:

**1. Room Temperature versus Elevated Temperature without Fly Ash**

Cubes made without fly ash and cured at an elevated temperature show a higher unconfined compressive strength initially (3 and 7 days), but appear to be weaker in the long run (14 and 28 days) when compared to cubes made without fly ash and cured at room temperature.

**2. Room Temperature versus Elevated Temperature with Fly Ash**

Cubes made with fly ash and cured at an elevated temperature show a higher unconfined compressive strength initially (3 and 7 days) and a significantly higher compressive strength with time (14 and 28 days) when compared to cubes made with fly ash and cured at room temperature.

**3. Fly Ash versus No Fly Ash at Room Temperature**

Cubes cured at room temperature, without fly ash, have consistently lower values of unconfined compressive strength when compared to cubes with fly ash.

**4. Fly Ash versus No Fly Ash at Elevated Temperature**

Cubes cured at an elevated temperature, without fly ash, have higher values of compressive strength initially (3 and 7 days), but a lower values of unconfined compressive strength after time passes (14 and 28 days).

Table 9: Unconfined compressive strength, in psi, of cubes with and without fly ash after curing at room or elevated temperature

| Mold Number | Days Cured | Room Temperature | | Elevated Temperature (120° F) | |
|---|---|---|---|---|---|
| | | No Fly Ash | Fly Ash | No Fly Ash | Fly Ash |
| | | 3000 | 2525 | 3650 | 3163 |
| | 7 | 4188 | 3825 | 5238 | 4900 |
| | 14 | 5188 | 4375 | 4688 | 6288 |
| | 28 | | 5200 | | 6113 |
| 2 | 3 | 2788 | 2475 | | 3338 |
| | 7 | 4313 | 3888 | 5250 | 4513 |
| | 14 | 5213 | 4438 | 5450 | 5975 |
| | 28 | 5625 | 5063 | 5063 | 5338 |
| 3 | 3 | 2763 | 2488 | 3038 | 3400 |
| | 7 | 4438 | 3963 | 5025 | 4725 |
| | 14 | 5888 | 4113 | 5125 | 5813 |
| | 28 | 5375 | 5325 | 5325 | 6163 |

(Bad data is shown as missing. Samples were run in triplicate.)

Table 10: Average unconfined compressive strength (psi) of hot and room temperature cured cubes with and without fly ash

| Days Cured | Room Temperature | | Elevated Temperature (120° F) | |
|---|---|---|---|---|
| | No Fly Ash | Fly Ash | No Fly Ash | Fly Ash |
| **3** | 2850 | 2496 | 3344 | 3300 |
| **7** | 4313 | 3892 | 5171 | 4713 |
| **14** | 5429 | 4308 | 5088 | 6025 |
| **28** | 5500 | 5196 | 5194 | 5871 |

## Petrographic Microscope

Each of the concrete samples that were made with different fillers were photographed with a petrographic microscope, for evaluation. The photographs were taken after the concrete had been broken and made into slides. A number of the pictures were selected and are shown below:

The concrete made with FDOT sand is presented in a petrographic photograph (Figure 10). This photograph creates a clear picture of the irregular shaped grain. The silica grains are smaller than the granulated grains of the other samples.

A petrographic photograph of PC concrete made with Ottawa sand seen above (Figure 11). This photograph shows the rounded grains of Ottawa sand. The particles show little variation in size and again are smaller than the granulated particles of other samples.

A petrographic photograph of PC concrete made with Ag82 PC reveals some details about the concrete (Figure 12). A crack runs through the cement paste and straight into the particle, suggesting a strong bond.

A close look at the interface between the PC concrete made with granulated AgDry PC particles is seen in Figure 13. This photograph shows a strong bond between the particles and the cement paste. A strong bond is seen in all of the concretes that were cemented with PC.

PC concrete made with Dol PC can be evaluated using petrographic photographs. Figure 14 demonstrates lower bond strength (compared to the other filllers bonded with PC) by depicting a crack running around the dolomite particle. Cracking is more common in the dolomite concrete, as compared to the other fillers that were bonded with PC.

Petrographic photographs show details about the construction of the granulated particles. Figure 15 clearly depicts the circular structure of granulated dolomite filler particle. This same pattern was seen in the granulated AgDry PC.

Fillers made with sodium silicate as the bonding paste for the granulated material showed more extensive cracking than cements made with PC as the bonding paste. Figure 16 shows a large crack running through a number of granulated AgDry SS particles. Some of the grains seemed to have reacted with the cement paste, making the particles appear to have been broken or dissolved.

The concrete made with granulated dolomite using sodium silicate is pictured above (Figure 17). The photograph shows an extensively crack-filled specimen. These dolomite samples often appeared to have reacted with the cement paste, as the AgDry SS samples were.

Petrographic photographs of Ag82 SS showed very extensive cracking in the specimens that were made with sodium silicate binded filler (Figure 18). Many voids and large cracks were seen throughout the sample.

Petrographic photographs of Ag82 SS depicted an interesting color variation. As seen in Figure 19, there is a ring around the particle. This change in color and texture could be seen in a number of the Ag82 SS particles.

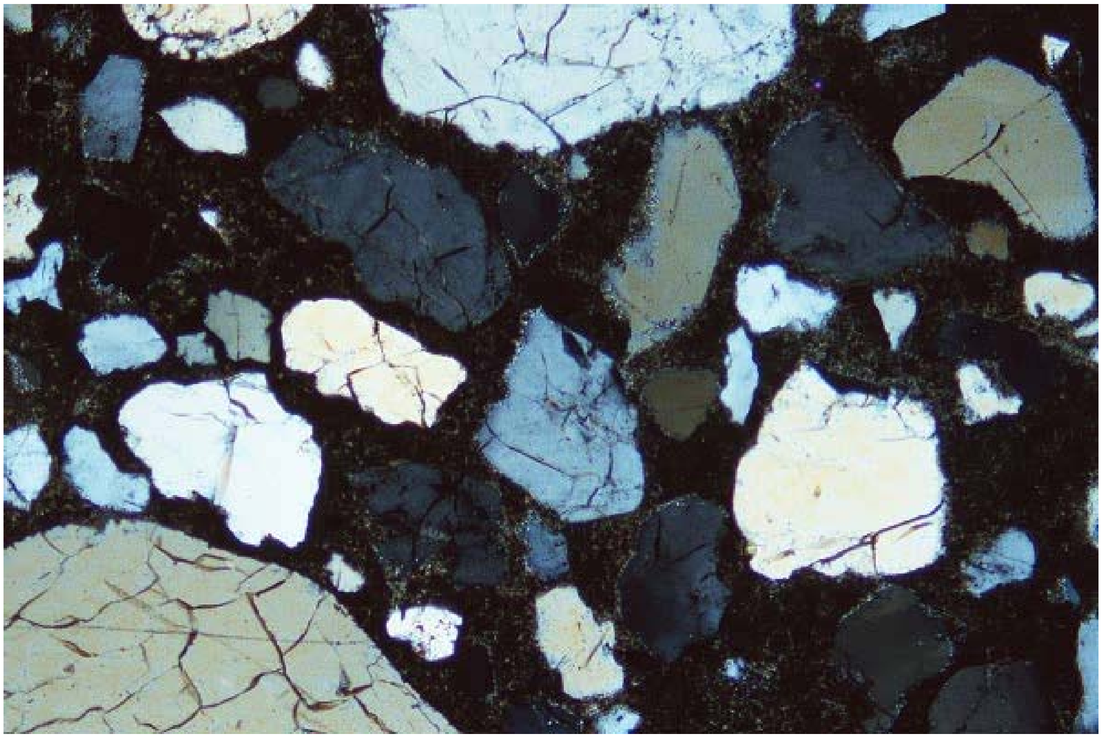

Figure 10 PC concrete made with FDOT sand PPL (FV=2.5mm)

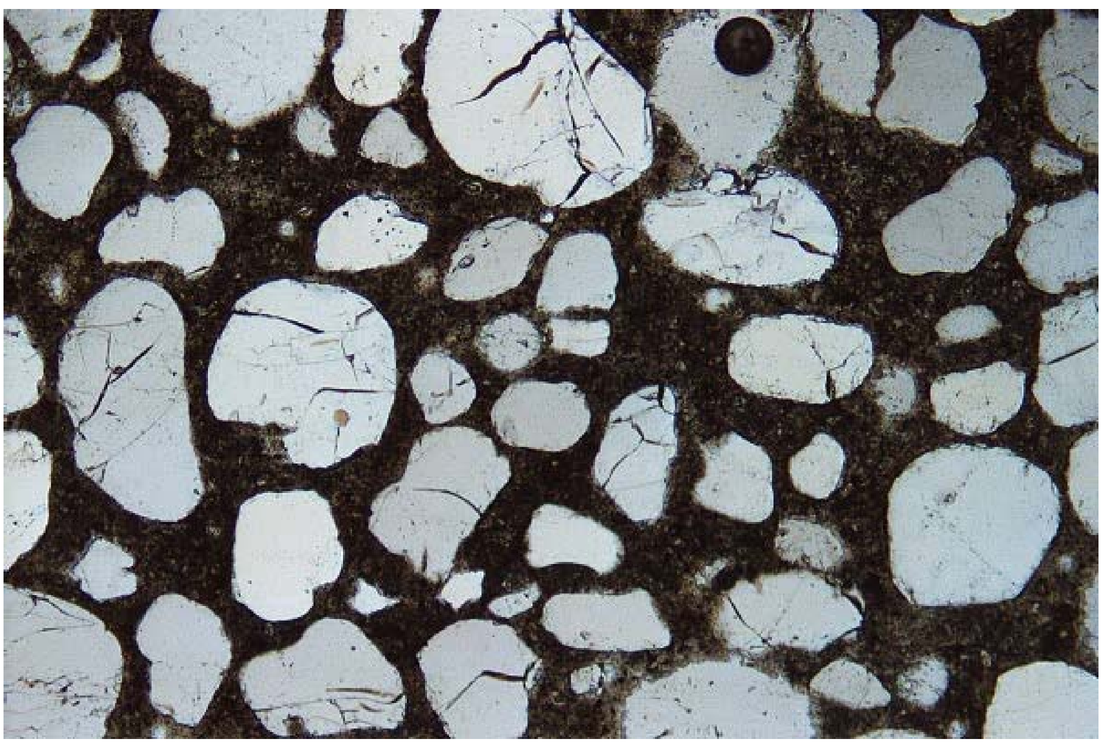

Figure 11: PC concrete Ottawa sand in plain light (FV -= 2.5mm)

