THE EFFECTS OF AGGREGATE COATINGS ON THE PERFORMANCE OF PORTLAND CEMENT CONCRETE

by

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A thesis submitted in partial fulfillment of

the requirements for the degree of

Master of Science

(Civil and Environmental Engineering)

at the

UNIVERSITY OF WISCONSIN – MADISON

2002

APPROVED Steven M. Cramer 8/23/02

Steven M. Cramer, Professor Date

ACKNOWLEDGEMENTS

First and foremost, I would like to thank my advisor Steven Cramer, Professor of Civil and Environmental Engineering. Without his interest and confidence in my work, I would have been overwhelmed by the challenges that this project presented. His dedication helped shape my academic goals and will serve as an example as I continue my professional career.

I extend my appreciation to the Wisconsin Highway Research Program for their financial support and the Wisconsin Concrete Pavement Association for their direction during the course of the project. I would especially like to thank Jim Parry, Kevin McMullen, Dennis Brush, and Tom Ptaschinski who were extremely helpful in defining the aggregate coating problem and the overall scope of the project.

I am grateful to Prof. Hussain Bahia and Prof. Tuncer Edil for serving on my thesis committee and for their suggestions during the review process.

I acknowledge the following individuals and corporations for their technical assistance: Prof. David Mickelson, for answering our questions regarding the geology of Wisconsin aggregates; Andrea Carpenter for sharing her knowledge of concrete petrography; Michael Finnegan for x-ray diffraction training; Brian Hess for preparing thin-sections; Construction Technology Laboratory for verifying our x-ray diffraction results; American Petrographic Services for conducting hardened air void analyses. I am also grateful to Bill Lang, Carole Kraak, Tony Walls, Amy Dowell, Kyle Hu, Nick Mason, Steve Preston, Brent Ballweg, and Josh Koch for their help managing and completing the laboratory tests.

I thank the following contributors who donated the materials used in the research: Wingra Stone for their donations of fine aggregate; Lafarge Corp. for their donations of cement; Xiaodong Wang for identification of the Sauk Co. fines; Rick Shartrow for donation of the Barron Co. fines; all of the aggregate producers who were willing participants in the study and donated the coarse aggregates evaluated in the study.

Finally, I am thankful to those family and friends who supported me during my graduate work, especially to my parents who for their sincere interest in my progress – it is finally finished.

ABSTRACT

Specifications which limit the amount of fine material that adheres to coarse aggregates tend to be vague. For example, the Wisconsin state specifications require coarse aggregates used in portland cement concrete to be free of deleterious substances and adherent coatings; however, neither the washing procedure nor the desired end result of aggregate washing is specified. The purpose of this research was to identify which aggregate coatings in Wisconsin are deleterious and to determine what degree they affect concrete durability and strength. To this end, coarse aggregates containing surface coatings were collected throughout the state and characterized with the California cleanness test, methylene blue adsorption test, and x-ray diffraction. Based on the results of these tests, a subset of the sampled aggregates was selected to further study the effects of the coatings on concrete performance. It was hypothesized that coatings containing clay material are more deleterious than coatings containing either dust or carbonate material.

The 10-batch mixing plan tested coarse aggregates from three different sources and in the following three conditions: (1) coated aggregates that were sampled in the field, (2) washed aggregates that were washed in the laboratory to remove the existing coating, and (3) coated aggregates that were manufactured with dust and clay fines to increase the extent of the coating. The effect of each aggregate coating on concrete performance was assessed by comparing the relative changes in compressive strength, tensile strength, drying shrinkage, freeze-thaw durability, and chloride ion penetrability between batches containing the washed and coated aggregates. The test results confirmed that coatings with high clay contents are

more deleterious to concrete strength and durability than coatings that consist largely of dust or carbonate material. While the carbonate coatings appeared to slightly improve performance, the clay coatings appeared to significantly decrease strength and durability. Although current washing procedures do not need to be changed, it was recommended that the WisDOT require the California cleanness test whenever aggregate coatings are suspected of influencing strength or durability during concrete construction.

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CHAPTER 1 – INTRODUCTION

1.1 SIGNIFICANCE

In recent Wisconsin Department of Transportation (WisDOT) projects, the cleanliness of coarse aggregates has been perceived to significantly influence the performance of portland cement concrete. Of specific concern are the silt and clay particles that remain after washing and adhere to the surface of some igneous gravels. The adherent material, referred to as an aggregate coating, results from the weathering and processing of aggregates and is often difficult to remove. In northern and southeastern Wisconsin, some concrete batches mixed with coated gravel have suffered from low early compressive strength and premature cracking around the face of the aggregate. State engineers and concrete pavers have speculated that the coating affects concrete strength and durability by reducing the aggregate-cement paste bond and disrupting the air void system.

Although the effects of deleterious aggregate coatings are generally linked to the aggregate/cement paste bond, there have been few laboratory studies that have examined the nature of coatings or how they affect concrete performance (see Section 2.3). Because the potential influences of aggregate coatings are not well understood, specifications restricting their use in concrete construction tend to be vague. For example, the Wisconsin specifications require coarse aggregates to be "clean" and "free of adherent coatings which could be considered injurious" but do not define what coatings are injurious or how they

should be washed (WisDOT 1996). As a result, the engineer is left to decide which adherent coatings are deleterious and which are innocuous.

The benefit of removing coatings is related to the cost of washing and the stockpiling of the fines that are removed. Consequently, a better understanding of aggregate cleanliness could have significant impacts on the economics of aggregate processing. Identifying which coatings are harmful may indicate when more rigorous washing is required, while identifying which are harmless may indicate when washing procedures can be relaxed. Distinguishing between these coatings also provides a first step in determining the marketability of the removed fines for other uses.

1.2 HYPOTHESES

Aggregate coatings are usually controlled by specifying a maximum allowable percentage of material passing the No. 200 sieve (p200). For coarse aggregates, the WisDOT specification (1996) limits the amount of p200 material to 1.5% by mass. A slightly more restrictive limit of 1.0% is specified by the American Society of Testing and Materials (ASTM C33). However, the ASTM specification allows the limit to be increased to 1.5% if no clay or shale are present or if the specified p200 limit of the fine aggregate is not reached. The exceptions to the ASTM requirement suggest that the effects of aggregate coatings are closely associated with amount of clay in the coating.

Clay refers to particles that "develop plasticity when mixed with a limited amount of water (Grim 1953)." The term can be confusing because it is often used to describe both the particle size and the mineralogy of fine material. In general, clay material includes both

clay-sized (< 2 μm) and silt-sized (< 75 μm) particles and both clay minerals, such as kaolinite, illite, and montmorillonite, and non-clay minerals, such as quartz, feldspar, and dolomite. Typically, clay minerals dominate the clay-sized fraction, although some non-clay minerals may be small enough to also be classified as clay-sized. Since different clays can contain varying amounts of clay-sized particles, the relative amounts of clay and non-clay minerals can also vary.

It is likely that the most severe coatings contain a significant percentage of clay minerals because of their unique physical and chemical properties. Clay minerals differ from non-clay minerals due to a net negative charge and a high specific surface, defined as surface area per unit mass. The high specific surface results from their small particle size and platy shape, and the electrical charge results from the exchange of metallic cations in its crystal structure (Gillot 1968). The two factors are related since larger negative charges are derived from larger specific surfaces (Das 1998). Table 1-1 shows how specific surface values vary between different types of clay minerals and particle size classifications (White 1997).

Table 1-1. Specific Surfaces of Various Materials (White 1997)

Material/Mineral	Specific Surface (m^2/g)
Clay/Kaolinite	15
Clay/Illite	80
Clay/Montmorillonite	800
Silt	0.1-1

Coatings containing clay minerals may influence concrete performance by disrupting the aggregate/cement paste bond and frustrating the control of water in the mix. Because of the net electric charge of the clay minerals, these coatings are likely to adhere more strongly to the aggregate surface than non-clay coatings. The presence of clay is also likely to increase the water requirement of a given concrete mix due to its high specific surface, or increased surface area, and affinity for water. Water is attracted to the surface of clay minerals by three mechanisms (Das 1998):

1. The net negative charge on the mineral surface.
2. The net positive charge of cations, such as Ca^{2+}, Mg^{2+}, Na^{+}, K^{+}, surrounding the mineral.
3. Hydrogen bonding between hydrogen atoms in the water molecule and hydroxyl ions on the mineral surface

The affinity for water, often described as the activity of the clay, depends on the specific surface and varies for different clay minerals.

Because the concentration of cations decreases with the distance from the surface of the clay, the force of attraction between water and the clay mineral is greatest near the clay surface (Das 1998). The layer of water closest to the particle surface, called adsorbed water, is held very strongly to the clay, while the outermost layer of water, termed double-layer water, is held less strongly. The relative thickness of these layers varies among the different types of clay minerals and depends on the specific surface. Although the double-layer water may be available to improve workability or react with the cement, the adsorbed water is more viscous than ordinary water and is unlikely to be used to hydrate the cement.

Based on the different physical and chemical properties of clay and non-clay minerals, the following hypotheses are made:

1. Deleterious coatings found in Wisconsin contain significant amounts of clay minerals.
2. Innocuous coatings found in Wisconsin contain significant amounts of non-clay minerals.
3. The performance of concrete mixed with aggregates containing clay coatings can be improved if additional washing is used to remove the clay particles.
4. Specifications and tests that monitor aggregate coatings in the field should distinguish clay coatings from non-clay coatings.

1.3 OBJECTIVES

The purpose of this research was to investigate the mineralogy of aggregate coatings in Wisconsin and determine their effects on concrete strength and durability. In the study, the effects of the coatings were measured by comparing the performance of concrete samples made with and without coated aggregates. The need for changes in current specifications and aggregate processing methods was examined by reviewing washing techniques and coating characterization tests. Final objectives of the study are listed below:

1. Identify areas in Wisconsin where aggregate coatings are perceived to exist.
2. Determine the mineralogy of aggregate coatings found in Wisconsin.
3. Determine to what degree coatings affect concrete strength and durability.
4. Evaluate the effectiveness of aggregate washing.
5. Identify test methods to monitor aggregate cleanliness.

The methods used to achieve these objectives were divided into three phases. In Phase I, previous research was reviewed to determine the current state of knowledge of aggregate coatings and the existing tests and specifications that limit their use. In Phase II, WisDOT district offices and concrete contractors were surveyed to identify the location of suspect aggregates in Wisconsin. Samples collected from these aggregate sources were tested to determine the quantity and mineralogy of the aggregate coatings. In Phase III, a subset of the aggregates identified in the second phase was chosen for concrete mixing. The aggregates used in the mixing plan included coated aggregates as sampled in the field, washed aggregates in which the existing coatings were removed by washing, and coated aggregates in which fines were artificially attached to the aggregate surface to increase the extent of the coating. Concrete performance was evaluated by measuring strength and durability and by observing thin-sections under a stereomicroscope.

1.4 SCOPE

The coarse aggregate sources examined in this study were limited to areas in Wisconsin where coatings were perceived to exist. Most of the samples were taken from stockpiles of ¾- in. washed stone that met the gradation and deleterious substance requirements of the Wisconsin Department of Transportation. As a result, the initial amount of material passing the No. 200 sieve did not exceed 1.5% by mass. The locations of the aggregate sources are reported by county but not by a specific quarry name in order to keep the specific sources confidential. The same crushed limestone fine aggregate was used in

each concrete batch; however, the extent of any fine aggregate coatings was not measured or considered when analyzing concrete performance.

The performance of concrete mixed with the coarse aggregate was examined in laboratory conditions in batches with a fixed water-cement ratio of 0.45 and a target air content of 6.0%. To separate the effects of aggregate coatings from the effects of fine mineral admixtures, the mix design did not include fly ash or granulated ground blast furnace slag. Testing included slump and air content measurements on fresh concrete and compressive strength, tensile strength, shrinkage, freeze/thaw durability, and rapid chloride ion penetrability measurements on hardened concrete samples.

CHAPTER 2 – BACKGROUND

2.1 DESCRIPTIONS OF AGGREGATE COATINGS

The earliest description of surface coatings on coarse aggregates was documented by Goldbeck (1932) for the Highway Research Board. The properties of the seven coating types discussed in the report are summarized below:

1. Stone dust coatings are created from the fines generated during the crushing, screening, and handling of the aggregate. When mixed with water, the dust particles cling to the aggregate surface forming a visible film around the aggregate. The attraction of the coating depends on the mineralogy, moisture condition, and surface roughness of the aggregate. In most cases, the dust (especially siliceous dust) does not adhere firmly to the aggregate and is easily removed with washing. However, some types of limestone dust can adhere strongly to the aggregate and require more vigorous washing.
2. Clay coatings originate from overburden or seams of clay material in the aggregate deposit and from contaminated water used during the washing process. The characteristics of the coating vary depending on the type of clay particles present. Some types of clay are held so tightly to the aggregate surface that they may not be displaced after washing, while other types of clay may break up in water and are removed during concrete mixing.

3. Organic coatings are deposited from seepage through overlying layers of organic material or from streams carrying organic matter. The principle organic substances that have been investigated, dihydroxstearic acid and humic acid, can prevent a bond to form between the aggregate and the cement paste. These coatings can be removed by washing, burning, or adding salts or acids to flocculate the organic particles.
4. Alkali and salt coatings are carried in solution by streams and groundwater. The coatings, made up of sulfate and carbonate compounds, precipitate onto the aggregate surface and may crystallize in the pores of concrete.
5. Bituminous oil coatings develop from natural pockets of oil that have been found in some limestone deposits. The oil coating may also trap dust particles generated during processing of the aggregate.
6. Calcareous coatings consist of sand fragments cemented to the aggregate surface. The coating can vary from a very thin, small spot to a thick film surrounding the entire aggregate.
7. Sugar coatings result from contamination during shipping and storage of the aggregate. Even if they are present in small amounts, sugar coatings can have dramatic effects on concrete performance.

Based on the work of Goldbeck, several researchers have developed more general classifications of aggregate coatings. Lang (1943) divided coatings into three categories depending on their mineralogy and strength of adherence: clay coatings, dust coatings, and cementation coatings. Clay coatings consist of clay particles that are held tightly to the aggregate surface. Because the material usually adheres to the aggregate even after the

concrete is mixed, it is believed to interfere with the aggregate-cement paste bond. Unlike clay coatings, dust coatings are easily removed during mixing and affect concrete performance by increasing the amount of fines dispersed in the mix. Cementation coatings, similar to the calcareous coating type described by Goldbeck (1932), are deposited from solutions in calcite deposits. However, little was known about the nature of this coating due to lack of research at that time.

A similar classification of aggregate coatings was given by Schmitt (1990) who distinguished between dust coatings, cementation-type coatings, and impregnation-type coatings. Dust coatings are developed during crushing operations and consist of silt-sized grains with the same mineralogy of the aggregate. Depending on their composition, the dust particles can act as a pozzalonic admixture if they are removed during mixing. Cementation-type coatings include contaminants such as clay, calcium carbonate, salts, phosphates, and iron oxides. These coatings, usually calcium carbonate in nature, are deposited on the aggregate by the weathering of overburden deposits or the precipitation of water-soluble particles carried by groundwater (Dolar-Mantuani 1983). Impregnation-type coatings form from oils that are generated during aggregate processing. Since the oil impregnation is not developed naturally in the aggregate deposit, these coatings are different from the bituminous oil coating type described by Goldbeck.

Ozol (1979) and Forster (1994) classified aggregate coatings according to their source instead of their composition or strength of adherence. Ozol linked the nature of the aggregate coating to the geology of its formation. Coatings deposited by water are mostly mineral in nature and include calcium carbonate, iron oxide, gypsum, and alkali sulfates. Coatings

containing clay, silt, and organic matter are found in the overlying layers of the deposit or are placed on the aggregate during processing. Similarly, Forster divided coatings into those that are naturally occurring and those that are artificially generated. Naturally occurring coatings, typically calcium carbonate and silica, develop from weathering processes in the aggregate deposit. Artificially generated coatings include the fine dust that is created by the impact and abrasion of the aggregate when it is crushed and stockpiled.

A review of existing literature shows that descriptions of aggregate coatings have not changed much over the years. Most coatings that are encountered in the field appear to consist of stone dust, clay particles, or calcium carbonate. Distinguishing between these coating types is important because the mineralogy of the coating is closely related to its physical properties, such as strength of adherence, and its effects on concrete performance.

2.2 PROBLEMS WITH AGGREGATE COATINGS IN WISCONSIN

The major aggregate types found in Wisconsin are outlined in Figure 2-1. As shown in the map, igneous gravel and sandstone dominate the northern and western part of the state, while limestone deposits cover regions in the southeast.

The cleanliness of aggregate in Wisconsin appears to be related to the source from which it is produced. As a general rule, river gravel is cleaner than glacial till since the sand and gravel carried by the river are deposited in separate locations. Coatings that adhere to these aggregates most likely consist of water-soluble material transported by the stream. Unlike river gravel, glacial deposits are susceptible to clay coatings because the aggregate is mixed with large amounts of sand and clay (Buckley 1903). According to Mickelson

(personal communication), the percentages of kaolinite and illite in these deposits are fairly uniform across the state, while the montmorillonite content is only significant in the deeply weathered regions of northern Wisconsin. In the southern part of the state, the erosion and processing of limestone deposits are likely to generate carbonate coatings.

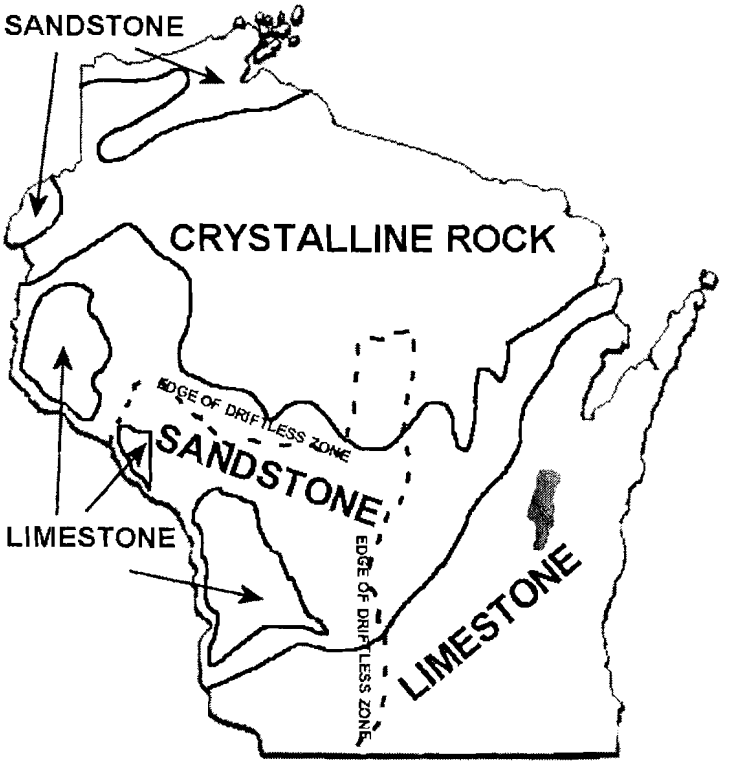

Figure 2-1. Major Aggregate Types in Wisconsin (after Hotchkiss and Steidmann 1914)

The descriptions of aggregate coatings above match the perceived problems with aggregate cleanliness in Wisconsin. Examples of the aggregate coatings observed in state concrete projects are listed below. Note that no coatings have been perceived to be

problematic in the driftless area in southwestern Wisconsin (see Figure 2-1) where the glaciers did not advance.