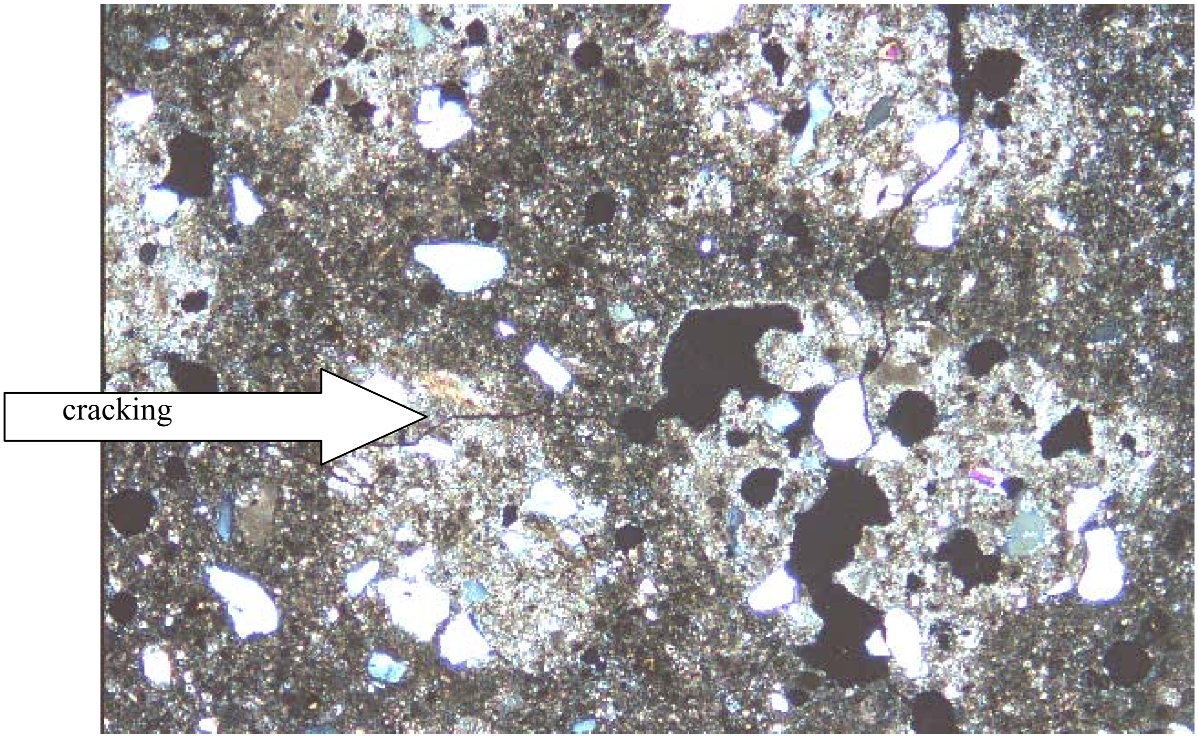


Figure 12: Ag82PC (PPL, FV=2.5mm)

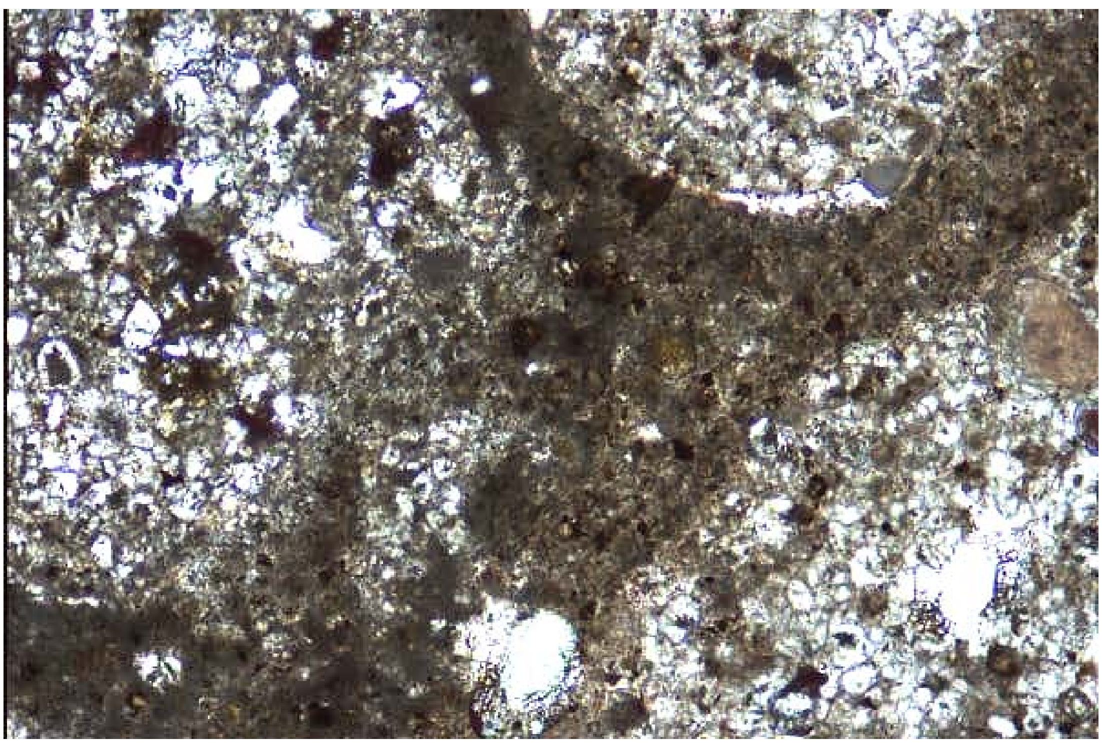

Figure 13: Interface between AgDry PC particles (PPL, FV=1.25mm)

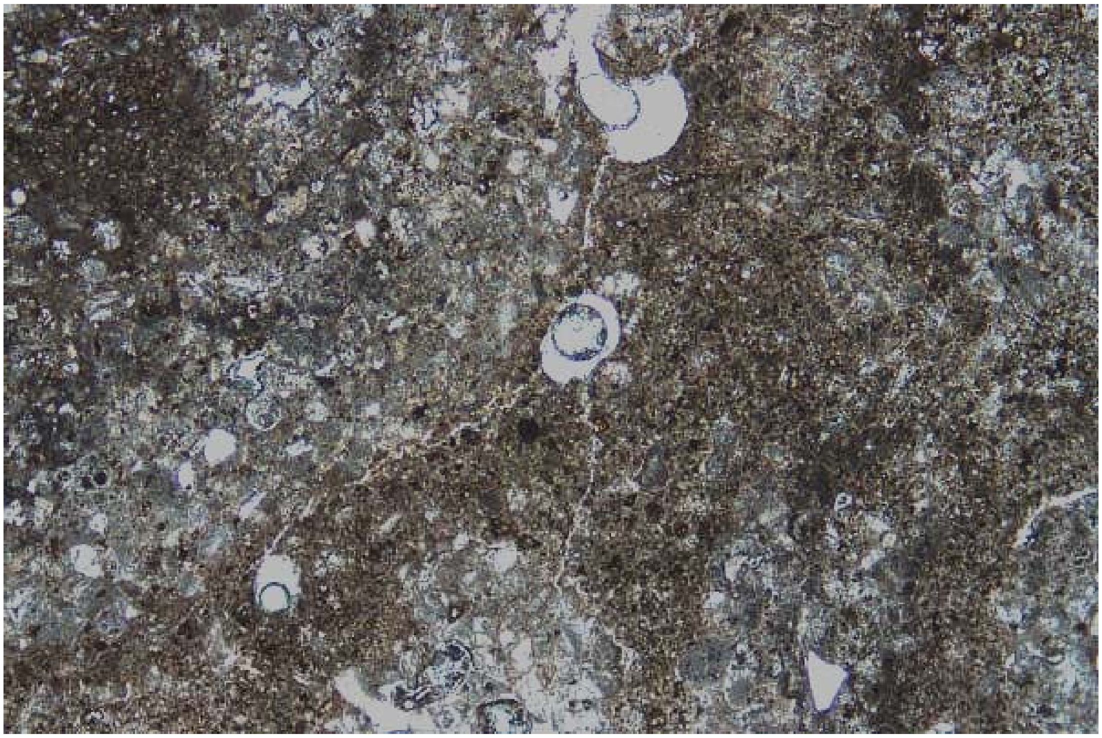

figure 14: Dol PC in plain light (FV=2.5mm)

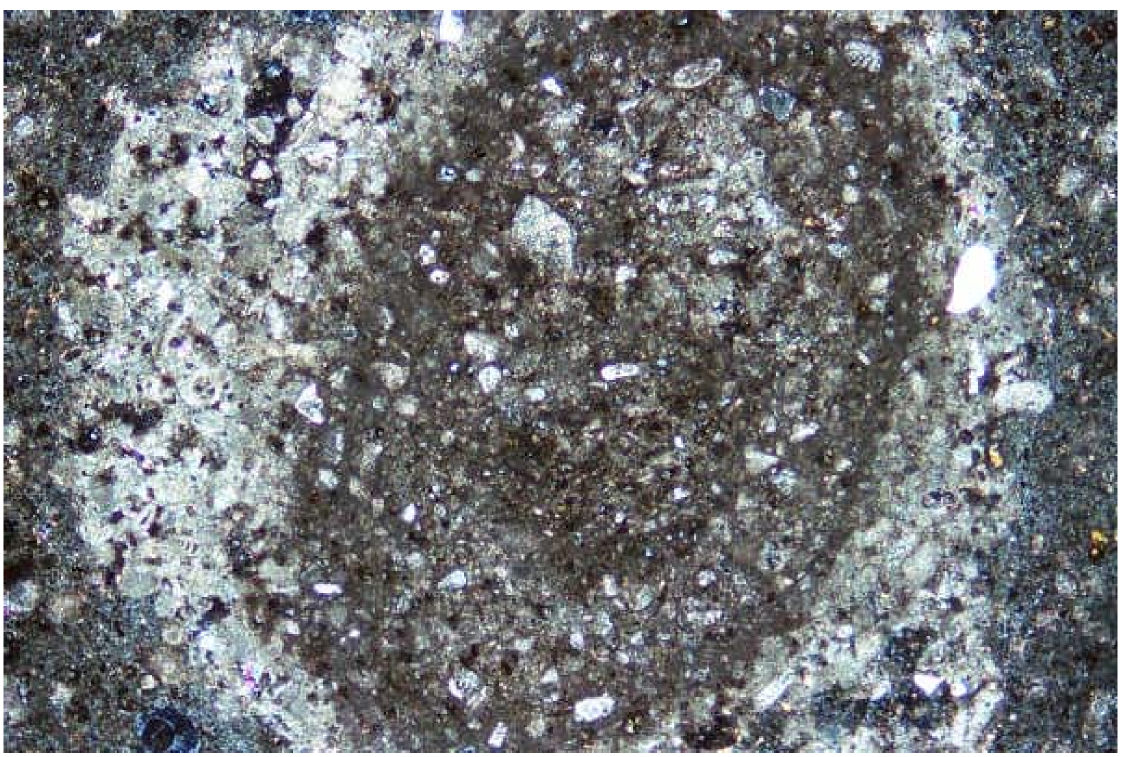

Figure 15: Dol PC (PPV, FV=2.5)

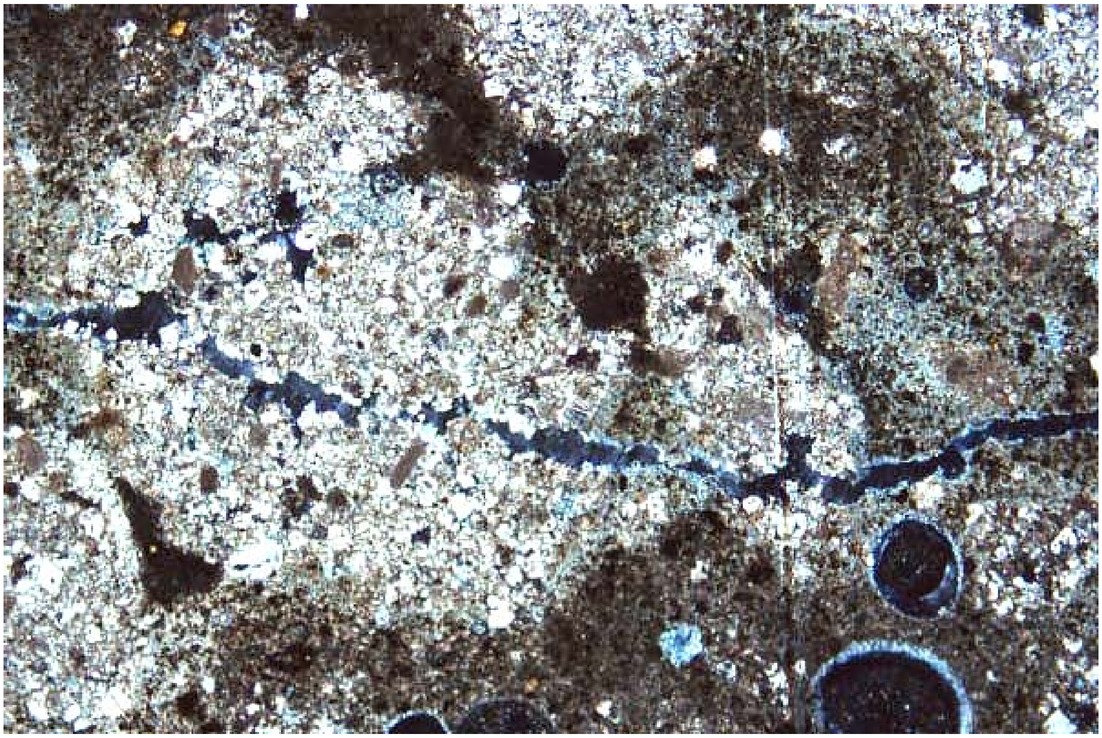

Figure 16: Granulated AgDry SS photographed in PPV (FV=2.5mm)

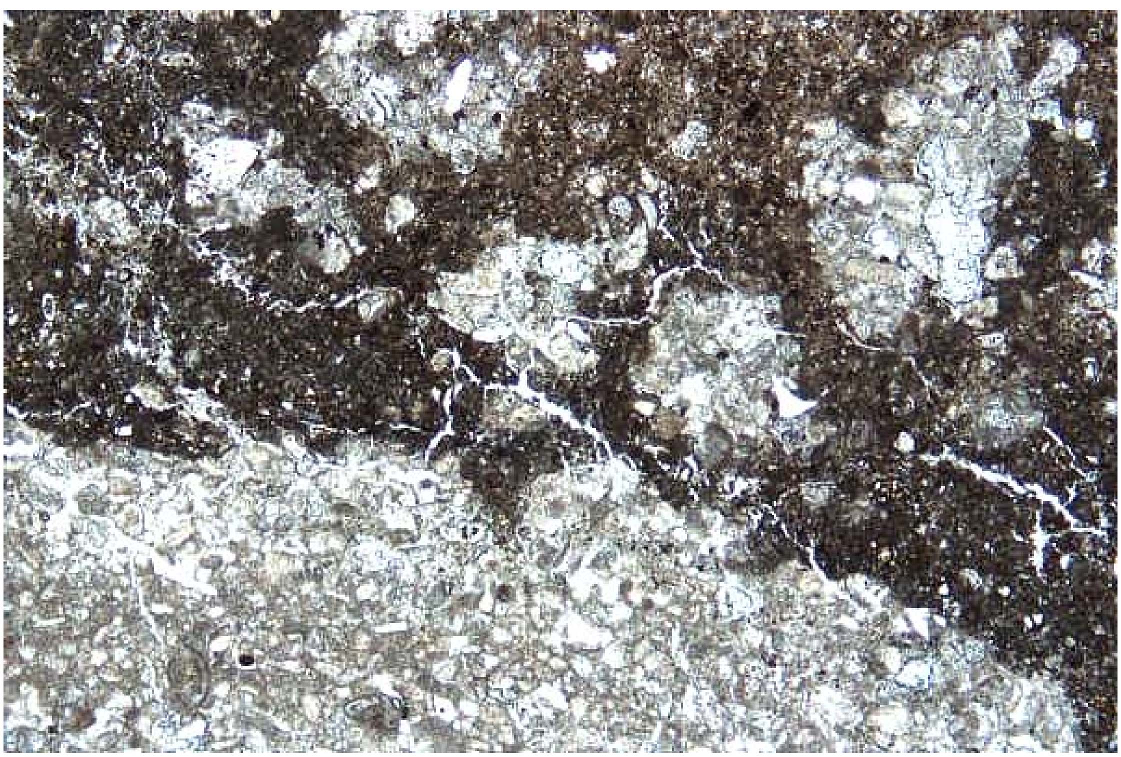

Figure 17: Dol SS in plain light (FV=2.5mm)