1. Coatings, presumably clay, adhering to igneous gravel in northern Wisconsin have been used in unusually stiff concrete, giving the impression that the cement was defective (D. Kircher, personal communication).
2. An unidentified coating in southeastern Wisconsin was responsible for cracking at the aggregate-concrete interface (R. Gutierrez, personal communication). The adhering strength of the coating was strong enough to survive vigorous washing.
3. Carbonate coatings on crushed limestone aggregate have been blamed for low strength concrete in southern Wisconsin (G. Mueller, personal communication).

2.3 EFFECTS OF AGGREGATE COATINGS ON CONCRETE

It is generally believed that the effect of aggregate coatings depends on whether or not the particles adhere to the aggregate surface after mixing. Because clay fines are bound to the aggregate by electrostatic forces, many researchers suggest that clay coatings disrupt the aggregate-cement paste bond (Dolar-Mantuani 1977, Forster 1994, Schmitt 1990, Goldbeck 1932, Neville 1996). If the bond between the cement paste and the coating is stronger than the bond between the coating and the aggregate, a weak zone may develop at the coating-aggregate interface and significantly reduce concrete strength and durability (Forster 1994). However, if the coating is as strongly bonded to the aggregate as it is to the cement paste, the coating may only affect concrete performance by absorbing mix water (Ozol 1979). Unlike clay coatings, dust coatings and carbonate coatings are removed from

the aggregate during mixing. Depending on the chemical composition and water demand of the dispersed fines, these coatings can either have beneficial or detrimental effects on concrete performance.

2.3.1 Effects of Coatings on the Aggregate-Cement Paste Bond

The link between coatings and the aggregate-cement paste bond dates back to Goldbeck (1932) who cited research examining the effect of stone dust on concrete strength and wear resistance. In the study, dust coatings were created by moistening the coarse aggregate and adding fines from granite, limestone, or gneiss rock in amounts up to 5.7% of the coarse aggregate weight. It was believed that the dust would either adhere to the aggregate and disrupt the aggregate-cement paste bond or separate from the aggregate and form a weak layer at the surface of the concrete. While the dust did not significantly affect the wear resistance, it did influence the strength of the concrete. For each 1% increase in the amount of dust, the compressive strength decreased between 0% and 2% and the modulus of rupture decreased between 1% and 1.5%.

Although many conclude that aggregate coatings weaken the aggregate-cement paste bond, there is little experimental evidence that supports the claim that this effect reduces concrete strength and durability. In fact, a literature review on the aggregate-cement paste bond reports some controversy regarding the relationship between bond strength and concrete performance (Struble, Skalny, and Mindess 1980). Some researchers suggest that poor bonding allows microcracks to propagate at a lower stress, while others believe that the stress level of microcrack propagation does not affect the ultimate concrete strength (Struble

et al. 1980). According to Tasong, Lynsdale, and Cripps (1998), the bond between the aggregate and the cement paste depends on three mechanisms:

1. Mechanical interlocking of hydration products with the aggregate surface
2. Epitaxial growth of hydration products at some aggregate surfaces
3. Chemical reactions between the hydrating cement and the aggregate

Aggregate coatings may influence bond strength and concrete performance by directly affecting one of these mechanisms.

Zimbelmann (1985, 1987) offered a theoretical description of the interfacial zone between the aggregate and the cement paste based on observations made using x-ray diffraction and scanning electron microscopy. According to his description, half the thickness of the interfacial zone (25 μm) consists of a water film, which forms around the aggregate during mixing. As the cement begins to hydrate, hydration products dissolve in the mixing water and diffuse into this film. There the dissolved substances crystallize, expelling part of the water and forming a contact layer of $Ca(OH)_2$ crystals around the aggregate. Although the $Ca(OH)_2$ crystals strengthen the bond between the aggregate and the cement paste, the water film increases the porosity of the contact zone by as much as 50% and significantly reduces the bond strength (Zimbelmann 1985). As a result, Zimbelmann concluded that the bond could be strengthened by reducing the water film layer or by increasing the hydration products in the contact zone.

While discussing the contribution of mechanical interlocking to bond strength, Dolar-Mantuani (1983) suggested that the quality of the bond is related to the penetration of cement paste into the pores of the aggregate. Darwin and Slate (1970) tested this theory by

comparing concrete mixed with an aggregate coated with polystyrene and concrete mixed with an uncoated aggregate. In the experiment, the strength of the aggregate-cement paste bond was measured using 1-in. by 1-in. by 1-3/8 in. aggregate prisms encased in a cement paste briquette. It was assumed that the polystyrene coating would significantly decrease the bond strength and, in turn, decrease the strength and stiffness of the concrete. However, Darwin and Slate found that the coating only slightly reduced concrete strength (10%-15%) despite causing large reductions in bond strength (60%-95%). Although the results raise questions about the importance of bond strength to concrete strength, it should be noted that the exact effect of the polystyrene coating on the bonding mechanisms between the aggregate and the cement paste was unknown.

In a similar study, Patten (1973) tried to investigate the relationship between bond strength and the surface chemistry of the aggregate. Using a non-adhesive silicone release agent, a thin coating was placed around the aggregate to disrupt any chemical reactions in the interfacial zone. While Patten assumed that the coating eliminated adhesive bonds without affecting the mechanical interlock, Struble et al. (1980) questioned the assumption and suggested experimental verification. On average, the silicone coating decreased compressive strength by 25% and tensile strength by 17%. Because the strength reductions were greatest for early age concrete, it was concluded that mechanical interlocking is more important as the concrete cures.

The importance of the chemical bonding mechanism depends on the mineralogical composition of the aggregate. Typically, carbonate aggregates produce higher bonding strengths than silicate aggregates because reaction products increase the contact surface

between the aggregate and the cement paste and improve mechanical interlock (Grandet and Olivier 1980). However, Tasong et al. (1999) indicated that chemical interactions between the aggregate and the cement paste may also reduce bond strength. For example, reactions with limestone aggregates can increase the porosity of the interfacial zone in early age concrete, and reactions with basalt aggregate can breakdown feldspar into swelling clay minerals in later age concrete. Although research has also associated the expansion of certain carbonate aggregates to an alkali-carbonate reaction, the problem appears to be limited to aggregates with a specific type of mineralogy (Ozol 1994).

The possible effects of coatings on the aggregate-cement paste bond can be deduced from research attempting to improve the bonding characteristics of silicate aggregates. Based on the physical and chemical bonding mechanisms, Popovis (1987) tried to improve the aggregate-cement paste bond using an epoxy coating to attach fine sand or unhydrated cement particles to the aggregate surface. However, only the aggregates coated with cement increased strength. In a more successful study, Zimbelmann (1987) increased the bond strength above the tensile strength of the concrete with a mixture of sodium silicate and pumice, which reduced the water layer around the aggregate and produced $Ca(OH)_2$ crystals from pozzalonic reactions with the cement paste. Similarly, Graves, Eades, and Smith (1992) increased the epitaxial growth of hydration products at the aggregate surface by treating granite aggregate with a solution of $Ca(OH)_2$ to promote chemical bonding. They reported a 21% increase in compressive strength but only small changes in tensile strength.

Because it is nearly impossible to isolate the contribution of the aggregate-cement paste bond to concrete strength, studies investigating the bond using comparative

compression and tensile strength tests are at a distinct disadvantage. In recent research, this problem has been overcome by examining the characteristics of the interfacial bond with finite element models. Using this method, Darwin (1999) estimated that the greatest effect of interfacial bond strength on compressive strength is 15%, which is significantly less than the estimates of previous researchers. Since the significance of the aggregate-cement paste bond is still being debated, models used to explain the effects of aggregate coatings must be inferred from the theories discussed above. Some possible physical and chemical interactions between the coating and the cement paste are listed below for each coating type.

1. Dust coatings that are not displaced during mixing may decrease bond strength if they decrease the permeability of the aggregate surface and disrupt the penetration of cement paste into the pores of the aggregate. However, the hydration products may also interlock with the grains of the coating without disrupting the mechanical bonding mechanism.
2. Clay coatings may attract significant amounts of water to the aggregate surface, as described in Section 1.2. This water absorption could decrease bond strength by increasing the thickness of the water film in the interfacial zone.
3. Carbonate coatings may react with $Ca(OH)_2$ in the cement paste. The reaction products can increase the contact zone between the aggregate and the cement paste and improve mechanical interlocking.

2.3.2 Effects of Coatings as Dispersed Fines

If coatings are removed during mixing, fine particles that were not accounted for when the aggregates were batched are dispersed into the concrete. These dispersed fines can

be either beneficial or detrimental to concrete strength depending on the quantity and mineralogy of the added material. In general, the addition of clean stone dust and carbonate fines can increase strength, but the addition of clay fines almost always causes strength reductions (Pike 1992).

One of the earliest studies that investigated the effects of fines in concrete was led by researchers at the University of Colorado (Goldbeck 1932). During the study, crushed granite dust was dispersed in the fine aggregate just before mixing. When the water absorption of the added fines was ignored in the mix design, the slump decreased and the concrete strength increased. However, when the water absorption of the fines was controlled by keeping the slump constant, the gross water-cement ratio increased and the concrete strength decreased. The researchers found that these two effects canceled out for dust additions up to 15% to 20% of the fine aggregate portion. Based on these results, it appears that typical percentages of crusher dust do not significantly affect strength, although the dust may frustrate the control of water during mixing.

Bonavetti and Irassar (1994) investigated the effects of stone dust on a larger scale by replacing a portion of mortar sand (0% to 20%) for an equal weight of quartz, granite, or limestone fines. As the percentage of fines in the mix increased, Bonavetti and Irassar found that more water was required to maintain constant workability. This effect increased the water-cement ratio of the mortar, which in turn increased the porosity and shrinkage of the mix. Unexpectedly, however, the increase in water-cement ratio also corresponded with increases in the 7-day compressive and flexural strengths. To explain this result, Bonavetti and Irassar reasoned that the fines act as void fillers, which decrease porosity and accelerate

hydration of the cement paste. It was concluded that stone dust can either increase or decrease the strength of mortar depending on the balance between the water requirement and the filler effect.

In a similar study, Ahn, Fowler, and Hudson (2000) tested mortars using several different types of manufactured sands with p200 percentages ranging from 10%-20%. According to their results, the clay content of the aggregate, as measured by the modified methylene blue value described in Section 2.4.5, had the greatest influence on compressive strength and drying shrinkage. Although the water absorption capacity and p200 content also affected the mortar strength and shrinkage, their strength of correlation was not as significant. Other researchers have explained these results by suggesting that clay fines have a higher water demand than non-clay particles since they act primarily as adherent coatings on the sand grains instead of void fillers (Pike 1992).

Unikowski (cited in Pike 1992) provides further evidence that clay fines act differently than dust fines. In his experiment, mortar was prepared using seven different types of fines – kaolinite, illite, palygorskite, montmorillonite, limestone dust, and two kinds of siliceous dusts – that were replaced for equal weights of sand up to a percentage of 10%. Unikowski found that the water demand of the clay fines was related to their specific surface, with montmorillonite having the largest water requirement and kaolinite having the lowest. In contrast, some limestone and siliceous fines improved workability without the addition of any water. Due to the change in water-cement ratio, mortar mixed with the clay fines had lower strengths and higher shrinkages than mortar containing the non-clay fines. For example, a 3% replacement of montmorillonite for sand decreased compressive strength

nearly 40% and doubled the amount of shrinkage. However, a 3% replacement of limestone for sand increased compressive strength by 15% and slightly decreased shrinkage. Incidentally, small amounts of each fine type (~1%) increased strength by as much as 5%.

Other researchers suggest that the effects of dispersed fines depend on the quantity and activity of the entire p200 fraction. Buth, Ivey, and Hirsch (1968) showed that as the sand equivalent value decreases from 80 to 60, compressive strength decreases 16%, shrinkage increases 17%, but freeze-thaw durability remains unchanged. For a 3% mixture of silt and clay, this change in the sand equivalent value is equivalent to an increase in liquid limit from 95% to 276% (Buth et al. 1966). Although these results have been verified in other studies, research reviewed by Pike (1992) indicates that the shrinkage of mortar containing mixtures of silt and clay falls within acceptable levels when the silt and clay is limited to 3% of the sand weight.

Anecdotal evidence reported by Davis, Mielenz, and Polivka (1967) suggests that clay particles can also affect concrete by reacting chemically with the cement. During the construction of a dam in a Western state, engineers and contractors became concerned when the slump of the concrete decreased between 60% and 80% within the first 15 minutes of mixing. At first, it was believed that defective cement produced a false set; however, it was later discovered that the sand contained significant amounts of montmorillonite. A petrographic examination revealed that the sand, taken from a previously undeveloped, deeply weathered glacial deposit, was contaminated with coatings and dispersed particles of very fine clay grains and mineral dust. After a series of lab tests, it was concluded that the clay material reacted with the hydrating cement and adsorbed significant amounts of water.

Interestingly, this case study seems to match the experiences in Northern Wisconsin described in Section 2.2.

Noble (1967) described the reactions between clay and cement in more detail based on his research on stabilized soils. In clay-cement mixtures containing kaolinite, illite, or montmorillonite, Noble suggested that the following reactions occur:

1. Hydration of cement compounds with the production of $Ca(OH)_2$
2. Adsorption of Ca^{2+} within the clay structure by cation exchange
3. Adsorption of $Ca(OH)_2$ on the clay surface
4. Encapsulation of $Ca(OH)_2$ crystallites by fine clay particles and hydration products
5. Possible encapsulation of cement grains by clays and hydration products
6. Chemical combination of $Ca(OH)_2$ with SiO_2 and Al_2O_3

Noting these reactions, Pike (1992) theorized that the loss of strength in mortars containing clay fines is caused by clays adsorbing part of the mix water and forming "impermeable envelopes" around the cement grains. Ultimately, these effects slow hydration and disrupt the adhesion of the fines to the cement.

The need to account for displaced aggregate coatings in the mix design depends on the quantity and mineralogy of the dispersed fines. Dust fines can either increase strength by accelerating hydration or decrease strength by increasing water demand (Bonavetti and Irassar 1994). According to Hughes and Ash (1986), reasonable amounts of dust fines are usually acceptable in concrete. On the other hand, significant amounts of dispersed clay fines are unacceptable because they increase the water demand of the aggregate and prevent

adhesion to the cement paste (Pike 1992). Pike suggests the following rules-of-thumb to estimate the effect of clay fines on concrete strength:

1. An addition of 1% kaolinite by mass of cement causes a loss of strength of 1%.
2. An addition of 1% illite by mass of cement causes a loss of strength of 2%.
3. An addition of 1% montmorillonite by mass of cement causes a loss of strength of 4%.

2.4 TESTS TO CONTROL AGGREGATE COATINGS

Several tests are available to characterize the nature of aggregate coatings. Although some of these tests are currently being used to monitor the cleanliness of coarse aggregates, the majority are adaptations of fine aggregate tests or are only used as research tools. The application of these tests to aggregate coatings is described in the following sections.

2.4.1 Material Passing the No. 200 Sieve By Washing (ASTM C117)

The amount of silt and clay sized particles in a coarse aggregate sample is determined by washing the aggregate over a No. 200 sieve. During the test, the sample is rinsed with either plain water or a washing agent, which helps remove stronger adhering material. This procedure is more efficient than dry sieving since the water removes coatings and breaks apart particles that cling together. The percentage of fines in the sample is calculated as the percent mass passing the No. 200 sieve, often referred to as the p200 percentage.

Most states control aggregate coatings by specifying a maximum p200 percentage. In Wisconsin, the state specification limits the amount of p200 material on coarse aggregate to 1.5% (WisDOT 1996). A similar restriction is recommended in the ASTM standard (ASTM C33), although the allowable percentage ranges from 1.0% to 1.5% depending on the amount

of clay in the aggregate and the cleanliness of the fine aggregate. However, the specifications used in the United States seem to be more conservative than those used in Europe. The British standard, for example, allows a p200 percentage of 2.0% on river gravel and 4.0% on crushed aggregate (Neville 1996).

The p200 test is suited to investigations of aggregate cleanliness because the washing process removes loosely adhering fines and water-soluble particles from the surface of the aggregate. These particles correlate well with the dust coatings that are removed during concrete mixing (Dolar-Mantuani 1977). However, the p200 percentage may not accurately measure the extent of the coating since it includes fines dispersed in the aggregate and excludes strongly adhering particles that might not be removed during the washing procedure. In addition, the test does not distinguish between fines that may not affect concrete performance, such as some types of stone dust, and those that are harmful to concrete performance, such as the swelling clay particles. As a result, coarse aggregates that pass current p200 specifications could potentially perform poorly in the field.

2.4.2 California Cleanness Test (Caltrans Test No. 227)

The California cleanness test, similar to the sand equivalent test for fine aggregate, measures the relative amount of clay-sized particles clinging to the aggregate. In the test, adherent material is removed by mechanically washing a sample of coarse aggregate in a special container. After collecting the wash water that passes through a No. 200 sieve, the water is placed in a graduated cylinder and mixed with a solution of glycerin and calcium chloride. The height of the sediment that settles at the bottom of the graduated cylinder after 20 minutes is translated into a cleanness value from 0 (dirtiest) to 100 (cleanest). A

minimum cleanness of value of 75 is specified by the California Department of Transportation (Caltrans, 1999), which uses the test to measure aggregate cleanliness instead of the p200 test.

Unlike the p200 percentage, which only measures the amount of silt and clay in the sample, the cleanness value indicates the quantity, particle size, and activity of the material adhering to the aggregate (Benson and Ames 1975). Theoretically, lower cleanness values are determined for samples with very fine particles, which take longer to settle in solution, and active clay particles, which increase in volume by adsorbing water. Although the test has not been evaluated in many published research studies, existing research on the sand equivalent test raises questions about its sensitivity to different clay minerals and its accuracy for samples with significant amounts of non-clay fines (Pike 1992, Tourenq and Tran 1997).

2.4.3 Petrographic Examination (ASTM C295, ASTM C856)

During a petrographic examination, laboratory techniques are used to determine the composition and properties of concrete and aggregate. This type of analysis is often used during a forensic investigation to study the cause of concrete failure or poor aggregate performance (Kelley, Bell, Brainerd, and Scali 2001). A typical petrographic examination consists of basic observations made with optical microscopes and, if needed, more advanced tests with scanning electron microscopes or x-ray diffraction. As described below, the type of equipment required depends on its capability and the properties to be determined.

1. The stereomicroscope has a range of magnification of 10x to 100x or more. It is used to determine the composition and texture of aggregates and analyze the air void system of concrete.