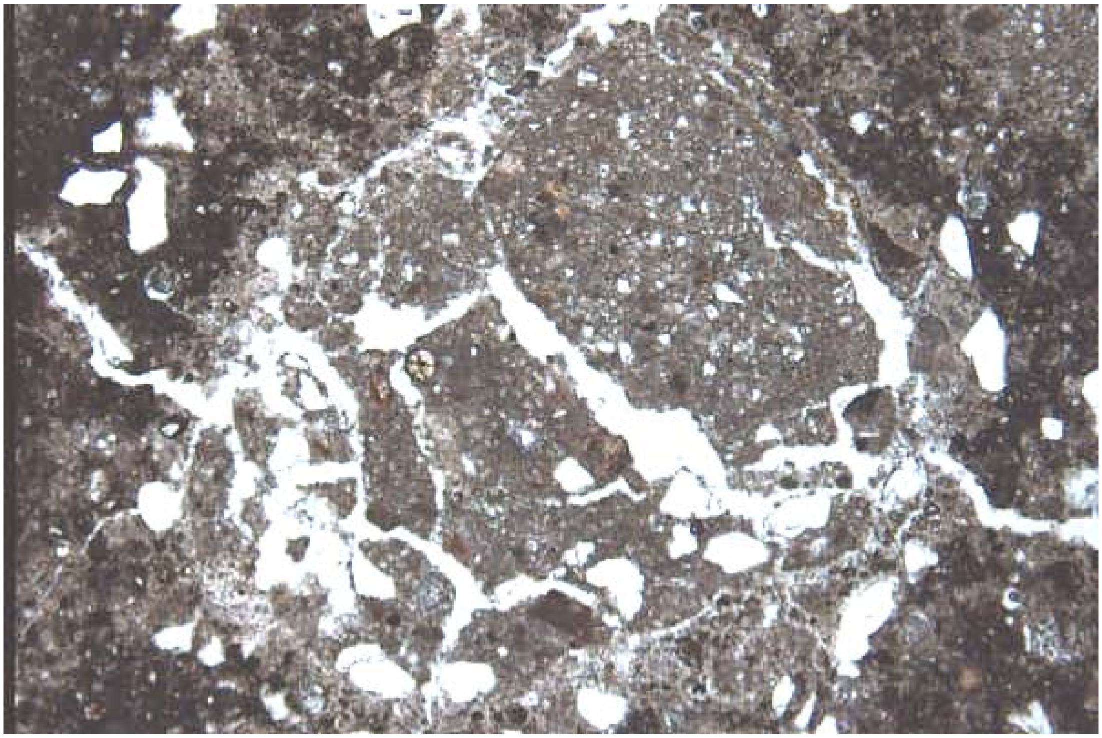

Figure 18: Granulated Ag82 SS (plain light, FV=2.5mm)

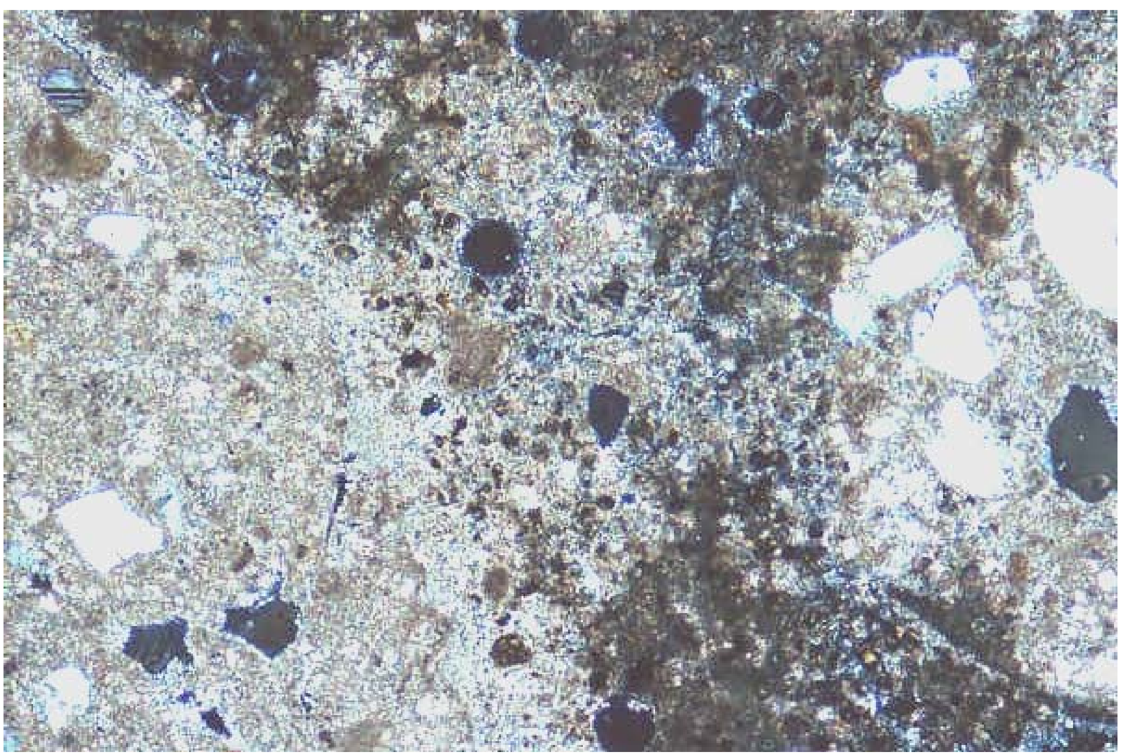

Figure 19: Ag82 SS (PPV, FV=1.25mm)

**SEM Pictures**

SEM photographs were taken of each of the PC concrete samples that were made with different types of filler. The photographs were taken after the samples were broken and made into petrographic slides. A number of the pictures were selected and are seen below.

The SEM image of the PC concrete made with FDOT sand, relates a number of facts about the physical composition of this particular concrete (Figure 20). The sand grains are not rounded, they are irregularly shaped with some sharp edges and are generally less than 0.0394 inches (1mm) in length. These sub-angular to angular, irregularly shaped, dense, silica grains impart good strength characteristics on the PC concrete cube.

The SEM image of PC concrete made with Ottawa sand imparts information on the bonding characteristics of the mixture. This picture (Figure 21) visualizes the lack of bonding between the sand grains and the PC with the defined white line between the two. The hard, dense silica particles seen to be coarser, rounder and more uniform in size and rounded shape than were the FDOT sand particles. The rounded will impart less strength to the PC concrete cube than the sub-angular to angular grains of the FDEP sand (Figure 20). A crack seems to propagate through the PC paste and along the boarder of the Ottawa sand that demonstrates the relative weakness of the PC paste as compared to the silica grain. The filler's characteristics seem to be greatest factor in determining strength of the PC concrete made with Ottawa sand and FDEP sand.

The SEM image of PC concrete made with granulated Ag82 PC granules (Figure 22) appears much different than the concrete made with pure sand particles. The granulated particles are much larger than the sand particles seen in the concrete made with FDOT and Ottawa sand. This PC concrete made with Ag82 showed the best compressive strength values from the cubes made with granulated fines and the reason for this fact is seen in the SEM images. As seen in the petrographic photograhs the particles are well cemented to the grains seen by the obscure line between the granulated particles and the PC cement is obscure. There is little space in the grain, cement paste, or in the bond between the two.

The cubes made with this AgDry PC granules did not perform as well as the Ag82 PC or silica sand cubes, but was determined to be relatively strong compared to the cubes made with fine aggregate binded with sodium silicate. Similar to the PC concrete made with Ag82 fine aggregate agglomerated with PC binder, the strength of this sample is seen in the good grain-paste bond (Figure 23), as it is difficult to determine where the line between the PC and the AgDry PC granulized fines is located. Again, the particles are much larger than the sand particles seen in the DOT and Ottawa sand mixtures. The weakness of this sample is seen in the general lack of bonded strength of the granulated grains. The granulated AgDry PC grains are weaker than the quartz grain standards and the Ag82 PC grains.

This SEM image shows the strengths and weaknesses of this PC concrete made with granulated Dol PC (Figure 24). The cracks caused by breaking the cube run straight through the cement paste into the pelletized particle. This cracking pattern is seen in each of the cubes made with PC filler and shows good bonding between the granulated filler and the PC. SEM imaging makes differences in the particles, from the center to the edges, obvious d The SEM image of PC cement made with dol SS granules shows details about the concrete (Figure 25). This PC concrete demonstrates a weak bond, seen as a space between the granulated dolomite and the cement paste. Furthermore, the granulated dolomite seems to have more void space than anywhere else (Figure 25). This characteristic was only seen in the dolomite concrete that was bonded with sodium silicate,.due to the changes in color (Figure 24) seen in all of the filler materials bonded with PC.

Concrete made with granulated AgDry SS demonstrated characteristics of a weak concrete (Figure 26). The samples granulated with a sodium silicate binder performed poorly in unconfined compression testing and the SEM imagery imparts information as to why this is true. The SEM image shows the AgDry SS particles are loosely packed. The AgDry SS granules are not likely to impart any strength to the PC cement as they appear weaker than the PC. Finally, a good grain-paste bond is not observed in these samples.