2. The polarizing light microscope has a range of magnification of 25x to 1000x. It is used to determine the crystallinity and grain size of aggregate and investigate microcracks and the aggregate/cement paste bond in concrete.
3. The scanning electron microscope can produce images up to a magnification of 50000x. However, its most powerful application is analyzing elemental composition from the X-rays produced by its electron beam. It is used to investigate clay minerals in aggregate, alkali reaction products in concrete, and hydration products at the aggregate-cement paste bond.
4. X-ray diffraction can identify the mineral phases present in aggregate and cement powders (see Section 2.4.4). It is used to determine clay mineralogy and reactive carbonate material in aggregate.

Coarse aggregate samples are usually examined for coatings during the initial stages of a petrographic examination (Dolar-Mantuani 1983). At this time, the coating is removed by washing the aggregate and evaporating the wash water. The portion of the coating that passes the No. 200 sieve is tested for the presence of clay minerals, carbonate material, and other deleterious substances. If a more detailed description of the coating is needed, the coarse aggregate is examined under a stereomicroscope to determine the extent, chemistry, and adherence of the coating. Thin-section concrete samples can also be inspected, although aggregate coatings may be difficult to detect unless higher-powered microscopes are used (St. John et al. 1998).

A petrographic examination is useful in evaluating aggregate cleanliness because it can determine the feasibility of removing deleterious substances. By comparing the

composition of a surface coating to the rest of the aggregate, a petrographer can identify exactly what substances should be removed during washing (Mielenz 1994). Despite the value of this type of examination in studying aggregate coatings, it is not usually used as an aggregate acceptance test. The complexity of the equipment and the analysis procedure make the success of the tests dependent on the training and experience of the petrographer (Mielenz 1994). According to Dolar-Mantuani (1977), a petrographic examination is best used as a compliment to standard acceptance tests to verify results and identify additional testing needed.

2.4.4 X-ray Diffraction

X-ray diffraction identifies the mineral phases in a powder sample from the pattern of waves diffracted through a crystalline structure. During the test, x-rays are passed through the sample and collected by a detector, which measures the angle and intensity of the x-ray reflections (Gillot 1987). The spacing between planes of atoms in the crystal is calculated from the wavelength of the x-rays and the angle of diffraction according to Bragg's Law. The intensity of x-ray reflections at each interplanar spacing determines the diffraction pattern of the sample. By matching the diffraction pattern to a database of known patterns, the presence of each mineral or compound can be identified (St. John et al. 1998).

Due to the sophistication of the equipment, x-ray diffraction is not widely used to control aggregate coatings. However, it is a potentially valuable tool for research and forensic investigations because it can classify aggregate coatings as dust coatings, clay coatings, or carbonate coatings. Forster (1994) recommends using x-diffraction to separate clay minerals from fracture dust in coatings. According to Ozol (1994) and Dolar-Mantuani

(1983), it may also be helpful in identifying carbonate material susceptible to alkali-carbonate reactions. Recently, the test has been used in research in Iowa and Minnesota and has been effective in determining the mineralogy of suspect concrete aggregates (J. Parry: personal communication).

Besides the complex equipment, difficult procedural requirements may limit the use of x-ray diffraction as a practical method for identifying clay coatings. In order to detect the main types of clay minerals in a coating, special pretreatments are required to remove coarse grained non-clay particles and cementitious compounds from the sample. Without these pretreatments, other mineral phases can interfere with the orientation of the clay minerals and reduce their measured x-ray intensity (Pike 1992). However, even if a qualitative estimate of the clay content is determined, calculating the exact quantity of clay in the sample is very difficult. According to Gillot (1987), the percentage of clay in the sample can be estimated with an error of 10% by comparing the areas under the peaks in the diffraction using a rigorous standardized procedure.

2.4.5 Methylene Blue Adsorption Test (AASHTO TP 57)

Methylene blue adsorption uses the unique properties of clay minerals to measure the relative amount of clay in a sample of aggregate. The test procedure consists of titrating a mixture of water and p200 material with methylene blue dye, a cationic solution that is adsorbed to the surface of clay minerals (Bernsted 1985). During the titration, the dye is added to the mixture in small increments until the material no longer adsorbs the dye. This end-point is observed when a light blue halo surrounds a drop of the mixture after it has been

placed on filter paper. Based on the volume of dye adsorbed, a methylene blue value (MBV) is calculated for the sample.

$$\text{MBV} = \frac{\text{solution concentration} \times \text{titration volume}}{\text{sample mass}} \qquad (2\text{-}1)$$

Because the sample mass in Equation 2-1 represents the p200 material removed from an aggregate sample, the modified methylene blue value (MMBV) should be used to indicate the overall clay content of the aggregate (Tourenq and Tran 1997).

$$\text{MMBV} = \frac{\%\,\text{p200}}{100} \times \text{MBV} \qquad (2\text{-}2)$$

The methylene blue test is currently used in some European countries to monitor the cleanliness of concrete sands. In the French specifications, the methylene blue value of fines extracted from fine aggregate is limited to 10 mg/g (Pike 1992). In England, Pike proposed a maximum value equivalent to 20 mg/g after assessing the cleanliness and variability of over 200 washed and unwashed sands. It should be noted, however, that some question the use of the test as an aggregate specification because the ratio of methylene blue values listed in Table 2-1 (1:5:20) does not reflect the harmfulness of the clay fines to concrete strength (1:2:4 or 1:2:10), as described in Section 2.3.2 (Yool, Lees, and Fried 1998).

Table 2-1. MBV of Common Clay Minerals (Pike 1992)

Clay Mineral	MBV (mg/g)
Kaolinite	13
Illite	65
Montmorillonite	260

According to research conducted in Europe, the results of the methylene blue test may be used to predict the quality of concrete aggregates. Hosking and Pike (1985) tested 47 different aggregates in Great Britain and showed that the standard test for aggregate drying shrinkage correlates with the methylene blue value at a 99% significance level. This assessment agrees with the work of Hills and Pettifer (1985) who described the test as a good indicator of the swelling potential of road aggregate produced in Northern Ireland. In France, Unikowski (cited in Pike 1992) attempted to link the methylene blue value with the water demand of several types of clay fines used in mortar but found that the test overemphasized the adsorption of montmorillonite. Incidentally, research in the United States suggests that the water demand of fine aggregate is significant when the modified methylene blue value is greater than 0.30 (Ahn and Fowler 2000).

In the United States, the methylene blue test has mostly been used to predict the performance of asphalt pavements. According to Aschenberger (1992), the general relationship between the methylene blue value and performance shown in Table 2-2 can be used to judge the moisture susceptibility of asphalt aggregates.

Table 2-2. MBV in the Asphalt Industry (after Aschenberger 1992)

MBV (mg/g)	Expected Performance
5-6	Excellent
10-12	Marginally Acceptable
16-18	Problems or Possible Failure
20+	Failure

The methylene blue test is well suited to investigations of aggregate coatings because it can detect the presence of harmful clay particles. Adsorption of the dye involves an irreversible substitution of metallic cations for methylene blue in the structures of the particles (Taylor 1985). Since only clay minerals can exchange cations, the dye is adsorbed by clay coatings but not by dust and carbonate coatings, which consist of minerals like quartz, feldspar, dolomite, and limestone (Bernsted 1985). The amount of dye adsorbed is related to the activity and specific surface of the coating and may be used to determine its water demand. As a result, the methylene blue value can potentially be used to classify the aggregate coating.

Despite the potential of the methylene blue test to identify harmful aggregate coatings, its poor repeatability may limit its use as a quality control test. According to Pike (1992), the coefficient of variation associated with the repeatability and reproducibility of the test is 20% and 30%, respectively. Although error is introduced by the instability of the solution and the lack of standardized equipment, the greatest source of variation is operator

recognition of the light blue halo at the end of the test. However, the end-point can be easily observed by a spectrophotometer or a color comparator solution, which determine the amount of excess dye in the mixture (Taylor 1985; Pike 1992). Like x-ray diffraction, the methylene blue test is mostly qualitative, because it is extremely difficult to identify the types of clay minerals that are present from the test results.

2.4.7 Atterberg Limits

When water is added to clay material, cohesion develops between the clay particles and the material transforms from a solid to a viscous liquid. The Atterberg limits, as described below, represent the moisture contents at which the physical properties of the material change (Das 1998).

1. The shrinkage limit marks the transition from the solid state to the semi-solid state. It is measured at the point the sample does not shrink as moisture is lost.
2. The plastic limit marks the transition between the semi-solid state and the plastic state. It is measured at the point a 1/8 in. diameter sample begins to crumble when it is rolled.
3. The liquid limit marks the transition between the plastic state and the liquid state. In the Casagrande Method, it is determined at the point a 1/2" groove closes when the sample is dropped 25 times from a height of 0.3927 in. In the Cone Penetrometer Method, it is measured at the point a special cone assembly drops 20 mm into the sample in 5 seconds.

The plasticity index is defined as the difference between the liquid limit and plastic limit and represents the range of plasticity of the material.

Currently, the Atterberg limits are not used in concrete specifications to control the cleanliness of concrete aggregate. However, some researchers have used the liquid limit and plasticity index to predict the harmfulness of fines used in concrete and mortar. Buth et al. (1966) showed that in concrete fine aggregate the liquid limit of the p200 material can have dramatic effects on performance. When the liquid limit increases from 0 to 200, their research predicts a 20% decrease in compressive strength, a 40% decrease in modulus of rupture, and a 45% increase in drying shrinkage. On the other hand, Bonavetti and Irassar (1994) reported that the water requirement, workability, and shrinkage of concrete mortar are not affected when the plasticity index of a sand passing the No. 40 sieve is less than 4.

The Atterberg limits are related to the type and amount of clay minerals in the sample since the plasticity of a material is caused by the water held to the surface of the clay particles. Because clay minerals are concentrated in the clay-sized fraction (< 2 μm), the measured plasticity is lower if a significant amount of coarse particles is present. As a result, it is important to note whether the Atterberg limits are measured for samples passing the No. 40 sieve (commonly used in the geotechnical industry) or the No. 200 sieve (sometimes used in the asphalt industry). This difficulty has limited the use of the Atterberg limits in aggregate cleanliness specifications (Parry: personal communication).

2.4.7 Particle Size Distribution

Particle size distributions, obtained from hydrometer analyses or automated devices, indicate the particles sizes present in material passing the No. 200 sieve. For many years, the hydrometer analysis (ASTM D422) has been a standard size distribution method in soil engineering. In the test, the percentage of particles finer than a given diameter is calculated

from the time particles take to settle in solution. Automated devices are based on the absorption of lasers or x-ray beams by particles suspended in water.

In some cases, a particle size distribution can be used to distinguish between dust coatings and clay coatings. The dust generated by aggregate processing is generally coarser than 2 μm, although finer dust grains can be produced naturally in severe environments (Pike 1992). Since the clay minerals are smaller than 2 μm, it is generally believed that particles finer than this size can be classified as clay material. However, particle size distributions are generally not used to describe aggregate coatings because they cannot identify the specific types of clay minerals present. When particle sizes are needed, automated devices are usually used since the hydrometer analysis takes a few days to complete and is subject to considerable error.

CHAPTER 3 – METHODOLOGY

3.1 GENERAL INFORMATION

In general, testing was completed at the Wisconsin Structures & Materials Testing Lab (WSMTL) on the campus of the University of Wisconsin-Madison from January 2000 to July 2002. However, outside testing labs were contracted in some cases to conduct selected tests or verify certain results. All of the test procedures used in the study were adapted from current ASTM and DOT standards.

Because aggregate coatings are perceived to reduce aggregate quality and impair concrete performance, some of the information contained in this report may be commercially valuable to Wisconsin aggregate producers. It is not the intent of this study to endorse or discredit aggregates produced from any specific supplier or quarry. As a result, all of the aggregate sources that are cited are identified by region but not by specific quarry names.

3.2 SAMPLING OF AGGREGATE COATING

Coarse aggregate deposits susceptible to coatings were identified through a survey of concrete paving contractors and WisDOT district offices. A copy of the survey and a list of the participants can be found in Appendix A. In all, 24 of the 25 surveys sent out were returned, providing a service record of the locations and effects of aggregate coatings in Wisconsin since 1995. According to the survey results, seven of the eight WisDOT districts

outlined in Figure 3-1 have had previous problems with aggregate cleanliness, as described in Section 2.2. The counties shaded in the map represent the specific areas where aggregate coatings were reported to affect state concrete pavement projects.

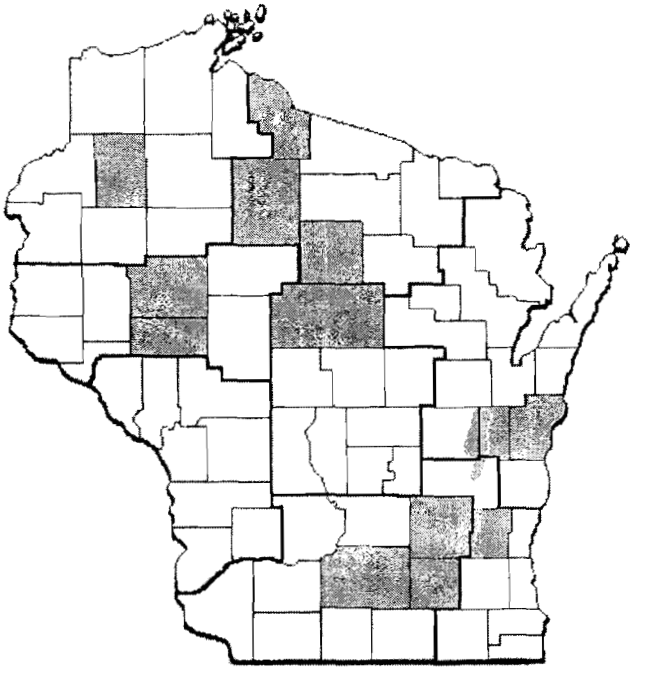

Figure 3-1. Suspected Locations of Aggregate Coatings

Based on the information collected in the survey, aggregate samples were obtained from the 10 locations shown in Figure 3-2 to get a general idea of the types of aggregate coatings found in Wisconsin. Most of the samples consisted of washed, ¾-in. aggregate, although samples of pit-run and unwashed material were also collected at some of the sites. After testing the samples to measure the extent and nature of any existing coatings, larger samples were collected from Source C, E, and H to evaluate the effects of the coated aggregate on concrete performance.

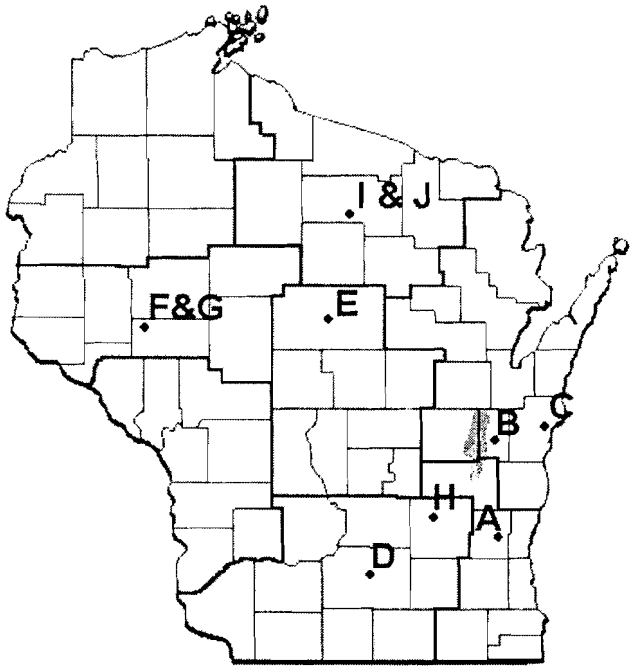

Figure 3-2. Locations of Sampled Aggregate Coatings

In many cases, sampling was hindered by the sensitivity of gravel producers and the variability of cleanliness within the deposit. For some of the locations in Figure 3-1 owners refused to participate in the study, and for others the sampled aggregate was virtually free of any adhering coating. The latter problem may derive from varying perceptions of what constitutes a deleterious aggregate coating or from the fact that coatings are often isolated in a specific seam of a deposit. To offset the sources that declined involvement in the research, samples were collected from alternative sites in the same region.

3.3 AGGREGATE COATING CHARACTERIZATION

Several of the characterization tests described in Section 2.4 were chosen to measure the properties of each aggregate coating. A summary of the tests conducted and the properties considered is provided in Table 3-1. Note that for many of the tests only indirect measurements could be made since the test results have not been directly correlated to the property tested or intricacies in the test complicate the measurement. Detailed descriptions of the test procedures are provided in the sections that follow.

Table 3-1. Summary of Coating Characterization Tests

Characterization Test	Property of Aggregate Coating Tested			
	Extent	Adherance	Mineralogy	Clay Content
Visual Examination	✓	✓		
Material Passing No. 200 Sieve	✓✓			
California Cleanness Test	✓			✓
Methylene Blue Adsorption			✓	✓
X-ray Diffraction			✓✓	✓

✓✓ = direct measurement ✓ = indirect measurement

3.3.1 Material Passing the No. 200 Sieve

The procedure used to determine the amount of material passing the No. 200 sieve conformed to ASTM C117, *Standard Test Method for Material Finer than 75 μm (No. 200 Sieve) in Mineral Aggregates by Washing.* In the test, the required sample mass was collected from a batch of oven-dried aggregate and placed in a stainless steel container. The

contents of the container were then charged with water, shaken vigorously, and decanted over a No. 200 sieve. The process was repeated until the resulting wash water was clear. After all the adhering fines were washed from the aggregate, the sample was thoroughly rinsed over a nest of No. 8 and No. 200 sieves. The material that was retained on each sieve was flushed into a drying pan and dried at 230°F to compute the p200 percentage, while the fines that passed through the No. 200 sieve were discarded.

3.3.2 California Cleanness Test

The California cleanness test was conducted in general compliance with the standard procedure in Caltrans Test No. 227, *Method of Test for Evaluating Cleanness of Coarse Aggregates*. During the test, a 2500-g sample of oven-dried aggregate was placed in a stainless steel washing vessel with 1000 g of distilled water. After the aggregate soaked for 1 minute, the vessel was clamped shut and agitated in a sieve shaker for 2 minutes. The fines in the container were then brought into suspension and passed through a No. 8 and No. 200 sieve nested over an 8-in. diameter collection pot. To ensure that all the p200 material was collected, parts of the wash water were passed through the No. 200 sieve until the water flowed freely through the sieve. Subsequently, a sand equivalent test cylinder, marked with 150 graduations, was filled to the 3-unit mark with a calcium chloride solution and to the 150-unit mark with the wash water. After the mixture was allowed to stand for 20 minutes, the sediment height in the cylinder was recorded and translated into a cleanness value, according to the tabulated relationship in the standard.

3.3.3 Methylene Blue Adsorption Test

The procedure of the methylene blue adsorption test was based on AASHTO TP 57, *Methylene Blue Value for Clays, Mineral Filler, and Fines*, which is currently being used in several states to test asphalt aggregates (Kandhal and Parker 1998). Prior to testing, the p200 coating was collected by mechanically washing the coarse aggregate according to Caltrans Test No. 227 and evaporating the wash water at 140°F. For each test, 10 g of the removed p200 material was dispersed in 30 g of distilled water using a magnetic stirrer. The suspension was then titrated with a 5-mg/mL methylene blue solution added from a buret in 0.5-mL increments. After the suspension was stirred for 1 minute, a drop of the slurry was sampled using a glass stirring rod and placed on a piece of filter paper to observe the appearance of the drop. A light blue halo around the drop indicated that the suspended fines were no longer absorbing dye, i.e. the end-point of the titration. When the halo was first observed, the sample was stirred for 5 minutes and tested again. Smaller increments of solution were added as needed until the halo remained visible for 5 minutes. After reaching the end-point of the titration, the total volume of added dye was recorded and used to calculate the methylene blue value and modified methylene blue value using Equations 2-1 and 2-2.