Similar to the PC concrete made with granulated AgDry SS, the PC concrete made with granulated Ag82 SS appears to have many weaknesses, according to the SEM and petrographic imagery (Figure 27). The granulated particles are full of large cracks. Cracks also have formed in the cement and run around the particle suggesting a weak grain-paste bond. These cracks indicate the Ag82, granulated with sodium silicate, is weaker than the PC matrix that runs around the granulated particles. Overall, this concrete appears to have been badly damaged by the strength test.

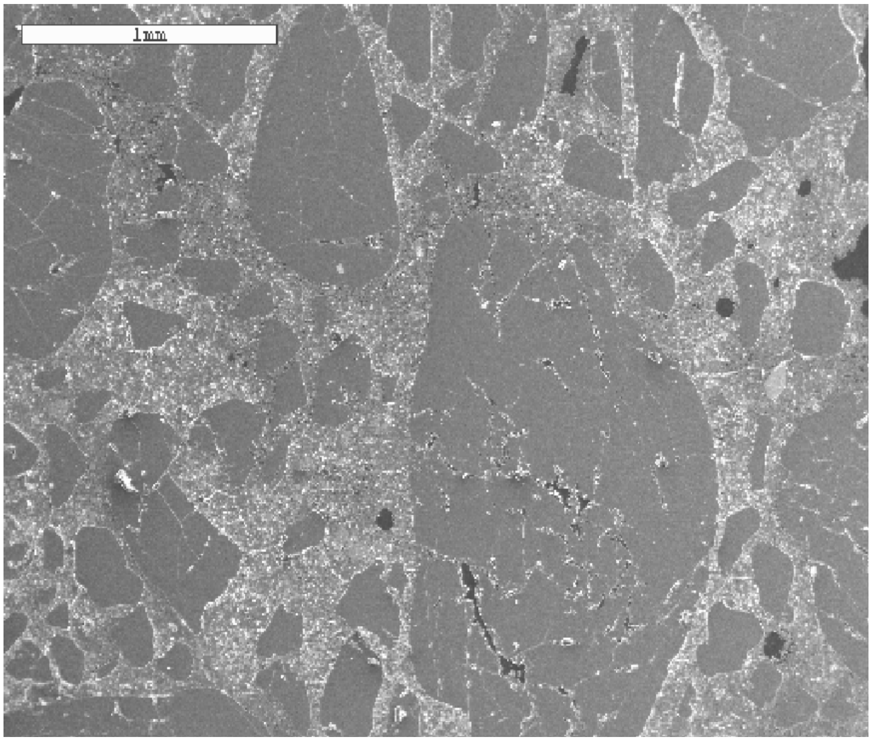


Figure 20: SEM image of the PC concrete made with FDOT sand

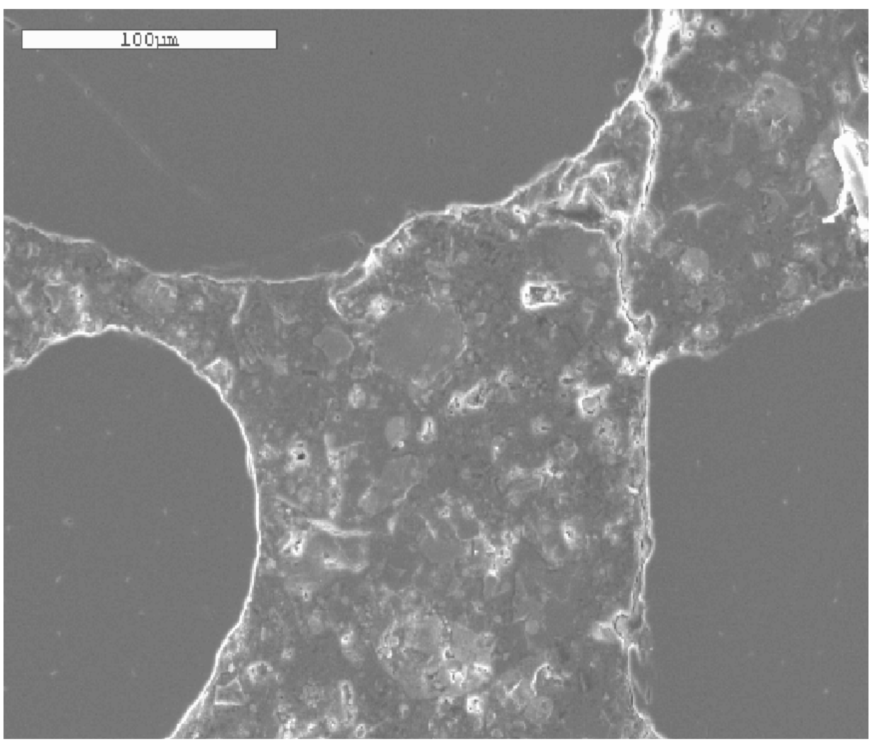


Figure 21: SEM image of PC concrete made with Ottawa sand

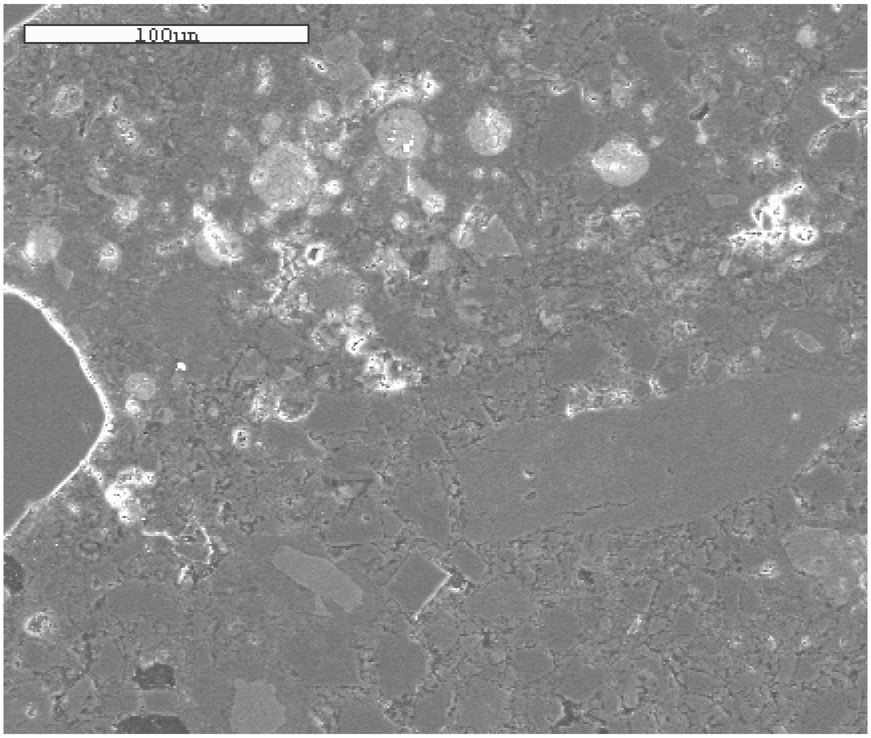


Figure 22: SEM image of PC concrete made with Ag82 PC

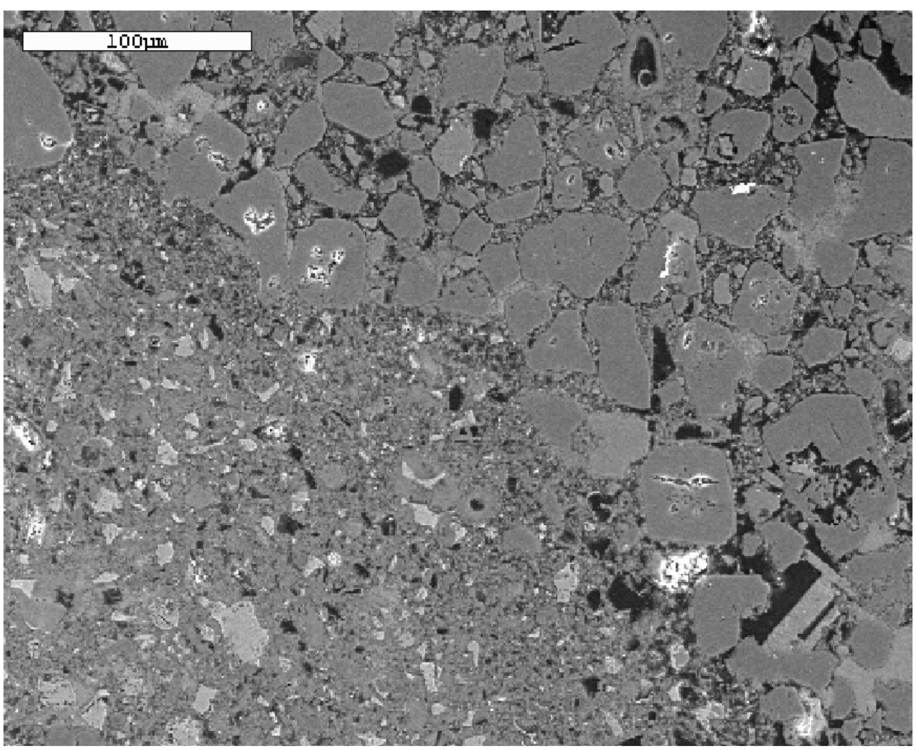


Figure 23: SEM image of PC concrete made with AgDry PC

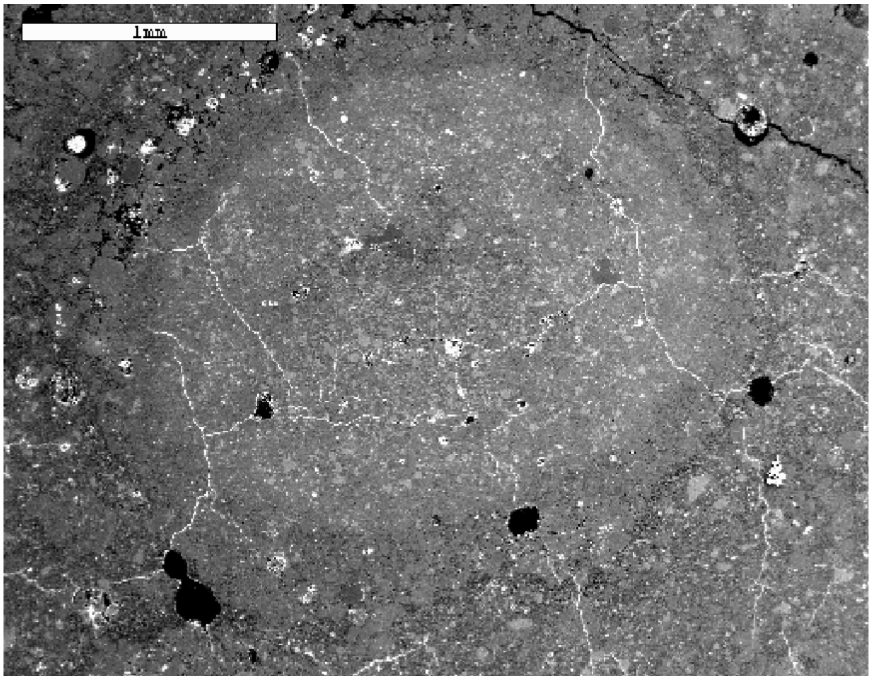


Figure 24: SEM image of PC concrete made with Dol PC

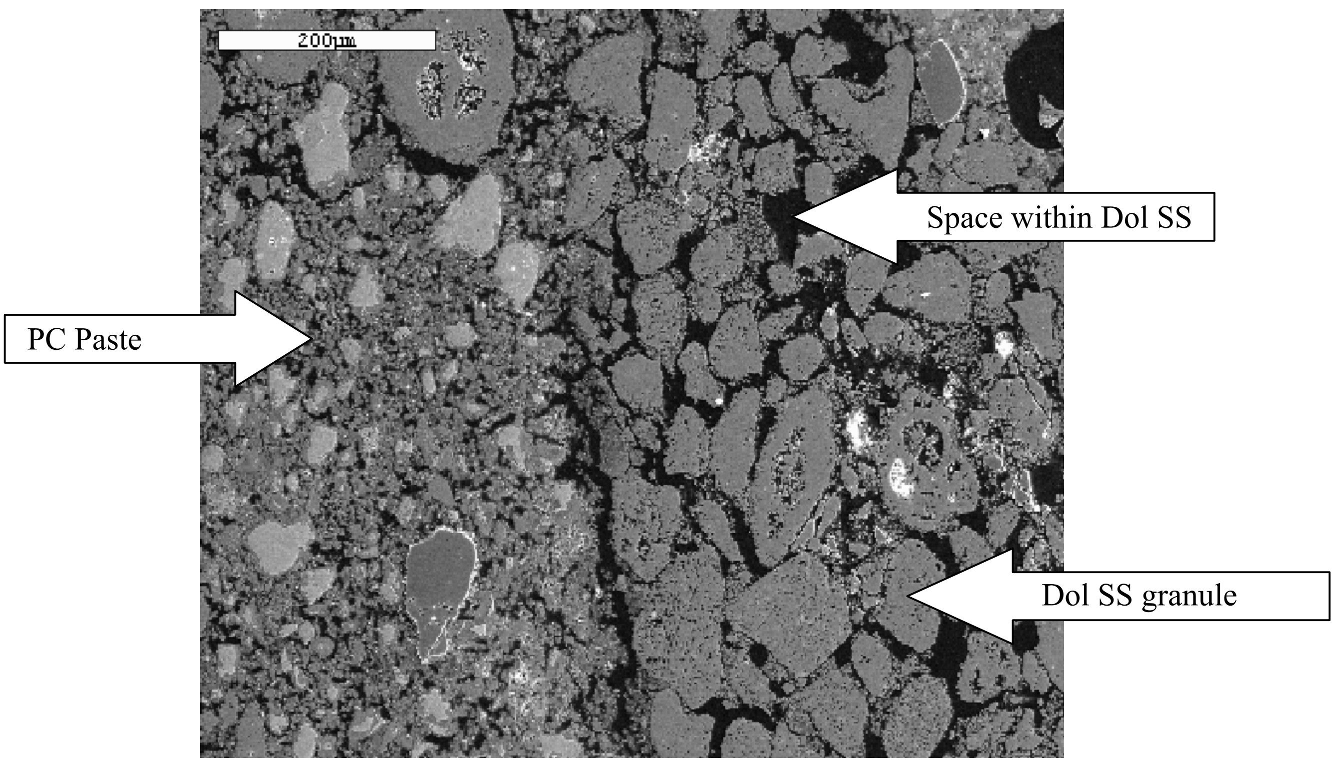


Figure 25: SEM image PC cement made with Dol SS

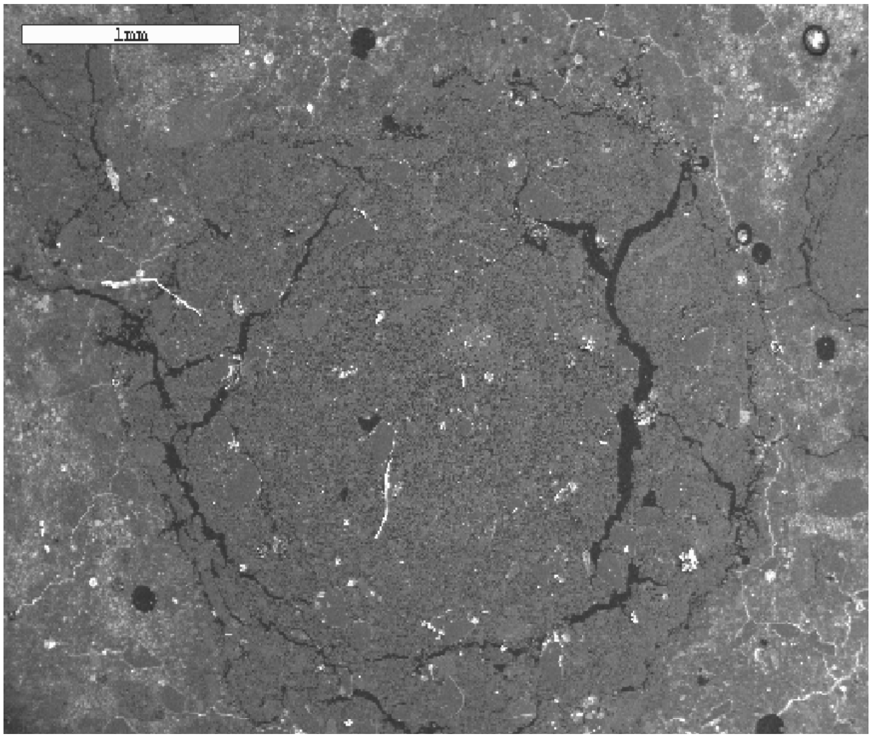


Figure 26: SEM image of PC concrete made with AgDry SS

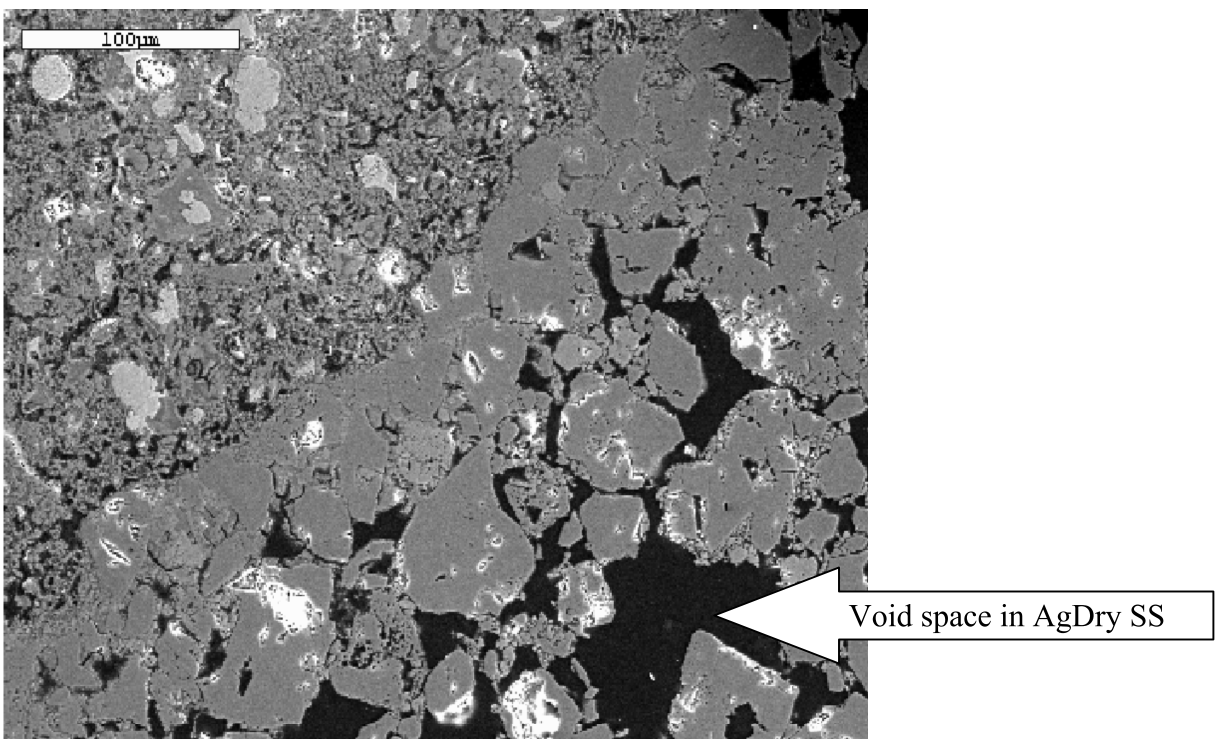


Figure 27: SEM image PC concrete made with Ag82 SS

# CHAPTER 7
# DISCUSSION

## Granulated Samples

The unconfined compressive strengths of PC concrete made with Ottawa sand and FDOT sand are higher than those of samples made with granulated filler material. The largest factor in the PC concrete's strength difference is likely the inherent strength of the quartz sand grains. As determined by SEM and petrographic analysis, another factor in the strength discrepancy is probably linked to the grain-paste bonding characteristics.

The PC concrete made with FDOT sand as the fine aggregate was stronger than the PC concrete made with Ottawa sand with 7071 and 4450 psi average 28-day unconfined compressive strength, respectively. The quartz sand grains of the Ottawa sand and the FDOT sand have similar strength and density, the difference in strength between the PC concrete made with these materials is likely to be due to the differences in size, shape and gradation of sands. The FDOT sand's irregularly shaped particles apparently creates enough friction and surface space for better grain-paste bonding when compared to the Ottawa sand. Furthermore, the gradation of the FDOT sand must include enough fine particles to prevent nestling of the particles which can leave less room for the binder (Portland Cement Association, 1952).

The PC cements made with granulated fines of Ag82 PC (4200 psi), AgDry PC (3167 psi) and Dol PC (2983 psi) have the next strongest unconfined compressive strength values. These concretes were made with filler material that was granulated with PC and have significantly stronger unconfined compressive strengths then seen in the PC concretes that were included filler that was granulated with sodium silicate binder. These concretes were weaker than the samples made with Ottawa sand and FDOT sand because the filler material's inherent particle strength was less. Similarly, these concretes were stronger than the samples made with sodium silicate binder because the filler material's inherent particle strength was less. Furthermore, these concretes were stronger than those made with sodium silicate binder because the PC binder and the PC of the concrete seemed to react with one another creating a strong grain-paste bond. PC concrete made with granulated fines of Ag82 PC had a compressive strength that showed potential for use as recycled aggregate in commercial concrete.

PC concrete made with Dol SS (1432 psi), AgDry SS (1429 psi), and Ag82 SS (971 psi) produced concrete with the three lowest unconfined compressive strengths. The low unconfined compressive strength is due to the low inherent strength of the granulated filler material in the concrete. When submerged in water, it is possible the sodium silicate binder dissolved and decreased aggregate strength. All three of the samples looked as if some dissolution of the particles into the cement paste had occurred when water was added to the cement mix. The unconfined compressive strength of the cubes was negatively affected if strength was lost from the granulated particles due to this dissolution. It is also a possibility that the sodium silicate binder caused alkali silica reaction to occur in these concretes, causing the extremely low compressive strengths, although no definitive evidence of this was seen all three of the samples that were granulated with sodium silicate had extensive cracking.

SEM and petrographic analysis showed physical evidence for the greater unconfined compressive strength in the PC concrete made with PC granulated fines, as compared to sodium silicate granulated fines. The filler material that was granulated with the sodium silicate binder is visibly less dense than the filler material that was granulated with PC. The greater density filler material gives greater strength to the PC concrete than the less dense filler material. Furthermore, the fine aggregate granulated with sodium silicate appears to be soluble when mixed with water, also decreasing the strength of the resulting concrete. The PC concrete made with filler that had been granulated with PC binder showed better grain-paste bonding characteristics than PC concrete made with filler that had been granulated with sodium silicate. For examples, the PC concrete made with Ag82 PC (4200 psi) and AgDry PC (3167 psi) showed good bonding characteristics with cracks that ran from the paste into the granulated particle.

**Heated and Room Temperature Samples, With and Without Fly Ash**

Past research states that the addition of fly ash to a cement mix will result in lower values of compressive strength initially, but significant strength improvements will be seen over time (Babu, 1996). This statement agrees with our data that shows lower values of unconfined compressive strength for concretes made with fly ash as compared to without fly ash for samples tested after 3 and 7 days of curing. This statement further agrees with the data that shows higher values of unconfined compressive strength after curing for 28 days (5871 psi) for concretes made with fly ash and cured at elevated temperatures as compared to samples cured without fly ash at elevated temperatures (5194 psi). This statement was not proven for samples made with fly ash and cured at ambient temperatures which actually showed lower 28 day values of unconfined compressive strength (5196 psi) than samples made without fly ash and cured at ambient temperatures (5500 psi = 28 day unconfined compressive strength). More curing time may be necessary for this prediction to be correct on samples made with fly ash and cured at ambient temperatures.

Research predicts that samples of concrete that are cured at elevated temperatures will produce lower values of compressive strength in the long term (VTCR, 1998). As predicted, elevated curing temperature decreased the 28 day unconfined compressive strength of concrete made without fly ash (5500 compared to 5194 psi). Conversely, elevated curing temperature increased the unconfined compressive strength of concrete made with fly ash (5196 compared to 5871 psi). More time may prove this prediction correct for concrete made with fly ash, but it seems likely the combination of elevated curing temperature accelerated the pozzolonic reactions in the fly ash increases the unconfined compressive strength of the resulting PC concrete. This accelerated pozzolonic reaction appears to produce higher values of unconfined compressive strength in the long term in samples made with fly ash and cured at elevated temperatures.