Special precautions were taken during testing to reduce the variability of the test results. According to Pike (1992), the greatest sources of error in the experiment involve recognizing the end-point and maintaining the concentration of the solution. The end-point is particularly difficult to distinguish for inexperienced researchers because it often appears as a fuzzy blue layer around the test drop instead of a light blue halo (Hills and Pettifer 1985). In

these cases, the filter paper was held up to a 60W light bulb or observed through a magnifying glass to assist in determining the end-point. Due to the instability of the methylene blue dye, solutions were stored in a dark cabinet in amber bottles and mixed for 10 minutes before testing to maintain the measured concentration. Although some suggest that the shelf life of the solution is four to six months, methylene blue solutions that were prepared were only stored for two months or less.

3.3.4 X-ray Diffraction

The method used for x-ray diffraction analysis was based on a general test procedure developed by the S.W. Bailey X-ray Diffraction Laboratory at the University of Wisconsin-Madison. Before the analysis, p200 material from the aggregate coating was ground in acetone with an agate mortar and pestle. As in the methylene blue adsorption test, these samples were collected by mechanically washing the coarse aggregate and evaporating the resulting wash water. After carefully mounting the acetone slurry onto a glass slide, the sample was scanned over a 2θ range from 5° to 65° at a rate of 1° per minute using a Scintag PadV X-ray diffractometer, Ni-filtered Cu-Ka radiation (40 kV, 35 mA), and liquid-nitrogen cooled germanium detector. During the analysis, the Scintag program DMNST was used for data acquisition and peak identification. Unknown crystalline phases present in the sample were identified by matching the diffraction pattern to the patterns stored in the International Center for Diffraction Data (ICDD) database.

3.4 MIXING PLAN

Of the 10 sources that were initially sampled, aggregates from Source C, E, and H were chosen for mixing based on the results of the aggregate coating characterization tests (see Section 4.2.4). In the original testing plan, the effect of each surface coating was evaluated by comparing concrete containing the coated aggregate as sampled in the field (Coated Aggregate Series - Field) and concrete containing the same aggregate after the coating was removed in the lab (Washed Aggregate Series - Lab). After preliminary results showed only minor differences between these two series of mixes, however, a third series (Coated Aggregate Series - Lab) was added by manufacturing more severe aggregate coatings with silt and clay material. A summary of the different aggregate sources and concrete batches included in the testing program is provided in Table 3-2.

Table 3-2. Summary of Mixing Plan

Coated Aggregate Series Field		**Washed Aggregate Series Lab**		**Coated Aggregate Series Lab**	
Batch #	Source	Batch #	Source	Batch #	Source
1	C	4	C	7	E
2	E	5A	E	8	E
3	H	5B	E	9	E
		6	H	10	E

3.4.1 Coated Aggregate Series - Field

Batches 1-3 included the coated aggregates sampled from aggregate stockpiles at Source C, E, and H. As described in Section 4.2.4, these sources were selected in order to vary the p200 levels and clay contents of the aggregate coatings evaluated in the mixing plan. A brief description of each aggregate is provided below; sieve analyses are provided in Appendix B.

1. Aggregate C: fractured, dolomitic gravel essentially free of any surface coatings
2. Aggregate E: fractured, granitic gravel containing a coating with minor amounts of non-swelling clays and trace amounts of montmorillonite
3. Aggregate H: smooth, dolomitic river gravel containing an extensive carbonate coating

3.4.2 Washed Aggregate Series - Lab

Batches 4-6 tested aggregates C, E, and H after the adherent coatings were detached with a washing method modeled after Caltrans Test No. 227. During the procedure, approximately 100 lb of the coated aggregate was placed in a 1.5-ft^3 mixer with 50 lb of water. After the aggregate soaked in the mixer for 1 minute, the drum was positioned at an angle of 45° and allowed to rotate for 2 minutes. Subsequently, the suspended fines were decanted from the aggregate and a fresh 50 lb of water was poured into the mixer. Following six washing cycles, any remaining fines were removed from the aggregate by rinsing the sample over a No. 100 sieve cloth. The material that was washed through the sieve was discarded except for a few small samples collected for methylene blue tests. For each batch, a total of 400 lb of aggregate was washed and air-dried in a mixing pan lined with a plastic sheet before mixing.

3.4.3 Coated Aggregate Series - Lab

Batches 7-10 contained aggregate from Source E that was artificially coated with dust and clay fines to increase the extent or clay content of the coating[1]. For Batches 7 and 8, the manufactured coating consisted of stone dust collected from a sedimentation pond at a Barron Co. gravel pit. These two batches varied in the amount of material added to the aggregate – the measured p200 values of the aggregates were 1.4% and 2.0%, respectively. For Batches 9 and 10, the attached coating consisted of a clayey material that was dry sieved from a Sauk Co. soil. While the p200 values of the aggregates used in these two batches were similar, the total amount of water added in each batch was different. Because the clay material reduced the slump of Batch 9 to ¼-in., additional water was added to Batch 10 to correct for the absorption of the coating and match the slump of other batches mixed with the Source E aggregate.

The procedure used to create the manufactured coatings consisted of mixing the aggregate and a certain amount of the Barron Co. or Sauk Co. material to reach a desired p200 value. In each case, 400 lb of the coarse aggregate was placed in a 3-ft^3 mixer with enough water to form a visible film around each aggregate. Once the mixer was turned on, the measured amount of fines was sprinkled on the aggregate along with an additional 10% of material to offset the material that adhered to the drum of the mixer. During mixing, the drum was positioned at an angle to limit the abrasion of aggregate against the sides of the mixer. After the fines were evenly dispersed, the coated aggregate was placed on a plastic

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[1] Refer to Section 4.3 for a more detailed characterization of the fines used to manufacture these coatings.

sheet and air-dried to match the moisture contents of the aggregate used in the first two series of mixes.

3.4.4 Mix Design

Mix proportions were based on the WisDOT Grade A concrete mix design, as shown in Table 3-3 (WisDOT 1996). For each test batch, material quantities were determined to yield 3 ft^3 of concrete with a net water-cement ratio of 0.45 and a target air content of 6.0 ± 1.0%. The following materials were donated by local suppliers and used throughout the study:

1. Type I cement from LaFarge Corp.
2. Coarse aggregate from Sources C, E, and H
3. Crushed limestone fine aggregate from Wingra Stone Co.
4. Saponified rosin air entrainer (Daravair 1400) from Grace Products

The cement composition and aggregate gradations are included in Appendix B, and the batch quantities are shown in Appendix C.

Table 3-3. Wisconsin Grade A Concrete Mix Design

Material	Batch Weights (lb/ft^3)	Batch Proportions
Cement	20.9	1
Coarse Aggregate	69.4	3.5
Fine Aggregate	46.3	2
Water (Net)	9.4	0.45

To ensure a constant water-cement ratio, the amount of water added in each concrete batch was adjusted by the absorption (ASTM C127, C128) and moisture contents (ASTM 566) of the aggregates. While the moisture content of the fine aggregate was controlled by oven drying prior to batching, coarse aggregates were allowed to air dry to a moisture content between 1% and 2% in order to reduce the handling of the aggregate coatings. Because the mix proportions listed in Table 3-2 are based on dry aggregate weights, the measured moisture content also slightly affected the quantity of coarse aggregate batched for each mix. During mixing, the total weight of water was added in one fixed amount rather than incrementally to control workability.

The water demand of the aggregate coatings, especially the Coated Aggregate Series prepared in the lab, frustrated the control of the mixing water. Typically, adherent dust and clay particles are ignored when determining aggregate absorptions. For instance, ASTM C127 requires that coatings be washed from the aggregate surface before the absorption is measured. This material, however, has been shown to increase the water requirement of the aggregate depending on the quantity and mineralogy of the adhering particles (see Section 2.2.2). For the aggregates used in this study, it was assumed that the surface coatings did not significantly affect the net water-cement ratio. However, this assumption was checked using ASTM C127 and other test methods when the coarse aggregate approached the 1.5% p200 limit listed by WisDOT specifications.

3.5 MIXING PROCEDURE

Concrete batches were machined mixed in a 3-ft^3 drum mixer using the procedure specified in ASTM C192. For each test batch, materials were added in the following sequence: coarse aggregate, water containing air entrainer, fine aggregate, and cement. Immediately after the water was added, the drum was allowed to rotate for 10 seconds in order to activate the air entrainer. Any loosely adhering coatings may have been displaced from the coarse aggregate during this period of agitation. Each batch was mixed for 3 minutes, allowed to rest for 3 minutes, and mixed for an additional 2 minutes before the concrete was emptied from the mixer. As recommended in the standard, the drum was "buttered" with a 1-ft^3 batch prior to mixing to compensate for the mortar lost to the sides of the mixer.

Once mixed, the fresh concrete was tested for air content, unit weight, and slump. These properties were controlled during testing in order to limit the variability among the batches in the mix plan. Air content was measured with a Type B air meter and adjusted with the aggregate correction factor, following the standard procedure in ASTM C231. Before taking the measurement, the 0.25-ft^3 bucket of the air meter was used to calculate the unit weight. Slump was determined according to ASTM C143 and used to estimate the differing water demands of the aggregate coatings.

After testing the fresh concrete, the following specimens were cast based on the methods described in ASTM C192.

1. Six 4-in. by 6-in. cylinders for compressive strength and permeability tests
2. Four 6-in. by 12-in. cylinders for tensile strength tests

3. Three 4-in. by 4-in. by 11-in. prisms for shrinkage tests
4. Five 3-in. by 4-in. by 15-in. prisms for freeze-thaw durability tests

Reusable metal forms were used to prepare the concrete prisms, while disposable plastic molds with plastic lids were used to prepare the concrete cylinders. As soon as the test specimens were cast, they were covered with wet burlap and a plastic sheet to retain moisture as the concrete hardened. Forms were removed after 1 day and cured in a wet room at a temperature of 74°F and a relative humidity of about 100% until the specimens were tested.

3.6 HARDENED CONCRETE TESTS

Concrete performance was assessed by measuring the strength, durability, tendency for cracking, and air void distribution of each test batch. The hardened concrete tests used to determine these properties are summarized in Table 3-4.

Table 3-4. Summary of Hardened Concrete Tests

Test	ASTM Standard	Tests per Batch	Duration of Curing
Compressive Strength	C39	4	28 days
Tensile Strength	C496	4	28 days
Drying Shrinkage	C490	3	14 days
Freeze-Thaw Durability	C666	3	28 days
Rapid Chloride Ion Penetrability	C1202	2	28 days
Petrographic Examination	C857	1	28 days
Air Void Analysis	C457	1	28 days

3.6.1 Compressive Strength

The compressive strength of each concrete batch was determined by testing four 4-in. by 8-in. concrete cylinders after 28 days of wet curing. Before testing, the average diameter of each specimen was measured with a pi tape and used to calculate its cross-sectional area. Both ends of the cylinder were then capped with a sulfur compound to provide an even testing surface. During testing, specimens were loaded in axial compression at a constant rate of 1900 psi/min. After the cylinder failed, the maximum compressive stress in the concrete was calculated by dividing the recorded peak load by the cross-sectional area of the specimen. The average compressive strength of the four specimens was used to determine the compressive strength of the test batch.

Because air content is related to the porosity of the concrete, changes in air content can have significant effects on concrete strengths. In order to compare the strengths of batches with different air contents, the maximum compressive stress of each specimen was adjusted using the following equation:

$$\text{Corrected Strength} = \text{Measured Strength}\left(\frac{1-0.05\times 6\%}{1-0.05\times \text{Air Content}(\%)}\right) \qquad (3\text{-}1)$$

This correction, recommended by the American Concrete Institute, is based on the assumption that the compressive strength of air-free concrete is reduced by 5% for every 1% of air in the fresh concrete (Popovics 1998). Equation 3-1 slightly decreases or increases the

recorded compressive strength for mixes with air contents higher or lower than the target air content of 6%.

3.6.2 Tensile Strength

Concrete tensile strength was measured using the split-cylinder tension test described in ASTM C496. For each batch, four 6-in. by 12-in. cylinders were subjected to a compressive line load applied at a constant rate of 150 psi/min along the vertical diameter of the cylinder. Cardboard bearing strips were used to ensure that the load was transferred uniformly to the specimen surface. The load at failure was recorded and used to calculate the tensile strength using Equation 3-2:

$$\text{Tensile Strength} = \frac{2 \times \text{Peak Load}}{\pi \times \text{Length} \times \text{Diameter}} \tag{3-2}$$

In the data analysis, comparisons between test batches were made using the average tensile strength of the four specimens. Since no empirical relationship was found between the splitting tensile strength and air content in published research, the test results were not adjusted for varying air contents.

3.6.3 Drying Shrinkage

Drying shrinkage was used to assess the swelling nature of the aggregate coatings and the cracking potential of the concrete. Using the requirements of ASTM C490, tests were conducted on 4-in. by 4-in. by 11-in. concrete prisms with steel studs inserted at each end to provide a gage length of 10 $\pm$ 0.10 in. The gage length, or distance between the studs in the

concrete, was measured with a micrometer after tests were completed in order to calculate shrinkage more accurately. After 14 days of wet curing, the specimens were maintained in an environmental chamber at a temperature of 74°F and a relative humidity of 50%. Changes in length were recorded relative to an initial reading at various ages between 14 and 120 days using a length comparator gauge with a precision of 0.0001 in. After each reading, the percent shrinkage was calculated by dividing the relative length change by the gage length. The average 56-day shrinkage of three specimens was used to analyze the test results.

3.6.4 Freeze-Thaw Durability

Based on ASTM 666 Procedure A, durability was measured from the weight and stiffness degradation of concrete subjected to cycles of freezing and thawing. The curing conditions recommended in the standard (14 days in lime water) were modified slightly (28 days of wet curing) in order for the concrete to reach a strength level more representative of field conditions. Samples were placed in a freezer following the curing period to prevent loss of moisture and control cement hydration up to the time of testing. Once testing was ready to commence, the specimens were thawed in air for 24 hours and soaked for another 24 hours in a 3% NaCl solution. For each test batch, three specimens were kept immersed in solution and cycled between 1 °F and 50 °F in a freeze-thaw chamber at an average rate of 4.5 cycles per day. The temperature was regulated with thermocouples that were inserted into the center of a control specimen placed in the middle of the freeze-thaw chamber.

The weight and fundamental transverse frequency were recorded for each specimen every 7 days, or roughly every 30 cycles. These measurements were used to calculate the relative changes in weight and dynamic modulus of elasticity with respect to the initial

readings. The average changes for the three specimens at 100 and 300 cycles were interpolated from the data collected and used to make comparisons between test batches.

3.6.5 Rapid Chloride Ion Penetrability

The rapid chloride ion penetrability test (ASTM 1202) was used as an indicator of the permeability of each concrete batch. For the test, two 2-in. thick specimens were cut from a 4-in. diameter cylinder with a diamond blade saw. Each of the specimens was conditioned prior to testing with an epoxy coating to seal pores on the outer surface and a vacuum desiccator to remove air from the sample. During vacuum conditioning, the internal pressure of the specimen was reduced to less than 1-mm Hg for 3 hours in a dry condition and an additional 1 hour under de-aerated water. The specimen was then allowed to soak in the water for 18 hours before the test was conducted. The test procedure consisted of creating a voltaic cell by sandwiching the specimen between solutions of NaCl and NaOH and driving an electric current through the concrete. The total charge passed in 6 hours is related to the chloride ion penetrability of the specimen, as shown in Table 3-5.

Table 3-5. Prediction of Chloride Ion Penetrability (ASTM 1202)

Charge Passed (Coulombs)	Chloride Ion Penetrability
> 4000	High
2000 - 4000	Moderate
1000 - 2000	Low
100 - 1000	Very Low

3.6.6 Petrographic Examination

Petrographic concrete thin-sections were prepared by the University of Wisconsin-Madison Department of Geology and studied under a stereomicroscope following the guidelines specified in ASTM 856. Prior to preparing the specimens, samples were cut in the laboratory from concrete prisms with a cross-section of 3-in. by 4-in. Each specimen was then impregnated with an epoxy resin, ground to a thickness of 30 μm, and mounted on a 2-in. by 3-in. glass slide. Using a high-powered stereomicroscope, the specimens were examined at magnifications up to 400x. Specific characteristics that were examined included the quality of the aggregate-cement paste bond and the presence of microcracking in the cement paste.

3.6.7 Hardened Air Void Analysis

A petrography firm was contracted to analyze the hardened air void system for each test batch. Samples were cut in the laboratory into 2-in. thick slices with a surface area of 12 in^2 and tested using the linear-transverse method prescribed in ASTM C457, Procedure A. In each test, the following air void parameters were measured: hardened air content, specific surface, void frequency, spacing factor, and paste-air ratio. As is frequently done in research, only one sample was analyzed for each batch in the mixing plan. However, three samples were tested for Batch 2 to estimate the variability of the air system in any one specimen.

CHAPTER 4 – RESULTS OF AGGREGATE COATING TESTS

4.1 GENERAL INFORMATION

Before investigating the effects of aggregate coatings on concrete performance, the mineralogy and extent of each coating were determined with the characterization tests described in Chapter 3. The results of these tests were used to identify the coating types prevalent in Wisconsin in Phase II and to examine correlations between the test parameters and hardened concrete performance in Phase III. As discussed previously, the classification of coatings into dust, clay, or carbonate coating types is crucial to understanding their effects on concrete properties.

4.2 PHASE II CHARACTERIZATION

The objective of the Phase II characterization tests was to determine the mineralogy of aggregate coatings found in Wisconsin. This was accomplished by collecting coarse aggregate samples from the 10 locations shown in Figure 3-2 and analyzing the surface coatings with x-ray diffraction. Because this analysis is mostly qualitative, the California cleanness test and methylene blue adsorption test were included to quantify the differences in mineralogy among the aggregate coatings. Based on the characterization of each coating, a crude map was created to show the locations of different types of aggregate coatings in the state and used to select the aggregates for concrete testing in Phase III.

4.2.1 X-Ray Diffraction

The unknown mineral phases present in each aggregate coating were identified by x-ray diffraction. This information was particularly valuable in evaluating the cleanliness of the aggregates because it directly classified the coatings as dust, clay, or carbonate coatings. However, the analysis was complicated by the fact that each coating consisted of several different mineral phases. Since the x-ray diffraction peaks of the identified phases often interfered with each other, it was difficult to estimate the proportions of the minerals present. It was also probable that some of the minerals impaired the diffraction characteristics of the clay minerals by cementing the clay particles together. Due to such complexities, the test is not widely used to monitor aggregates in the field.