# CHAPTER 8
# CONCLUSIONS

1. PC concrete made with silica sand produced higher values of unconfined compressive strength than PC concrete made with granulated materials, which was likely due to the granulated fine aggregates' density and strength.
2. PC concrete made with FDOT sand had higher unconfined compressive strength values than concrete made with Ottawa sand, which was likely due to the FDOT sand's irregular sizing and angular to sub-angular shape.
3. PC concrete made with fine aggregate bonded with PC had higher unconfined compressive strength values than PC made with fine aggregate bonded with sodium silicate. This fact is likely to be a result of grain-paste bonding characteristics and the granulated aggregate's density and strength.
4. Ag82 granulated with PC has the potential for use as a fine aggregate in commercial PC concrete.
5. PC concrete cured at elevated temperatures, with fly ash, shows greater 28 day unconfined compressive strength compared to PC concrete cured at elevated temperatures without fly ash, at ambient temperatures with fly ash and at ambient temperatures without fly ash.
6. Fly ash may be a cost-effective and strength improving additive to concretes cured in Florida.

# CHAPTER 9
# SUGGESTIONS AND RECOMMENDATIONS

Some conclusions were drawn as a result of this study, and some questions were left unanswered, leaving room for future investigation into similar subjects. PC concrete made with granulated sodium silicate fines may have proved to have little potential for commercial usage in this study, but, if the weak unconfined compressive strength is a result of the solubility of the sodium silicate adding soluble calcium during processing may improve on the characteristics of the granulated fines. If the weak unconfined compressive strength is a result of alkali-silica reaction it might be advantageous to study the same particles in a concrete mix with an additive, such as fly ash, which should lessen the effects of the alkali-aggregate reaction.

It is likely that only the concrete that was granulated with PC particles warrant further investigation into their potential commercial usage. The PC concrete samples with this binder showed a correlation of greater strength with lower water to cement ratio and it may or may not have affected the results. Further investigations of the specifics of the granulation process, granule shape, gradation and water cement ratio may result in a better understanding of the properties that will create a better PC concrete. Each of these factors is likely to have a large bearing on the strength of the resulting PC concrete.

Finally, this project needs to be viewed from a construction manager's point of view. Cost of production needs to be evaluated for commercial construction. Questions need to be raised over the environmental benefits of recycling different fines as compared to the expense of granulating and making the fines into aggregate for PC concrete.

## LIST OF REFERENCES


ACI (American Concrete Institute) Committee 211, 1993, Guide for selecting proportions for high-strength concrete with Portland cement and fly ash: ACI Materials Journal v. 90 (May/June 1993), p. 272-283.

Ahmed, W. U., 1994, Petrographic methods for analysis of cement clinker and concrete microstructure; Petrography of Cementitious Materials PCN 04-012150-07: American Society for Testing and Materials, Philadelphia, PA.