The x-ray diffraction plots of the coatings investigated in Phase II are shown in Appendix D, and the minerals identified in each sample are summarized in Table 4-1. In general, most of the coatings contained feldspar or carbonate material that was consistent with mineralogy of the aggregate itself. While dolomite and anorthite coatings were identified on the dolostone aggregates in southern Wisconsin, albite and amphibole coatings were identified on the granite aggregates in northern Wisconsin. These materials can be classified as stone dust and should be distinguished from the clay material also found on the granite aggregates. The chlorite and illite clay identified in these aggregates are likely associated with the weathering of deposits in the northern portions of the state (D. Mickelson, personal communication). More detailed information on the composition and occurrence of each mineral is provided below based on the descriptions provided by Perkins (1998).

Table 4-1. Phase II Results of X-Ray Diffraction Analysis (Refer to Figure 3-2 for source locations)

Source	Identified Minerals in Order of Intensity				
A	Dolomite	Quartz	Anorthite		
B	Dolomite	Quartz	Anorthite		
C	Dolomite	Quartz			
D	Dolomite	Quartz	Anorthite		
E	Quartz	Albite	Chlorite	Illite	Amphibole
F	Quartz	Albite	Chlorite	Amphibole	
G	Quartz	Albite	Chlorite	Illite	Amphibole
H	Dolomite	Quartz	Anorthite	Chlorite	Amphibole
I	Quartz	Albite	Chlorite	Amphibole	
J	Quartz	Albite	Chlorite	Amphibole	

Albite

Albite, sodium aluminum silicate, is a feldspar mineral and a major component of igneous rocks, especially granite. It is associated with minerals such as quartz and other feldspars.

Amphibole

Amphiboles represent a group of minerals associated with many igneous and metamorphic rocks. Similar to albite, they are often associated with quartz and feldspar minerals.

Anorthite

Anorthite, calcium aluminum silicate, is a feldspar mineral found in igneous rock and metamorphic carbonate rocks. Its occurrence is less common than the other feldspar minerals.

Chlorite

Chlorite is a clay mineral commonly found in metamorphic rocks. Although its crystal structure slightly differs from the other clay minerals, it is typically grouped as a clay because of its platy structure. Chlorites have a surface charge density similar to illite and are closely associated with mica (Das 1998).

Dolomite

Dolomite is a carbonate mineral and a major component of dolostone, a frequently used concrete aggregate. Its crystal structure contains a layer of carbonate ions between layers of calcium and magnesium ions. Although dolomite is durable, it has been associated with alkali-carbonate reactions in concrete when it occurs with certain clay minerals (Ozol 1994).

Illite

Illite is a clay mineral frequently found in soils and several different rocks. Its structure is similar to montmorillonite but has less swelling capability due to stronger bonding. Associated minerals include muscovite and feldspar.

Quartz

Quartz is the most common mineral and an important constituent of igneous, metamorphic, and sedimentary rocks. Unlike other minerals, quartz is extremely durable and virtually unaffected by chemical weathering processes. However, it can react with alkalis in the cement when it is found as a clay-sized particle (Dolar-Mantuani 1983).

4.2.2 Cleanness Values

The California cleanness value was used to measure the amount of material adhering to each coarse aggregate sample. While the test parameter is related to the percentage of p200 material in the aggregate, it is specifically associated with the clay fraction. A lower cleanness value is measured for an aggregate with a p200 fraction containing a large quantity of clay material because the volume of these particles is magnified by the solution used in the test. According to the Caltrans specifications, coarse aggregates must have a cleanness value greater than 75 to be suitable for concrete construction.

The cleanness values of the Phase II aggregate samples are plotted in Figure 4-1. As shown in the figure, most of the values ranged between 90 and 95, which is well within the Caltrans specification limit. The lowest cleanness values, 89 and 85, were measured for the aggregate from Source E and Source H, respectively. This follows from the larger quantity of adherent material that was observed on these aggregates and the x-ray diffraction results, which identified small amounts of clay material in each coating. Although clay minerals were also found in the Source F and G aggregate coatings, the quantity of clay was too small to influence the cleanness value.

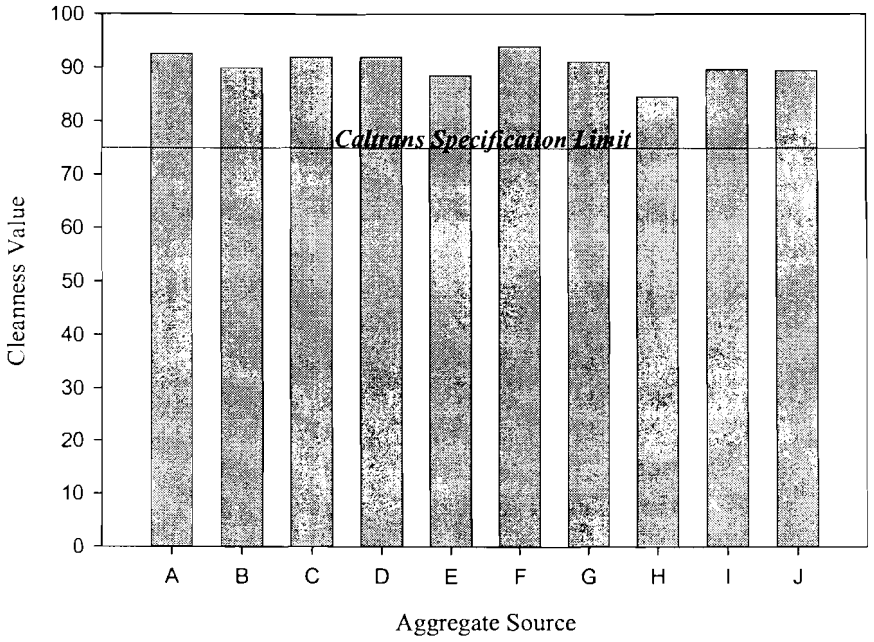

Figure 4-1. Phase II Results of California Cleanness Test

4.2.3 Methylene Blue Values

The clay content of each coating was assessed with the methylene blue value. Since the adsorption of methylene blue dye is related to the specific surface and cation exchange capacity of the minerals contained in a sample, the value can be used to distinguish between clay and non-clay coatings. Generally, high methylene blue values (greater than 10 mg/g) represent significant amounts of clay particles, which have a larger specific surface and higher surface charge than dust particles. Although the methylene blue value has not been used as a specification for coarse aggregates, a value equivalent to 10 mg/g is specified for fine aggregates in some European countries (Pike 1992). It should be noted that most specifications use the modified methylene blue value (Equation 3-2) because the test is conducted on the p200 sample and is not directly related to the extent of the coating.

The methylene blue values shown in Figure 4-2 confirm the differences in mineralogy between the coatings found in the northern and southern parts of the state. While the methylene blue values of the dolomite coatings were less than 3 mg/g, the methylene blue values of the feldspar coatings were greater than 5 mg/g. The exception was the coating on the unwashed aggregate from Source H, which had a methylene blue value of 9.3 mg/g despite consisting predominantly of dolomite. It is assumed that the higher values represent concentrations of the chlorite and illite minerals that were identified in many of the samples.

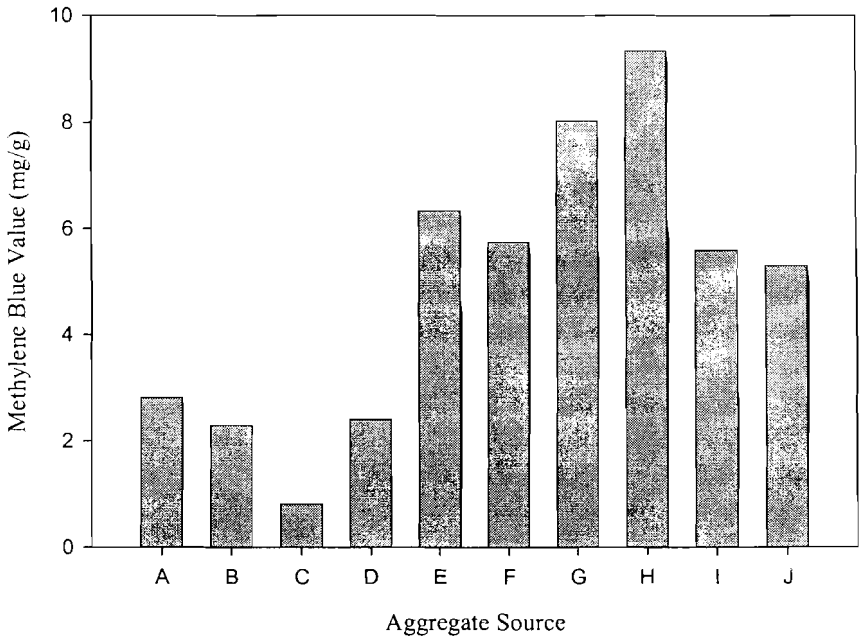

Figure 4-2. Phase II Results of Methylene Blue Adsorption Test

In spite of the high methylene blue values measured for the coatings from Sources G and F, the clay content is not as significant because the cleanness value of both aggregates was high. The variation in methylene blue values suggests that some locations may be more susceptible

to clay coatings than other locations. Because weathering is a major factor in clay formation, clay coatings are often limited to sources or seams in a deposit where leaching or overburden is extensive.

4.2.4 Summary

The results of the characterization tests indicate that aggregate coatings consist largely of particles that have the same mineralogy as the aggregate deposit. The coatings in southern Wisconsin contain mostly carbonate minerals, and the coatings in northern Wisconsin contain major amounts of feldspar minerals and minor amounts of clay. Essentially, these minerals are the basic rock-forming minerals of the aggregate types found in Wisconsin. Dolomite, quartz, and anorthite are important constituents of the dolostone and quartzite deposits in the southern part of the state; albite, quartz, and amphibole are important constituents of the igneous deposits in the northern part of the state. The clay minerals are most likely associated with the slow disintegration of the igneous rocks. Since the mineralogy of the coatings appears to be closely related to the type of aggregate, the map of the major aggregates produced in Wisconsin (see Figure 2-1) can be modified to show the expected mineralogy of aggregate coatings in the state (see Figure 4-3).

It is unclear whether the occurrence of aggregate coatings can be linked to the quality of washing procedures in the field. Although all of the samples, except for the Source H aggregate, were collected after the stone was washed and graded, coatings were visible on the surface of each aggregate. It is likely that most of the dust coatings were generated during the processing, crushing, and handling of the aggregates after they were washed. On the other hand, the clay coatings were created by weathering processes and remained attached to

the aggregate after washing. Since the coatings consisted predominantly of stone dust and only partially of clay, the measured cleanness values were well within the specification limit. Even if clay adheres to the aggregate after the concrete is placed, the clay contents of the coatings appear to be small and unlikely to affect performance.

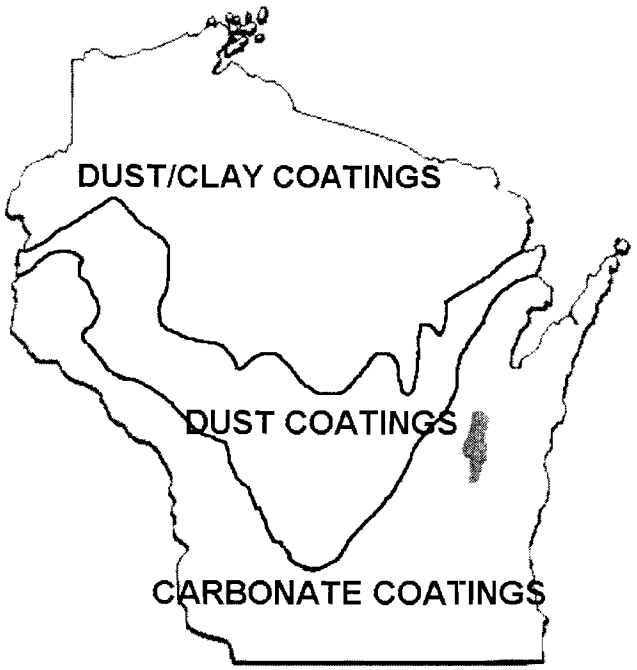

Figure 4-3. Mineralogy of Wisconsin Aggregate Coatings

Based on the Phase II characterization tests, a subset of the sampled aggregates was selected to study the effects of aggregate coatings on concrete performance. The criteria used to select these aggregates included the extent of the surface coatings and the mineralogy of the adherent material. Specifically, aggregates and coatings were chosen to vary the cleanness values and methylene blue values that were measured in Phase II. The coating types and aggregate sources selected for further study in Phase III are described below:

1. The carbonate coating from Source C contained significant amounts of dolomite. However, the coating was not extensive and did not contain clay minerals.
2. The dust/clay coating from Source E contained significant amounts of feldspar dust and minor amounts of clay. Compared to the other aggregates sampled in northern Wisconsin, the Source E aggregate had a lower cleanness value and a higher methylene blue value. To test the impact of more extensive dust and clay coatings, samples of fine material were collected and added to the aggregate.
3. The carbonate/clay coating from Source H consisted significant amounts of dolomite and minor amounts of clay. Unlike the other samples, the aggregate was sampled before it was washed on-site. As expected, it had the lowest cleanness value and highest methylene blue value of all the other aggregates.

4.3 PHASE III COATING CHARACTERIZATION

The purpose of the Phase III characterization tests was to measure the extent and mineralogy of the coatings that adhered to the washed and coated aggregates used for concrete mixing. As in Phase II, these properties were primarily assessed with the California cleanness value and methylene blue value. However, the percentage of p200 material and modified methylene blue value were also measured to compare the cleanliness of the aggregates to requirements specified in current standards or recommended in previous research. After determining the nature of each aggregate coating, the results were used to estimate their water demand.

4.3.1 Test Results

Table 4-2 shows the characterization results of the aggregates and coatings used in each concrete batch. The modified methylene blue values listed in the table were calculated using Equation 3-2 to estimate the overall clay content of the aggregate. The results are grouped by aggregate source so the differences between the coated and washed aggregates can be compared easily.

Table 4-2. Phase III Results of Coating Characterization Tests

Batch	Concrete Series	p200 (%)	Cleanness Value	MBV (mg/g)	MMBV (mg/g)
Source C Aggregate					
1	Coated - Field	0.3	94	0.8	0.002
4	Washed - Lab	0.1	97	0.8	0.001
Source E Aggregate					
2	Coated - Field	0.7	85	6.4	0.05
5A	Washed - Lab	0.2	97	5.1	0.009
5B	Washed - Lab	NA	NA	NA	NA
7	Coated - Lab[1]	1.4	82	4.0	0.05
8	Coated - Lab[1]	1.9	79	3.7	0.07
9	Coated - Lab[2]	1.4	23	8.7	0.12
10	Coated - Lab[2]	1.3	14	11.4	0.15
Source H Aggregate					
3	Coated - Field	0.9	86	6.0	0.06
6	Washed - Lab	0.2	97	3.4	0.006

[1] Coatings manufactured with dust fines from Barron Co.

[2] Coatings manufactured with clay fines from Sauk Co.

Source C Aggregate

The low p200 percentage and high cleanness value of the Source C aggregate indicate that only a small amount of material adhered to the aggregate surface. Since the p200 percentage was 0.3%, which is significantly lower than the WisDOT specification limit of 1.5%, it is unlikely that the coating would cause any noticeable changes in the concrete. The low methylene blue value suggests that the coating is predominantly dolomite and provides further evidence that the coating is innocuous. This carbonate material may even be beneficial to the concrete performance given that no clay was detected in the coating. Because of the cleanliness of the aggregate, washing did not significantly change the amount or clay content of the adherent material.

Source E Aggregate

The cleanness value and methylene blue value of the coated aggregate imply that the Source E coating contains moderate amounts of clay material. However, it is uncertain if the coating is harmful because the cleanliness parameters are within specification requirements. Although minor amounts of illite and chlorite were identified in the coating in Phase II, the cleanness value of 85 and the methylene blue value of 6.4 mg/g do not warn of any deleterious effects. To increase the extent and clay content of the coating, dust fines from Barron Co. and clay fines from Sauk Co. were added to the aggregate in Batches 7-10. The mineralogy of each sample, determined by x-ray diffraction, is shown in Table 4-3.

Table 4-3. X-Ray Diffraction Results of Phase III Fines

Sample	Identified Minerals in Order of Intensity
Barron Co.	Quartz Albite Anorthite Labradorite Microcline
Sauk Co.	Quartz Muscovite Anorthite Chlorite-Vermiculite-Montmorillonite

Effectively, the feldspar minerals identified in the Barron Co. fines and the clay minerals identified in the Sauk Co. fines modified the original dust/clay coating into a dust coating and a clay coating, respectively. The nature of these coatings is reflected in the results of the characterization tests tabulated for Batches 7-10. The most significant changes in the test parameters were associated with the additions of clay fines in Batch 9 and Batch 10. In particular, the cleanness value of both aggregates was well below the Caltrans specification limit, which proves that the test is sensitive to clay particles. Similarly, the modified methylene blue values (~0.15 mg/g) were considerably higher than the value measured for the coated aggregate in Batch 2. According to Ahn and Fowler (2000), fine aggregate with a modified methylene blue value of 0.30 mg/g can significantly increase the water demand of the aggregate. Considering that concrete contains more coarse aggregate than fine aggregate, the clay contents of these aggregates may be high enough to frustrate the control of mixing water. Unlike the additions of clay fines, the additions of dust fines in Batches 7 and 8 did not significantly affect the test parameters. Both the cleanness value and modified methylene blue value were similar to the values measured for Batch 2. Interestingly, the cleanness value of the Batch 8 aggregate was within the Caltrans

specification even though the p200 percentage was greater than the WisDOT limit, but the opposite was true for the aggregates used in Batches 9 and 10.

Source H Aggregate

Both the p200 percentage and methylene blue value of the coated Source H aggregate were high because the aggregate was not washed on-site before it was sampled. Despite the severity of the coating, the cleanliness of the aggregate washed in the lab was similar to the cleanliness of the other washed aggregates. The significant decrease in methylene blue value after washing was most likely associated with the removal of chlorite minerals during the washing procedure. Incidentally, the resulting methylene blue value (3.4 mg/g) closely matches the values measured for the dolomite coatings of Sources A, B, and D in Phase II. It appears that washing of dolostone aggregate commonly found in southern Wisconsin can slightly reduce the clay content of the aggregate.

4.3.2 Coating Classifications

Overall, the properties of the aggregates and coatings tested in Phase III were very similar to the properties measured in Phase II. As a result, it was assumed that the mineralogy of the coated aggregates obtained from Source C, E, and H during a repeat sampling also remained the same. Based on the x-ray diffraction analysis of the coatings in Phase II and the added fine material in Phase III, the coated aggregates could be classified according to the aggregate types described in Section 2-1. As shown in the Table 4-4, each of the basic aggregate coating types was included in the evaluation of concrete performance.

Table 4-4. Classifications of Phase III Aggregate Coatings

Coated Aggregate Series Field			Coated Aggregate Series Lab		
Batch	Source	Coating Type	Batch	Source	Coating Type
1	C	Carbonate	7	E	Dust
2	E	Dust/Clay	8	E	Dust
3	H	Carbonate/Clay	9	E	Clay
			10	E	Clay

4.3.3 Water Demand

Any fine material that is mixed in concrete exhibits a water demand caused by the electrostatic attraction of the double layer and the detention of water in surface voids. Water that is captured in the voids of the particles is not available for cement hydration and should not be included in calculations of the net water-cement ratio. However, water that adheres to the surface of the particle must be considered in this calculation because it is free to interact with the cement and contributes to the workability of the concrete mix.

If water does not sufficiently coat the surface area of the aggregates, workability is decreased because the particles cannot move easily within the fresh concrete. This water requirement is often associated with the amount of p200 material batched with the aggregates since the surface area of silt and clay-sized particles is significantly larger than the surface area of sand or gravel. As discussed in Section 1.2, the water absorption of clay particles is different than the water absorption of dust particles due to the layer of adsorbed water at the particle surface and the greater amount of water held in the double layer. While the double

layer water is similar to free water that adheres to the particle surface, the adsorbed layer of water is more viscous than ordinary water is assumed to be unavailable for cement hydration.

When coarse aggregate is batched for mixing, the absorption of surface coatings is typically ignored. It is assumed that the quantity of adherent material is too small to change the batch quantity of water. In addition, the standard test method for determining the water absorption of coarse aggregate (ASTM C127) requires that tests be conducted only after dust and other coatings are washed from the sample. If fines are included, they can be removed during the test and erroneously counted as water that is driven from the pores of the aggregate. In most cases, the amount of fines in the aggregate is not large enough to significantly affect the overall water absorption. However, clay particles may influence the water-cement ratio of the concrete even if the total p200 percentage is less than required values. In this study, the absorptions of the coatings were ignored until the clay coating tested in Batch 9 appeared to dramatically reduce the slump of the concrete. For Batch 10, water was added to the mix to compensate for the absorption of the fines and improve the workability of the fresh concrete.

In practice, determining the water absorption of aggregate coatings can be difficult. While ASTM C128 prescribes the cone test to determine the surface moisture of fine material, a minimum sample of 1 kg of fines would need to be washed from the aggregate and dried before the test could be conducted. Clay coatings present additional problems since the plasticity of the material would affect its shear strength, the property on which the cone test is based. To overcome these difficulties, researchers have related the water absorption of soils to other common test parameters. For example, Sridharan and Nagaraj

(1999) developed a relationship between the water-holding capacity of a soil and its liquid limit, as measured with the cone penetrometer method.

$$\text{Water Absorption (\%)} = 0.92(\text{Liquid Limit}) \qquad (4\text{-}1)$$

Similarly, Pike (1992) cited research that correlated the methylene blue value to the liquid limit and water demand of the aggregate.

A combination of the methods described above was used to estimate the water absorptions of each aggregate coating. The equation developed by Sridharan and Nagaraj (1999) was used to calculate the absorption of the highly plastic Sauk Co. fines, and the procedure prescribed in ASTM C128 was used to determine the absorption of the non-plastic Barron Co. fines. After measuring the methylene blue values of these materials, the absorptions of each coating were interpolated from the properties of Barron Co. and Sauk Co. fines, as suggested by Pike (1992). The results are tabulated in Table 4-5 and Table 4-6, which also shows the effective decrease in water-cement ratio caused by ignoring the absorptions in the mix design.

Table 4-5. Absorption Properties of Phase III Fines

Sample	Liquid Limit (%)	Water Absorption (%)	MBV (mg/g)
Barron Co.	Non-Plastic	1.7	1.7
Sauk Co.	78.0	72	14.3

Table 4-6. Estimated Absorptions of Phase III Coatings

Batch	Water Absorption (%)	Net W/C Ratio
1	0.0	0.450
2	28.1	0.443
3	31.9	0.440
4	0.0	0.450
5A	20.4	0.449
5B	NA	NA
6	11.1	0.449
7	14.5	0.443
8	12.6	0.442
9	40.5	0.431
10	55.5	0.467

The estimated water-cement ratios listed in Table 4-6 show that aggregate coatings can frustrate the control of mixing water. For typical concrete mixes, each 0.01 decrease in water-cement ratio would increase the 28-day compressive strength by 100 psi (Kosmatka and Panarese 1988). Note, however, that the tabulated estimates do not distinguish the water that is used to fill surface voids from the water that is attracted to the particle surface. As discussed previously, the latter may produce localized increases in the water-cement ratio at the aggregate-cement paste interface, while the former is captured by the aggregate and not used for cement hydration. If the electrostatic attraction of water is accounted for in the mix design, as was done in Batch 10, the net water-cement ratio is increased.

CHAPTER 5 – RESULTS OF CONCRETE TESTS

5.1 GENERAL INFORMATION

Although the WisDOT specifications require coarse aggregates to be free of adherent coatings, the results of Chapter 4 suggest that even washed aggregates are coated to a certain extent with dust, clay, or carbonate coatings. In this chapter, the effects of these coatings are determined by comparing the performance of concrete containing coated aggregate and concrete containing the aggregate after it was thoroughly washed. It is hoped that this analysis will help define which coatings are deleterious as well as the desired end result of aggregate washing.

The rest of this chapter summarizes the fresh and hardened concrete properties measured for each batch included in the mixing plan. These results are only compared among batches mixed with the same aggregate to remove the type, gradation, and surface texture of the coarse aggregate as variables in the analysis. Consequently, the tabulated test results are grouped by aggregate source (Source C, Source E, and Source H) and not by mix series (Coated Aggregate Series - Field, Washed Aggregate Series - Lab, Coated Aggregate Series - Lab). For each concrete property, the statistical significance of the coated vs. washed aggregate comparisons is evaluated at a 5% level of significance ($\alpha = 0.05$) using an analysis of variance (ANOVA) and Tukey pair-wise comparisons. The data and statistical analyses are summarized in the ANOVA tables provided in Appendix E.

5.2 FRESH CONCRETE PROPERTIES

Because of the multivariate nature of concrete, mixing parameters such as air content and water-cement ratio were carefully controlled throughout the testing program to isolate the effects of aggregate coatings. Without controlling the variation in these properties, differences in air entrainment and water content would have interfered with the effects of aggregate coatings and influenced the significance of the coated vs. washed aggregate comparisons. Accordingly, each concrete batch was proportioned with a target air content of $6.0 \pm 1.0\%$ and a fixed water-cement ratio of 0.45. Following mixing, the fresh concrete was tested for slump, unit weight, and air content to monitor the variability of these batch parameters and measure the water demand of the aggregate coatings. The fresh air contents were verified later with hardened air content measurements because the size and shape of air voids often change while the concrete is still plastic (Hover 1994).

The results in Table 5-1 show that, with the exception of slump, the fresh concrete properties did not vary significantly within each group of concrete batches. However, there appears to be large deviations between the fresh and hardened air contents measured for many of the batches. Because the unit weights listed in the table seem to have a stronger correlation with fresh air content than with hardened air content, the sample size of the hardened air content measurements was questioned. While the fresh air tests used 0.25 ft^3 of concrete, the hardened air tests were conducted on a concrete section with a surface area of 12 in^2. To assess the variability, hardened air void analyses were conducted on three different specimens that were cut from the same concrete prism. As shown in Table 5-2, the resulting air contents ranged from 5.3% to 6.3% with a standard deviation of 0.5%.

Although the average of the measured hardened air contents is nearly the same as the measured fresh air content (5.5%), the standard deviation indicates that there is a certain amount of variability in the readings. According to Hover (1994), the error of the hardened air content can be as high as 1.6% depending on specimen sampling, surface preparation, and operator subjectivity.

The comparison between the fresh and hardened air content measurements discussed above suggests that the fresh air content measurement may provide a better overall average air content than the single, discrete measurements of the hardened air content test. As a result, only the fresh air contents were used in the analysis of the test results.

Table 5-1. Fresh Concrete Test Results With Hardened Air Contents

Batch	Concrete Series	p200 (%)	Slump (in.)	Unit Weight (lb/ft^3)	Fresh Air Content (%)	Hardened Air Content (%)
Source C Aggregate						
1	Coated - Field	0.3	2 1/2	145.2	6.0	6.4
4	Washed - Lab	0.1	2 1/2	144.7	6.4	8.3
Source E Aggregate						
2	Coated - Field	0.7	1 1/2	145.9	5.5	5.8
5A	Washed - Lab	0.2	1	147.1	5.1	5.5
5B	Washed - Lab	NA	2 1/2	144.3	5.6	7.5
7	Coated - Lab	1.4	1 3/4	143.0	5.7	5.1
8	Coated - Lab	1.9	1	146.5	5.1	5.5
9	Coated - Lab	1.4	1/4	148.3	3.7	4.1
10	Coated - Lab	1.3	1 1/2	141.8	6.3	6.3
Source H Aggregate						
3	Coated - Field	0.9	1 3/4	144.7	6.0	6.6
6	Washed - Lab	0.2	3	144.9	5.9	6.8

Table 5-2. Variation in Hardened Air Contents

Specimen	Hardened Air Content (%)
2-1	6.3
2-2	5.7
2-3	5.3
Average	**5.8**

The results of the slump tests suggest that certain types of coatings can affect concrete behavior by increasing the water demand of the concrete. For the batches mixed with the Source E and Source H aggregates, concrete workability appeared to decrease as the extent and clay content of the coating increased. The observed changes in slump are described below for each aggregate.

Source C Aggregate (Dolostone with Carbonate Coating)

A constant slump was maintained between the concrete batches in the Coated Aggregate Series and the Washed Aggregate Series for Source C, implying that the carbonate coating did not have a large water absorption. This result was expected since the adherent material was well within the WisDOT p200 specification (0.26%) and the clay content, as measured by the methylene blue value (0.78 mg/g), was low.

Source E Aggregate (Granite with Dust and Clay Coatings)

With the exception of Batch 5A, which had an unusually low slump, the slumps measured for the coated aggregate batches were consistently lower than the slump of the

washed aggregate batch (Batch 5B). While the decreases in slump matched the expected effects of the dust and clay coatings, the low slump associated with the washed aggregate in Batch 5A appeared to be related to variations in the batch parameters. As shown in the batch information presented in Table C-2 (see Appendix C), the percentage and variation of moisture measured for the Batch 5A aggregate were significantly higher than the moisture properties measured for the other aggregates.

It is assumed that the unexpected slump measured for Batch 5A was caused in part by the inadequate control of aggregate moisture following the washing procedure. After the aggregate was washed, it was drained overnight and batched for mixing with a moisture content of 2.2%, or 0.8% greater than the absorption of the aggregate. Although this surface moisture was accounted for in the mix design, insufficient drying and drainage of the wet aggregate caused the standard deviation of the three moisture content readings (0.32%) to exceed the 0.28% limit specified in the precision statement of ASTM 566. With this amount of moisture variation, it is possible for the net water-cement ratio to vary by as much as 0.01, which could produce noticeable changes in concrete strength and durability. Due to this possible effect and the unexpected slump results, Batch 5A was reproduced with Batch 5B and was not included in the data analysis. In subsequent batches, the aggregates were allowed to air-dry until the average moisture content ranged from 1.4% and 1.7% with a standard deviation between 0.07% and 0.17%.

The 1-in. to 2-in. decrease in slump observed between the coated aggregate batches and the washed aggregate batch (Batch 5B) appears to be caused by the various types of clay minerals that were identified in the surface coatings (see Table 5-1). As predicted in Section

4.3.2, the clay material added in Batch 9 and Batch 10 significantly increased the water absorption of the aggregate. After a ¼-in. slump was measured in Batch 9, an additional 9% of mixing water was required in Batch 10 to maintain the same workability as the washed aggregate batch. In contrast, the slumps measured for Batch 7 and Batch 8 were comparable to the slump of the field coated aggregate batch even though considerable amounts of dust fines were added. Since a slump between 1 and 2½ in. is required for slip-formed concrete pavements, it is doubtful whether these extensive dust coatings would have been detected in the field.

The fresh concrete behavior of Batch 9 seems to support the interactions between clay and cement described by Noble (1967) and Davis et al. (1967). In separate investigations, these researchers concluded that clay fines can slow hydration by forming impermeable envelopes around the cement grains and absorbing significant portions of the mixing water. Because considerable amounts of montmorillonite and kaolinite were identified in the coating by x-diffraction, it is possible that these two occurrences were responsible for the lean appearance and lack of cohesion observed during the mixing of Batch 9. After recording the ¼-in. slump for Batch 9, it was apparent that the absorption of the coating needed to be accounted for in the calculation of the required amount of mixing water despite the standard practice of ignoring it. To determine the effect of this additional water requirement, enough water was added to Batch 10 so that the measured slump met the consistency requirements for concrete pavements. This was done by first estimating the water absorption of the fines from the liquid limit (see section 4.2.4) and then adjusting the amount of water in trial mixes until a slump between 1 and 1½ in. was obtained. Since the

2.6 lb of additional water required to meet the slump requirement (effective absorption of coating = 97%) was slightly greater than the 2.1 lb of water predicted from Equation 4-1 (theoretical absorption of coating = 72%), it is likely that the stiffening of the concrete was also related to the chemical interaction between the clay and cement theorized by Noble (1967) and Davis et al. (1967) rather than just a water absorption effect.

Source H Aggregate (Dolostone with Carbonate/Clay Coating)

Similar to the changes in slump attributed to the Source E aggregate coatings, the carbonate/clay coating on the Source H aggregate decreased the slump of the washed aggregate batch by nearly half. The presence of chlorite clay minerals in the coating was the probable cause of the decrease. Although chlorite is not as damaging as montmorillonite, it has a cation exchange capacity similar to that of illite and could significantly affect the water demand of the concrete (Gillot 1987). Incidentally, the field-coated aggregate from Source H had the highest p200 percentage (0.91%) and methylene blue value (7.13 mg/g) of all the coated aggregates sampled in the field.

5.3 CONCRETE STRENGTH

In previous studies, the effect of aggregate coatings on concrete strength was associated with the disruption of the aggregate-cement paste bond and the quantity of clay material present. Goldbeck (1932) suggested that the compressive strength of concrete decreases between 0% and 2% for each 1% of fines that adheres to the coarse aggregate. Similarly, Pike (1992) concluded that a 1% addition of kaolinite, illite, or montmorillonite fines causes a strength loss of 1%, 2%, and 4%, respectively. According to Popovics (1987),

aggregate coatings may be especially important to tensile strength since the property is related to the propagation of cracks at the aggregate-cement paste interface. In this study, the strength reduction mechanisms were investigated by comparing the relative changes in compressive and tensile strengths between the washed and coated aggregate batches.

The average compressive and tensile strengths of the four cylinders tested for each batch are tabulated in Table 5-3. The corrected compressive strengths listed in the table represent the adjustments made using Equation 3-1 to compensate for differences in air content. Qualitative comparisons between the extent of the coating and strength loss are provided below based on the p200 percentages measured for each aggregate.

Table 5-3. Average Concrete Strength Results

Batch	Concrete Series	p200 (%)	Compressive Strength (psi)		Tensile Strength (psi)
			Uncorrected	Corrected	
Source C Aggregate					
1	Coated - Field	0.3	4500	4500	410
4	Washed - Lab	0.1	4320	4450	345
Source E Aggregate					
2	Coated - Field	0.7	4970	4790	430
5B	Washed - Lab	0.2	4960	4820	410
7	Coated - Lab	1.4	4630	4630	380
8	Coated - Lab	1.9	5020	4710	395
9	Coated - Lab	1.4	5360	4600	470
10	Coated - Lab	1.3	4500	4600	365
Source H Aggregate					
3	Coated - Field	0.9	4780	4780	400
6	Washed - Lab	0.2	4560	4530	365

Source C Aggregate (Dolostone with Carbonate Coating)

Although both the compressive and tensile strength were higher for the coated aggregate batch than for the washed aggregate batch, only the average tensile strengths could be distinguished statistically. This result was surprising because (1) the coated aggregate performed better than the washed aggregate and (2) the extent of the coating was low. A possible explanation for the increase in strength is that the carbonate coating improved the quality of the aggregate-cement paste bond through the pozzalonic reactions described in Section 2.2.1. It should be noted, however, that the rule-of-thumb suggested by Goldbeck (1932) includes additions of carbonate material but only predicts strength reductions.

Source E Aggregate (Granite with Dust and Clay Coatings)

The differences in compressive strengths between the coated and washed aggregate batches generally agree with the strength losses predicted by Goldbeck (1932) and Pike (1992). As expected, the most notable changes in strength were observed for the batches in the Coated Aggregate Series - Lab. In Batch 8, a 1.7% increase in adherent dust fines produced a 2.2% decrease in strength relative to the washed aggregate batch. In Batch 9 and Batch 10, a 1% addition of dust, montmorillonite, and kaolinite fines decreased the compressive strength by 4.7%. These results suggest that the clay coatings were more detrimental to concrete strength than the dust coatings.

Although an ANOVA performed on the compressive strength results indicated that the strength differences were not significant, the test statistic calculated from the data ($F = 2.75$) is nearly equal to the critical value ($F_{0.05} = 2.77$). This implies that some of the strength differences are at or very close to statistical significance. As shown in Figure 5-1, the 95%

confidence intervals that were calculated with the mean square error from the ANOVA for Batch 9 and 10 did not overlap the 95% confidence interval for Batch 5B, the washed aggregate batch. It is likely that these differences would have been significant if the variation in the strength measurements could have been better controlled.

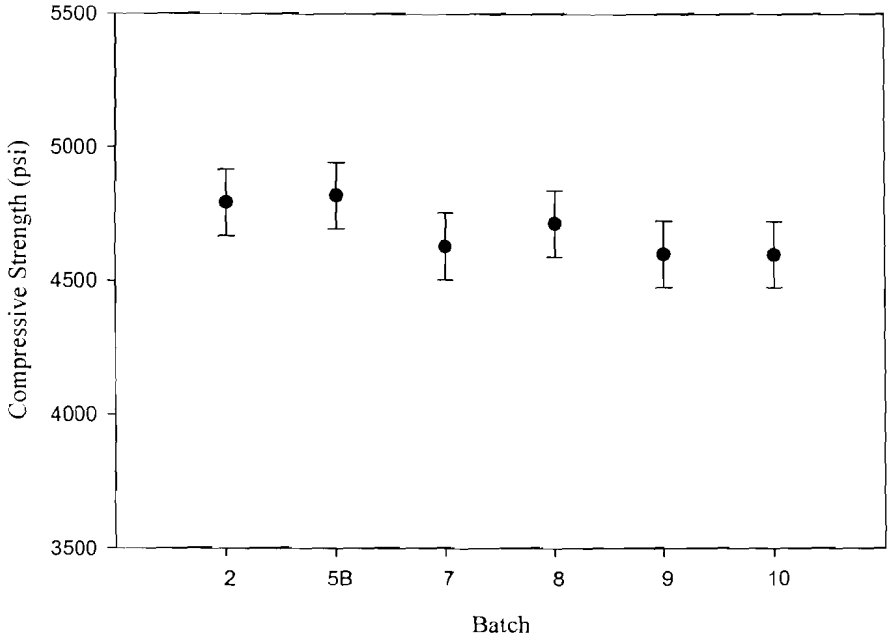

Figure 5-1. 95% Confidence Intervals of Compressive Strength

Based on the variability of the compressive strength data, it seems very difficult to associate 1% to 2% changes in strength to small increases in the p200 content of coarse aggregates. Even if the strength reductions predicted by Goldbeck (1932) and Pike (1992) are correct, the predicted changes in strength (50 - 100 psi) are nearly the same as the expected variability of the test results (118 psi). Although this suggests there is considerable uncertainty in the average compressive strengths, the coefficient of variation of each sample

is within or near the level of precision expected for 4-in. by 8-in. cylinder tests. In practice, it may be difficult to detect strength changes, such as the 2% strength decrease caused by the dust coating in Batch 8 (p200 = 1.9%), even when the p200 percentage is outside the WisDOT specification limit (1.5%). Because such strength changes are small, it is unlikely that corrective measures would be taken to account for the decreases in strength.