Anderson, Mark Ph.D., P.E. and Maj. Dov Dover, P.E., 2002, Bonded Fly Ash: A Low-Energy Replacement for Portland Cement Concrete to Improve Resistance to Chem-Bio Intrusion; NBC Defense Collective Protection Conference Papers, Panama City, FL.

Applied Chemical Technology, Inc, 1998, Evaluation and Production of Granular Limestone from Limestone Fines: Applied Chemical Technology, Inc; 4350 Helton Drive; Florence, Alabama.

ASTM (American Society for Testing and Materials) Designation: C 109/C 109M-95, 1998, Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens): Annual Book of ASTM Standards, Vol. 4.01, Philadelphia, PA.

ASTM Designation: C 150-97, 1998, Standard Specification for Portland Cement: Annual Book of ASTM Standards, Vol. 4.01, Philadelphia, PA.

ASTM Designation: C 305-94, 1998, Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency: Annual Book of ASTM Standards, Vol. 4.01, Philadelphia, PA.

ASTM Designation: C 511-96, 1998, Standard Specification for Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes: Annual Book of ASTM Standards, Vol. 4.01, Philadelphia, PA.

ASTM Designation C 618-05, 2004, Standard Specification for Coal Fly Ash and Raw Calcined Natural Pozzolon for Use in Concrete: Annual Book of ASTM Standards, Vol. 4.02, Philadelphia, PA.

ASTM Designation: C 778.92a, 1998, Standard Specification for Standard Sand: Annual Book of ASTM Standards, Vol. 4.01, Philadelphia, PA.

Babu, K. Ganesh, and G. Siva Nageswara Rao, 1996, Efficiency of fly ash in concrete with age: Cement and Concrete Research v. 26 n. 3 (March 1996), p. 465-474.

Barringer, William L., 1997, Before using fly ash. Concrete International v. 19 n. 4 (April 1997), p. 39-40.

Davis, Sir Charles, 1948, Portland Cement, Concrete Publications Limited, Portland Street, London.

DePuy, G. W., 1990, Petrographic investigations of concrete and concrete aggregates at the Bureau of Reclamation; Petrography Applied to Concrete and Concrete Aggregates PCN 04-010610-07: American Society for Testing and Materials, Philadelphia, PA.

Ellis, W. E., Jr., 1992, For durable concrete, fly ash does not "replace" cement: Concrete International v. 14 (July 1992), p. 47-51.

Gaynor, Richard D., Richard G. Meininger and Tarek S. Kahn 1985, Effect of temperature and delivery time on concrete proportions; ASTM Special Technical Publication 858 PCN 04-858000-07: American Society for Testing and Materials, Baltimore, MD.

Ghosh, Suji, and Karim W. Nasser, 1996, Effects of high temperature and pressure on strength and elasticity of lignite fly ash and silica fume concrete: ACI Materials Journal v. 93 (January/February 1996), p. 51-60.

Halstead, W. J., 1996, Use of Fly Ash in Concrete: NCHRP 127 (October) Transportation Research Board, National Research Council, Washington, D.C.

Hamernick, Jubal D., and Gregory C. Frantz, 1991, Strength of concrete containing municipal solid waste fly ash: ACI Materials Journal v. 88 (September/October 1991) p. 508-5117.

Idorn, G. M., and Della M. Roy, 1986, Opportunities with Alkalies in Concrete Testing, Research, and Engineering Practice; ASTM Special Technical Publication 930 Alkalies in Concrete PCN 04-930000-07: American Society for Testing and Materials, Philadelphia, PA.

Jalali, S., and M. Y. Abyaneh, 1995, Prediction of final concrete strength in hot climates: Magazine of Concrete Research v. 47 n 173 (December 1995) P. 291-297.

Joshi, R. C., and R. P. Lohtia, 1997, Fly Ash in Concrete: Overseas Publishers Association, Amsterdam.

Keck, Roy H., and Eugene H Riggs, 1997, Specifying fly ash for durable concrete: Concrete International v. 19 (April 1997) p. 35-38.

Lange, David A., 1994, Long-Term Strength Development of Pavement Concretes: Journal of Materials in Civil Engineering v. 6. n. 1 (February 1994) p. 78-87.

Leshchinsky, M. Yu, V. A. Velichko, and A. M. Leshchinsky, 1991, Use of fly ash in concretes subject to heat: Cement and Concrete Research v. 21 n. 2-3 (March-May 1991) p. 205-218.

Mittelacher, Martin, 1985, Effect of hot weather conditions on the strength performance of set-retarded field concrete; ASTM Special Technical Publication 858 PCN 04-858000-07: American Society for Testing and Materials, Baltimore, MD.

Murdock, L. J., and K. M. Brook, 1979, Concrete Materials and Practice: Halsted Press, New York.

Naik, Tarun R., 1985, Effect of hot weather conditions on the strength performance of set-retarded field concrete; ASTM Special Technical Publication 858 PCN 04-858000-07: American Society for Testing and Materials, Baltimore, MD. Geological Society of America, 1950, Application of Geology to Engineering Practice: The Geological Society of America, New York.

Neville, A. M., and J. J. Brooks, 1987, Concrete Technology: John Wiley & Sons, Inc, New York, New York.

Owens, Philip L., 1985, Effect of temperature rise and fall on the strength and permeability of concrete made with and without fly ash; ASTM Special Technical Publication 858 PCN 04-858000-07: American Society for Testing and Materials, Baltimore, MD.

Portland Cement Association, 1952, Design and Control of Concrete Mixtures: Portland Cement Association, Chicago, IL.

Sarkar, Shondeep L., and Pierre-Claude Aitcin, 1990, The importance of petrological, petrographical and mineralogical characteristics of aggregates in very high strength concrete; Petrography Applied to Concrete and Concrete Aggregates PCN 04-010610-07: American Society for Testing and Materials, Philadelphia, PA.

Schindler, Anton K., and B. Frank McCullough, PhD., P.E., 2002, The Importance of Concrete Temperature Control During Concrete Pavement Construction in Hot Weather Conditions: The Annual Meeting of the Transportation Research Board, Washington, D.C.

Smith, Robert L., and Carl F. Raba, Jr., 1986, Recent Developments in these of Fly Ash to Reduce Alkali-Aggregate Reaction, Research, and Engineering Practice; ASTM Special Technical Publication 930 Alkalies in Concrete PCN 04-930000-07: American Society for Testing and Materials, Philadelphia, PA.

Stutzman, Paul E., 2001, Scanning Electron Microscopy in Concrete Petrography; Materials Science of Concrete Special Volume: Calcium Hydroxide in Concrete (Workshop on the Role of Calcium Hydroxide in Concrete), The American Ceramic Society. Novcember 1-3, 2000, Anna Maria Island, Florida, p. 59-72.PA.

Swamy, R. N., and M. M. Al-Asali, 1986, Influence of Alkali-Silica Reaction on the Engineering Properties of Concrete, Research, and Engineering Practice; ASTM Special Technical Publication 930 Alkalies in Concrete PCN 04-930000-07: American Society for Testing and Materials, Philadelphia, PA.

University of Florida, Department of Geological Sciences, 2002, Research and Techno-Economic Evaluation Uses of Limestone Byproducts Final Report Sponsored by the Florida Department State Contract No. BA589 WPI 0520789, Gainesville, Florida.

## ADDITIONAL READING


Ahmed, Imtaz, 1992, Use of Waste Materials in Highway Construction: Noyes Data Corporation, Park Ridge, NJ.

Carette, Georges G., and V. Mohan Malhotra, 1985, Temperature Effects on Concrete; ASTM Special Technical Publication 858 PCN 04-858000-07: American Society for Testing and Materials, Baltimore, MD.

Foster, George Kevin, 1985, Concentrations of Selected Trace Elements in Portland Cement and their Effects on Cement Compressive Strengths: University of Florida, Geology Department, Master's Thesis, Gainesville, Florida.

Petrucci, Ralph H., 1982, General Chemistry Principles and Modern Applications: Macmillan Publishing company, New York, New York.

Sarkar, S. L., and B. Samet, 1994, The microstructural approach to solving clinker related problems; Petrography of Cementitious Materials PCN 04-012150-07: American Society for Testing and Materials, Philadelphia.

Walker, Hollis N., 1997, Alkali-Aggregate Reactions, US Department of Transportation Research and Development; Publication No. FHWA-RD, p. 97-146, Mclean, VA.


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## BIOGRAPHICAL SKETCH

I grew up in Rochester, New York, where I went to a small elementary and high school with only 23 people in my graduating class. I went on to attend a small college also in upstate New York. I went to Union College in Schenectady, New York where I fell in love with the geological sciences. At Union I received a Bachelor of Science in geology with a minor in civil engineering. I enjoyed mixing geological studies with everything I learned in my civil engineering classes.

In 1997 I moved to Gainesville, Florida, to pursue a love of the warm weather while continuing to study. As in undergraduate school, I took classes to complete a degree in geology with a minor in civil engineering. It was a natural decision for me to write a thesis on a subject that involved both subjects.

In 2000 I completed all of my class requirements and left Gainesville and moved to Tampa, Florida. I have been working for an environmental engineering firm in Tampa since that time. Also since then I have been making very slow progress on this thesis. I am very proud to say I have completed it and I will always appreciate the work I did, effort I put into it and the people that helped and pushed me along the way.