Unlike the trends observed for compressive strength loss, the relative changes in tensile strength do not appear to be related to the extent of the coating. For example, a 6% increase and 1% decrease in tensile strength were recorded for Batch 2 and Batch 8, respectively, despite significant amounts of p200 material on the aggregate. A larger strength reduction was anticipated for Batch 8 since the p200 percentage exceeded the 1.5% limit in the WisDOT specifications. Based on the results of Bonavetti and Irassar (1992), who related increases in the flexural strength to increases in the p200 content of mortar sand, it is possible that the addition of the dust improved the tensile strength by accelerating the hydration of the cement. As a result, dust coatings may have a greater influence on concrete strength as dispersed fines than as adherent material disrupting the aggregate-cement paste bond.

Although the results of ANOVA presented in Table E.5 show that there is a significant difference in the average tensile strengths measured for the Source E concrete batches, it is unclear exactly which of the strengths is different. As an additional step in the statistical analysis, a Tukey multiple comparison test was used to determine the differences between the average values. The results of this test indicate that Batch 9 can be distinguished

from Batch 7 and Batch 10. These differences match those predicted by the 95% confidence intervals shown in Figure 5.2.

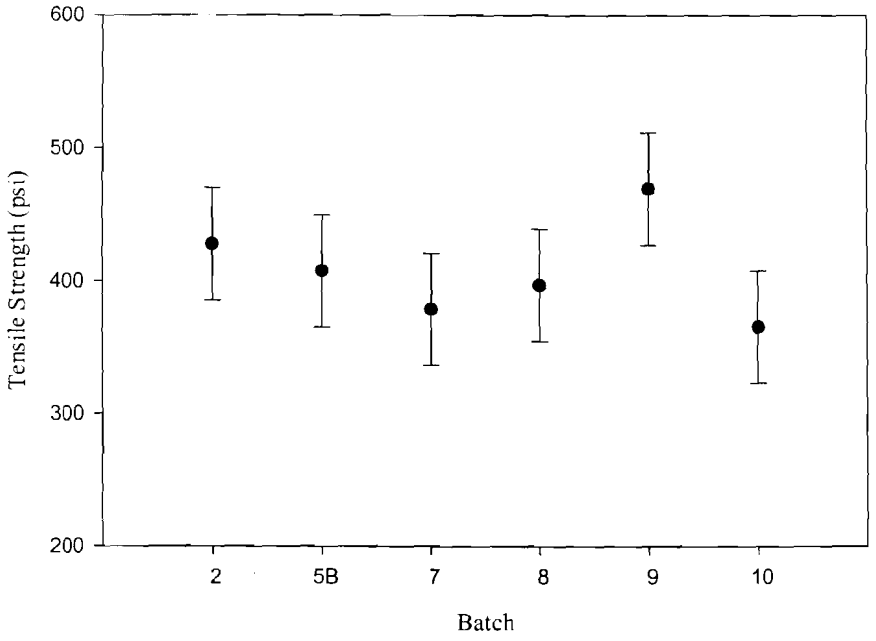

Figure 5-2. 95% Confidence Intervals of Tensile Strength

The significant strength gain of Batch 9 is likely associated with the high water absorption of the clay coating. Because this absorption decreased the workability of the concrete, any negative effect on the aggregate-cement paste bond was likely masked by the reduced air content (3.7%) and effective water-cement ratio. However, the 10% decrease in tensile strength for Batch 10 relative to the washed aggregate batches may indicate that adherence of the clay material can affect bond strength by the increased water absorption at the aggregate-cement paste interface. Although this difference could not be distinguished statistically, such a decrease would cause noticeable changes in performance.

Source H Aggregate (Dolostone with Carbonate/Clay Coating)

Slight increases in compressive and tensile strength were observed for the Coated Aggregate Series batch relative to the Washed Aggregate Series batch. However, neither of the differences was statistically significant. Since the coating consisted of a combination of carbonate and clay material, the result may represent a trade-off between the positive effect of the carbonate material observed for the Source C aggregate and the negative effect of clay material observed for the Source E aggregate. It is likely that any pozzalonic reactions between the carbonate minerals and the cement were counteracted by the stronger adherence and larger absorption of the chlorite minerals.

5.4 DRYING SHRINKAGE

Drying shrinkage is associated with the evaporation of adsorbed water and water held in the capillary pores of concrete. As this moisture is lost, the hydrated cement paste contracts and cracks if the induced shrinkage strains exceed the tensile capacity of the concrete (Sims and Brown 1998). If the cracking is extensive, shrinkage can also affect durability by decreasing the resistance of the concrete to water and chemical penetration (Mehta 1994).

The amount of drying shrinkage is principally controlled by the aggregates, which restrain the volume change and reduce the likelihood of cracking (Mehta 1994). However, aggregate properties that increase the water demand of concrete tend to decrease the ability of the aggregate to prevent excessive shrinkage (Sims and Brown 1998). It is generally believed that concrete mixed with unwashed aggregate shrinks significantly more than

concrete mixed with washed aggregate (Mehta 1994). This is particularly true for concrete containing aggregates contaminated with swelling clay minerals, such as montmorillonite, since they can also expand and contract as they gain and lose moisture. The excessive shrinkage of these minerals typically produces a distinctive cracking pattern around the aggregate as shown in Figure 5-3 (Sims and Brown 1999).

Figure 5-3. Cracking Pattern Due to Drying Shrinkage. (From Sims and Brown 1998).

For concrete tested in compliance with ASTM C157, the total drying shrinkage is expected to range between 0.040% and 0.080% (Mehta 1994). Although the results presented in Table 5-4 show that the 120-day shrinkage for each concrete batch fell within

this range, there appears to be a marked increase in shrinkage for the clay coatings on the Source E aggregates. Differences between the coated and washed aggregate batches are investigated below based on the 56-day shrinkage measurements in Table 5-4 and the water requirements discussed in Section 5-2.

Table 5-4. Average Shrinkage Results

Batch	Concrete Series	56-day Shrinkage (%)	120-day Shrinkage (%)
Source C Aggregate			
1	Coated - Field	0.038	0.048
4	Washed - Lab	0.039	0.046
Source E Aggregate			
2	Coated - Field	0.043	0.052
5B	Washed - Lab	0.034	0.046
7	Coated - Lab	0.042	0.053
8	Coated - Lab	0.046	0.058
9	Coated - Lab	0.046	0.060
10	Coated - Lab	0.056	0.065
Source H Aggregate			
3	Coated - Field	0.038	0.053
6	Washed - Lab	0.040	0.050

Source C Aggregate (Dolostone with Carbonate Coating)

As expected, similar shrinkage percentages were recorded for the concrete batches in the Coated Aggregate Series and Washed Aggregate Series for Source C. This result

supports the results of the slump test, which showed that the difference in water demands between the two batches was negligible. Since the clay content of the coating was low, the adherent material was not expected to significantly affect the ability of the aggregate to restrain shrinkage.

Source E Aggregate (Granite with Dust and Clay Coatings)

The shrinkages recorded for the Source E concrete batches consistently increased as the severity of the coating increased. An ANOVA and Tukey multiple comparison test indicated that each of the shrinkage of each coated aggregate batch can be distinguished from the shrinkage of the washed aggregate batch. From the 95% confidence intervals plotted in Figure 5-4, there appears to be a progression in the amount of shrinkage as the cleanliness of the aggregate decreases.

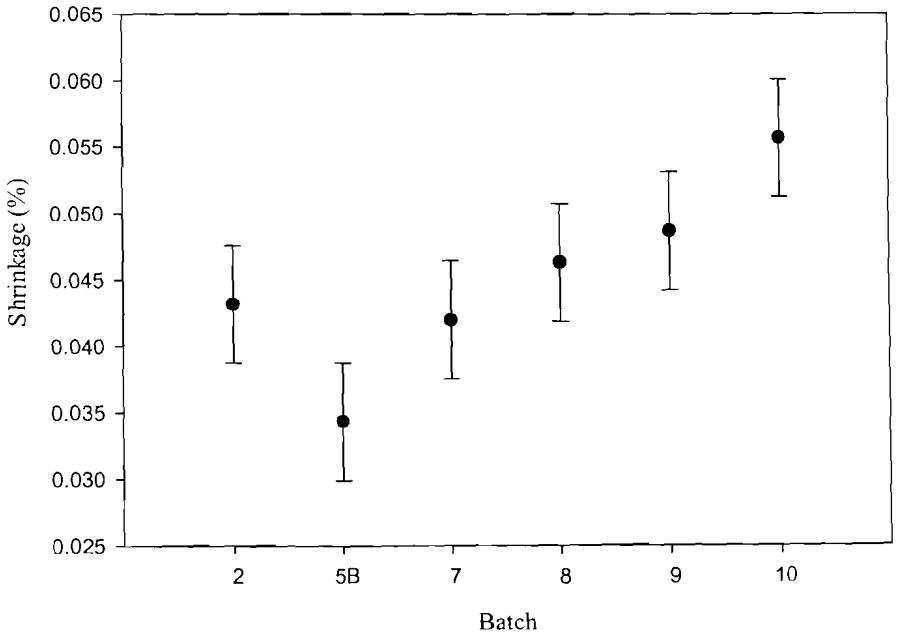

Figure 5-4. 95% Confidence Intervals of 56-Day Drying Shrinkage

According to the 56-day comparison tests, the coated aggregate batches can be divided into two groups: (1) Batch 2, 7, 8, and 9 with an average shrinkage of about 0.045% and (2) Batch 10 with an average shrinkage greater than 0.055%. It is interesting to note that the shrinkage of Batch 10 is statistically different than the shrinkage of Batch 9 even though similar amounts of clay material were added to the aggregates used for both batches. As suggested by Sims and Brown (1998), shrinkage was increased considerably (40%) when mixing water was added to meet the water demand of the concrete.

Source H Aggregate (Dolostone with Carbonate/Clay Coating)

Although the carbonate/clay coating from Source H appeared to increase the water demand of the concrete in the slump test, it did not seem to affect the amount of drying shrinkage. Only a 0.02% difference in 56-day shrinkages was recorded between the concrete batches containing the washed and coated aggregates. Note, however, that unlike Batch 10 (Source E Aggregate) additional water was not added to the coated aggregate batch to maintain the 3-in. slump measured for the washed aggregate batch. It is likely that this addition of water drives the additional shrinkage observed in many unwashed aggregates.

5.5 FREEZE-THAW DURABILITY

The cyclic freezing and thawing of concrete pavements is often associated with cracking, scaling, and spalling at joints and free edges. In previous research, this deterioration has been linked to the pressures generated by the movement of freezing water in the concrete pores (Newlon and Mitchell 1994). Hydraulic pressures develop from the flow

of water displaced by the volume expansion of ice; osmotic pressures develop from the flow of water diffused into regions of high salt or solution concentrations. The resistance of concrete to these destructive forces is related to several properties, including water-cement ratio, dispersion of entrained air, and degree of compaction. In general, concrete durability is increased as the water-cement ratio decreases and the percentage of entrained air increases.

Aggregate coatings may indirectly affect freeze-thaw durability by frustrating the control of mixing water and entrained air. Clay coatings are especially important since the likelihood of spalling and surface popouts increases if the coating absorbs significant amounts of water or weakens the aggregate-cement paste bond. On the other hand, dispersed dust fines are not likely to impair durability and may prevent surface scaling even if the dust content of the aggregate is high (Pike 1992).

The progressive degradation of weight and stiffness is shown for each batch of concrete in the figures that follow. Differences in relative durability between the coated and washed aggregate batches are tested for statistical significance at 100 and 300 cycles. Although the procedure in ASTM C666 is useful for making such comparisons, it is difficult to relate the results to field performance because exposure varies from location to location.

Source C Aggregate (Dolostone with Carbonate Coating)

Based on visual observations made during testing, it appeared that the concrete mixed with the washed aggregate (Batch 4) was slightly more durable than the concrete mixed with the coated aggregate (Batch 1). As shown in Figure 5-4, the relative stiffness of Batch 4 was consistently higher than the relative stiffness of Batch 1 over the duration of the test. The increased stiffness degradation of the coated aggregate batch may be traced to a small crack

that developed in one of the specimens during the first 125 freeze-thaw cycles. After 200 cycles, this crack expanded to expose a large aggregate in the interior of the specimen. For both concrete batches, however, little or no scaling or spalling was observed. This is indicated in Figure 5-5, which shows that the weight degradation in each batch was 0.5% or less at 300 cycles. Scaling during the first 25 cycles appeared to account for most of the weight loss for Batch 1, while an isolated popout marked the weight loss of Batch 4.

The relative weight and stiffness durability at 100 and 300 cycles are shown in Table 5-5. Although there is an apparent difference in performance between the washed and coated aggregate, the ANOVA test indicates that Batch 1 and Batch 4 cannot be distinguished statistically. Like the other concrete properties tested, this result was expected since the cleanliness of the coated aggregate and washed aggregate were similar.

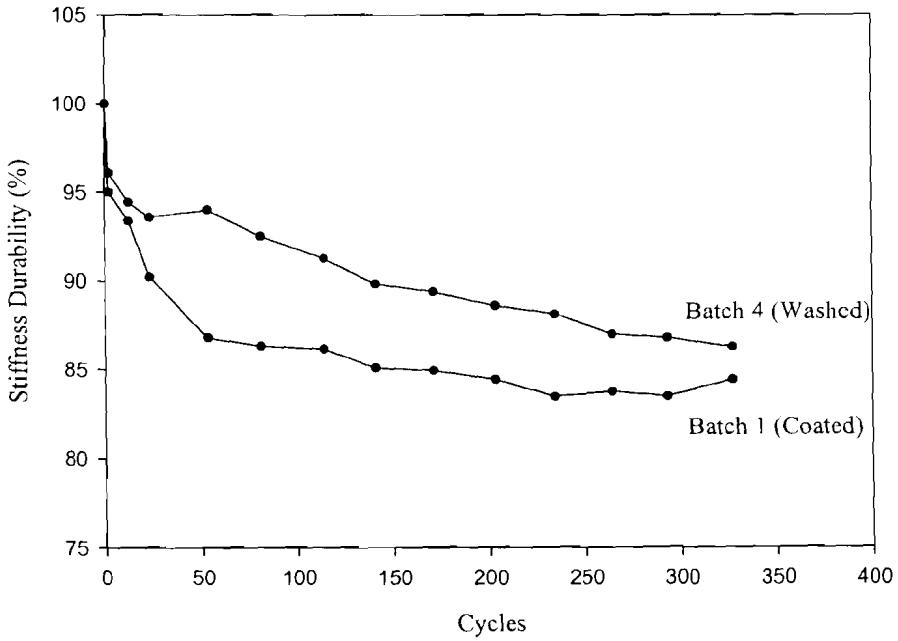

Figure 5-4. Stiffness Durability of Source C Aggregate Batches

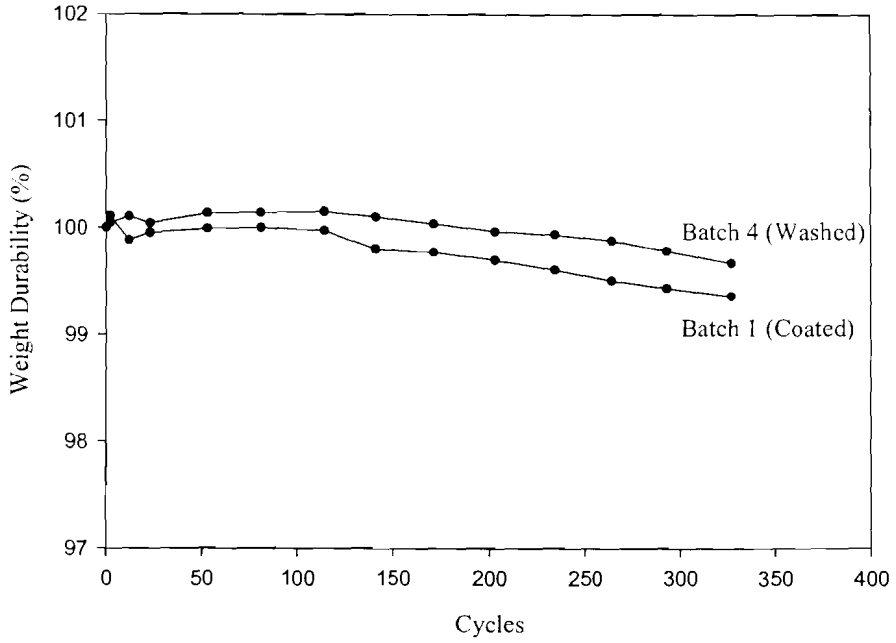

Figure 5-5. Weight Durability of Source C Aggregate Batches

Table 5-5. Durability Comparison of Source C Aggregate Batches

Batch	Concrete Series	Stiffness Durability (%)		Weight Durability (%)	
		100 Cycles	300 Cycles	100 Cycles	300 Cycles
1	Coated - Field	86.2	83.7	100.0	99.4
4	Washed - Lab	91.8	86.7	100.1	99.8

Source E Aggregate (Granite with Dust and Clay Coatings)

The results of the freeze-thaw tests suggest that the effect of the coatings depends on the mineralogy of the adherent material. As shown in Figures 5-6 and 5-7, more pronounced changes in durability were associated with the clay coatings in Batch 9 and Batch 10 than the

dust coatings in Batch 7 and Batch 8. While the initial weight and stiffness of the concrete was retained after 300 cycles in the specimens containing the dust coated aggregates, considerable scaling and stiffness degradation were observed in the specimens containing the clay coated aggregates. The largest differences in durability relative to the washed aggregate batch (Batch 5B) were measured for Batch 10, which suffered a 2.5% reduction in stiffness and a 2% reduction in weight after 300 cycles. It is likely that this increased deterioration was caused by the additional water used to achieve the 1 to1½-in. slump during mixing.

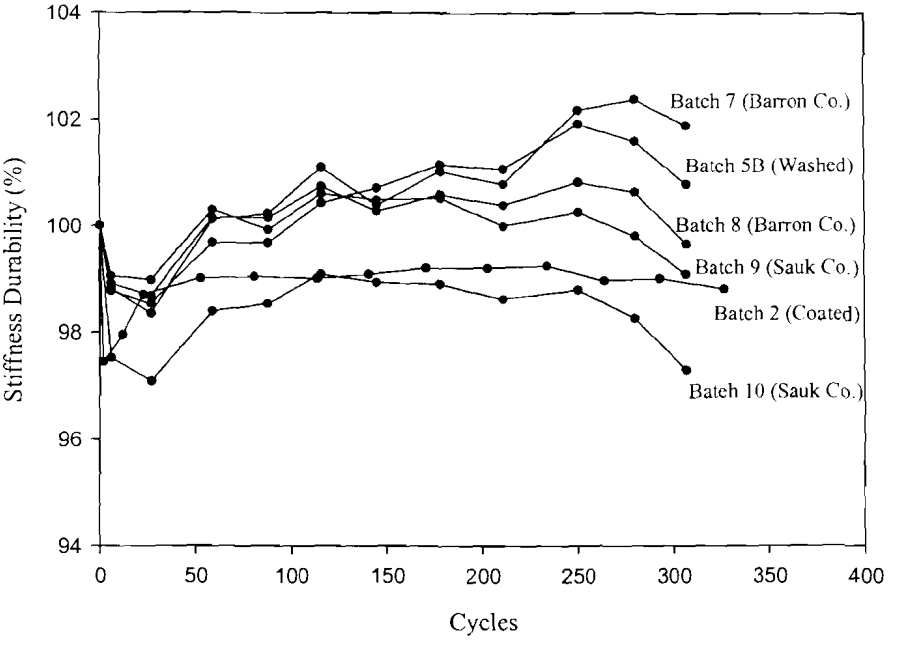

Figure 5-6. Stiffness Durability of Source E Aggregate Batches

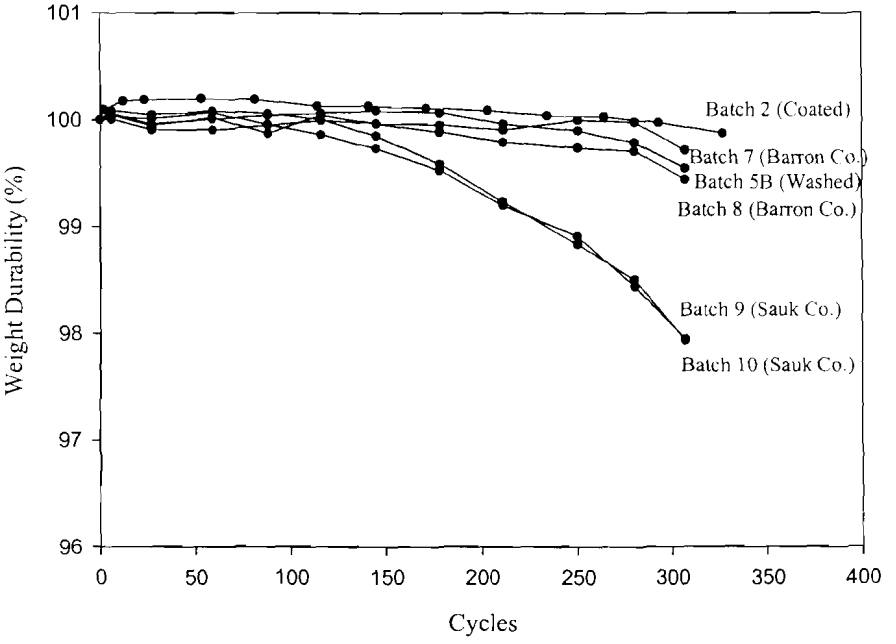

Figure 5-7. Weight Durability of Source E Aggregate Batches

The results for Batch 9 were somewhat surprising considering the high absorption of the clay fines reduced the air content to 3.7% and made proper consolidation of the specimens difficult. Although both of these factors were expected to significantly decrease the durability of the batch, the weight and stiffness degradation were similar to Batch 2 and Batch 10, respectively. The decreased effective water-cement ratio of the cement paste due to the absorbed mixing water may explain the higher than expected stiffness readings.

A summary of the freeze-thaw performance of the washed and coated aggregate batches is shown in Table 5-6. While the statistical analysis detected significant differences between Batch 2 and Batch 10 with respect to the 100-cycle dynamic modulus durability and between Batch 2 and Batches 9 and 10 with respect to the 300-cycle weight durability, none of the coated aggregate batches could be distinguished from the washed aggregate batch.

However, it is likely that the weight degradation of Batch 9 and 10 would reach significance shortly after 300 cycles since both batches were rapidly degrading as testing was completed.

Table 5-6. Durability Comparison of Source E Aggregate Batches

Batch	Concrete Series	Stiffness Durability (%)		Weight Durability (%)	
		100 Cycles	300 Cycles	100 Cycles	300 Cycles
2	Coated - Field	99.0	99.0	100.2	100.0
5B	Washed - Lab	100.0	101.0	100.1	99.6
7	Coated - Lab	100.6	102.0	100.0	99.8
8	Coated - Lab	100.4	99.9	100.0	99.5
9	Coated - Lab	100.2	99.3	100.0	98.1
10	Coated - Lab	98.8	97.5	99.9	98.1

Source H Aggregate (Dolostone with Carbonate/Clay Coating)

The data plotted in Figures 5-8 and 5-9 indicate that the concrete mixed with the Source H aggregate was less durable than the concrete mixed with either of the other aggregates. For both the coated aggregate (Batch 3) and washed aggregate (Batch 6) batches, the stiffness and weight degradation after 300 cycles was approximately 20% and 2%, respectively. Although more scaling and popouts were observed in the Batch 3 specimens, cracking appeared to accelerate the deterioration of two of the Batch 6 specimens. The jump at 200 cycles in Figure 5-9 represents the fracture of a large piece of concrete at one of these cracks. Since this fracture occurred through the aggregates, the failure was likely caused by the frost susceptibility of the aggregate rather than a weakness of the interfacial zone.

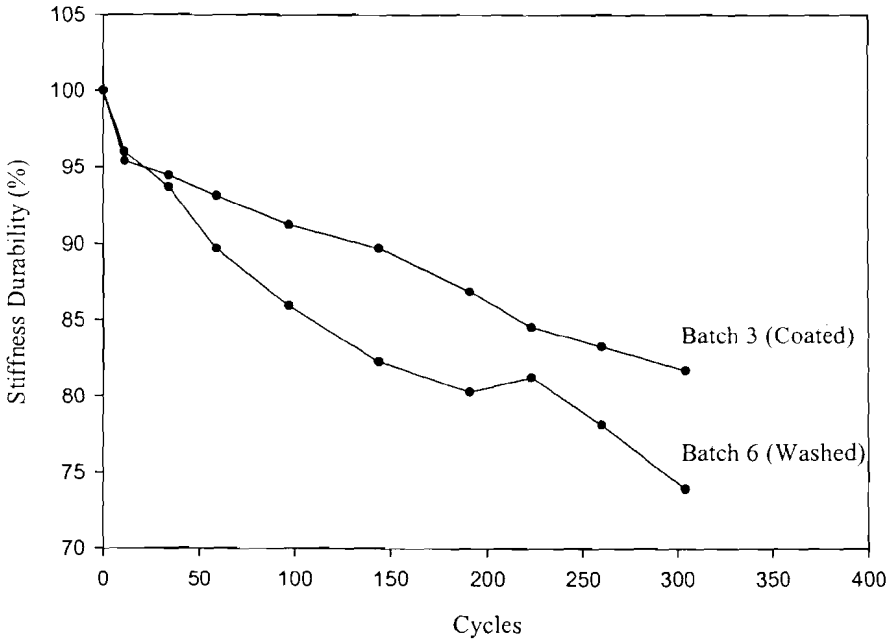

Figure 5-8. Stiffness Durability of Source H Aggregate Batches

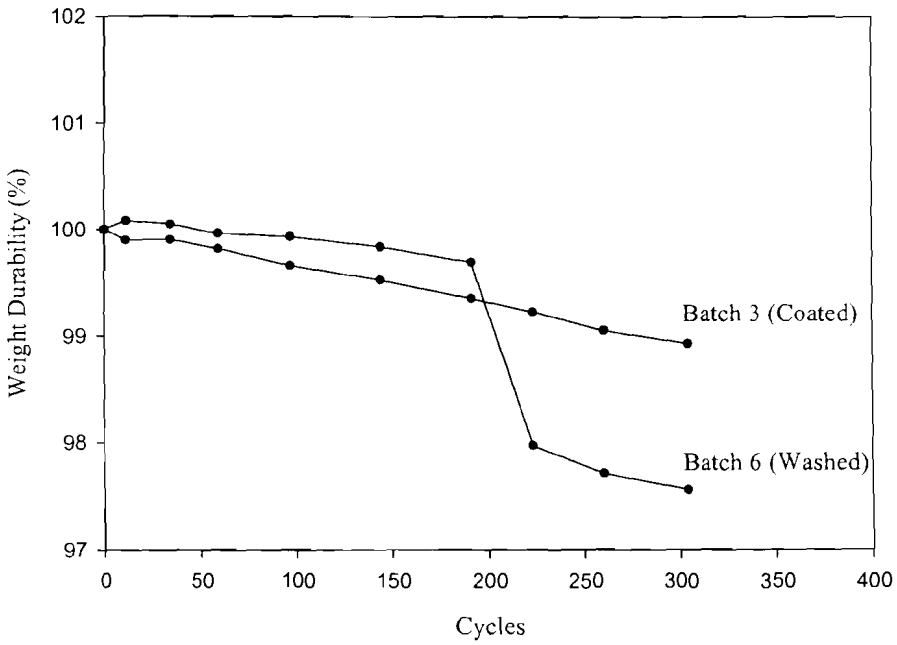

Figure 5-9. Weight Durability of Source H Aggregate Batches

Despite the failure in the Batch 6 specimen, the differences in durability summarized in Table 5-7 are not statistically significant. However, this conclusion was likely influenced by the unequal variances of the Batch 3 and Batch 6 measurements. The variation of the Batch 6 measurements was unusually high due to the cracking and subsequent failure of one of the specimens. Since the ANOVA assumes that the variance in the samples is equal, any significant differences would be difficult to detect.

Table 5-7. Durability Comparison of Source H Aggregate Batches

Batch	Concrete Series	Stiffness Durability (%)		Weight Durability (%)	
		100 Cycles	300 Cycles	100 Cycles	300 Cycles
3	Coated - Field	91.1	81.9	99.7	98.9
6	Washed - Lab	85.6	74.3	99.9	97.6

5.6 RAPID CHLORIDE ION PENETRABILITY

The rapid chloride ion penetrability test measures the resistance of concrete to chloride ion penetration, which is related to the permeability of the concrete. As discussed previously, the resistance to water and chemical penetration depends largely on the pore structure of the concrete and the microcracking caused by drying shrinkage. Generally, the diffusion of salt and water in concrete increases as the porosity and amount of cracking increases.

Aggregates affect chloride ion and water permeability primarily by influencing the water content of the concrete. Since an increase in water-cement ratio tends to increase the

porosity and shrinkage of the concrete, a high water content is often associated with chemical penetration. Consequently, aggregate coatings are expected to affect concrete permeability primarily by frustrating the control of mixing water. If water is added to compensate for the water demand of the coating, the water content of the cement paste is increased. However, if the water demand is not accounted for, the workability and consolidation of the concrete is decreased. In either case, the overall effect can be detrimental to concrete durability.

The results of the rapid chloride ion penetrability tests are shown in Table 5-8. Since the tabulated values represent the average of two different tests, it is difficult to make statistical comparisons between the coated and washed aggregate batches. When the sample size is small, the power of the ANOVA test to recognize significant differences in the data is reduced unless the sample variance is controlled. Because the precision of the rapid chloride ion penetrability test is poor[1], the differences described below are compared to the results of the other concrete durability tests to ensure significant differences are not disregarded.

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[1] The precision statement in ASTM 1202 permits a coefficient of variation of 12.3%, which indicates that a relatively large amount of variability is introduced by the test procedure.

Table 5-8. Rapid Chloride Ion Penetrability Results

Batch	Concrete Series	Charge Passed (Coulombs)	Chloride Ion Peneterability
Source C Aggregate			
1	Coated - Field	4430	High
4	Washed - Lab	3540	Moderate
Source E Aggregate			
2	Coated - Field	3330	Moderate
5B	Washed - Lab	3390	Moderate
7	Coated - Lab	3710	Moderate
8	Coated - Lab	3250	Moderate
9	Coated - Lab	3800	Moderate
10	Coated - Lab	5010	High
Source H Aggregate			
3	Coated - Field	4560	High
6	Washed - Lab	3460	Moderate

Source C Aggregate (Dolostone with Carbonate Coating)

Although the total charge passed during the testing of the coated aggregate batch was 22% higher than the charge passed in the washed aggregate batch, the difference was not statistically significant. Since the results of the freeze-thaw tests did not indicate any differences in durability, it is likely that the high chloride ion permeability of Batch 1 was caused by the variability of the test rather than the aggregate coating.

Source E Aggregate (Granite with Dust and Clay Coatings)

According to the results of the Source E aggregate batches, dust and clay coatings only affect chloride ion penetrability if the water demand of the adherent material is important. In the batches for which the absorptions of the coating were ignored, the total charge passed did not vary significantly from the washed aggregate batches. This includes Batch 9 in which the added clay material absorbed a considerable portion of the mixing water. However, water added to increase workability can increase the likelihood of chloride ion penetration. The increased water content of Batch 10 coincided with a 39% increase in the total charge passed relative to the washed aggregate batches. Based on the ANOVA and Tukey multiple comparison tests, Batch 10 can be distinguished from each of the other batches.

Source H Aggregate (Dolostone with Carbonate/Clay Coating)

The results of the Source H aggregate tests were similar to those recorded for the Source C aggregate. Despite a 27% difference between the charge passed for the Coated Aggregate Series batch and the charge passed in the Washed Aggregate Series batch, the batches could not be distinguished statistically. As with the Source E batches, this relative difference was less than the maximum variation allowed by the ASTM 1202 precision statement (35%). Since the washed aggregate batch performed better than the coated aggregate batch in the freeze-thaw test, the difference is likely associated with the variability of the test.

5.7 CONCRETE PETROGRAPHY

Although the effects of aggregate coatings were described in the previous sections using the washed vs. coated aggregate comparisons, the mechanisms through which the coatings influenced concrete performance were only implied in the discussion. The test results seem to support the theory that clay coatings behave differently than dust coatings because more pronounced changes in strength and durability were associated with the manufactured clay coatings studied in Batches 9 and 10 than with the manufactured dust coatings studied in Batches 7 and 8. Based on previous research (see Section 2.3), it was theorized that the observed decreases in tensile strength and durability were produced by the water absorption and adherence of the clay fines. According to St. John et al. (1998), the high absorption of tightly adhering clay material can increase the concentration of water at the aggregate-cement paste interface, causing the cement paste to separate from the aggregate and calcium hydroxide to crystallize in the spaces that are created. This effect often produces an aureole around the aggregate that can be observed during a petrographic examination.

In order to study the interfacial zone in detail, thin-sections were prepared for several of the Source E concrete batches (Batches 2, 5B, 8 and 10) and examined under a stereomicroscope at magnifications up to 400x. Photomicrographs of the thin-sections are provided in Appendix F. Analogous to the washed vs. coated aggregate comparisons used to analyze the hardened concrete properties, comparisons of the Batch 5B observations and the Batch 2, 8, and 10 observations were used to investigate the apparent effects of the dust/clay coatings.

The results of the petrographic examination did not provide any clear evidence of air voids, microcracks, or other signs of distress at the aggregate-cement paste interface in any of the samples. Although ASTM C857 allows the use of stereomicropes to study the aggregate-cement paste bond, any concentrations of mixing water and hydration products in the interfacial zone would best be characterized using a scanning electron microscope, which can produce images up to a magnification of 5000x and identify the chemical compositions of the constituents. Such an analysis, however, would require the services of an experienced petrographer.

CHAPTER 6 – SIGNIFICANCE OF AGGREGATE COATINGS

6.1 SCOPE OF DISCUSSION

The distinction made between the carbonate coatings in southern Wisconsin and the dust/clay coatings in northern Wisconsin seems to be related to the relative changes in concrete performance discussed in Chapter 5. Based on the coated aggregate vs. washed aggregate comparisons, carbonate coatings slightly increase concrete strength and durability whereas dust/clay coatings tend to decrease these properties. Although the effects of the dust/clay coatings appear to depend on the extent and clay content of the adherent material, it is unclear exactly what makes these coatings more deleterious than carbonate coatings.

In this chapter, the nature of aggregate coatings is examined more closely by correlating the parameters of the coating characterization tests with the properties of the hardened concrete tests. However, meaningful comparisons can only be made using the results of the Source E aggregate tests because of the additional data points provided by the concrete batches in the Coated Aggregate Series - Lab. While the performance of the washed aggregate from Source E was compared to both the performance of coated aggregates collected on-site and coated aggregates manufactured in the lab, the washed aggregates from Source C and Source H were only compared to the source coated aggregates. As a result, the significance of the carbonate coatings cannot be included in the subsequent statistical analysis.

6.2 CORRELATION OF TEST PARAMETERS

Before the results of the aggregate coating tests were correlated with the results of the hardened concrete tests, the data was organized to account for irregular aggregate and concrete properties that were intended to be controlled during batching and mixing. In some cases, adjustments were necessary to improve the accuracy of the correlations. Each of the required data modifications are justified below:

1. As discussed in Section 5.2, Batch 5A was excluded entirely from the analysis due to improper control of the aggregate moisture content at the time of batching and unusual variation in the fresh concrete properties.
2. The unrecorded characterization parameters of the Batch 5B aggregate were assumed to be identical to the characterization parameters measured for the Batch 5A aggregate. This is a valid assumption because similar changes in p200 percentage, cleanness value, and methylene blue value were observed for each of the washed aggregates relative to the coated aggregates.
3. The tensile strength of Batch 9 was not included in the correlations due to the high water absorption of the fines and the low air content of the concrete. These two factors caused the tensile strength of Batch 9 to be abnormally higher than the tensile strengths of the other batches. Unlike compressive strength, which was adjusted to a nominal air content of 6.0% in each batch, tensile strength has not been related to air content in previous research and could not be corrected for deviations in air content.

Table 6-1 shows the correlation coefficients between the aggregate coating parameters presented in Chapter 4 and the concrete properties discussed in Chapter 5. The

resulting trends are used in the following sections to discuss the effects of aggregate coatings and evaluate potential screening tests. By comparing the correlation coefficients in the table, it is possible to isolate the characteristics that make a coating deleterious and identify tests that can be used to monitor them in the field.

Table 6-1. Correlation Coefficients of Source E Test Parameters

	Concrete Strength		Concrete Durability			
Parameter	Compressive Strength	Tensile Strength	56-day Drying Shrinkage	Stiffness Durability	Weight Durability	Chloride Ion Penetration
P200 Percentage	-0.68	-0.51	0.62	-0.14	-0.30	0.17
Cleanness Value	0.81	0.72	-0.82	0.72	0.96	-0.81
MBV	-0.52	-0.42	0.71	-0.83	-0.85	0.85
MMBV	-0.82	-0.68	0.93	-0.76	-0.89	0.80

6.3 IMPLICATION OF COATING CHARACTERIZATION

Table 3-1 indicated that the coating characterization tests provide either direct or indirect measurements of the extent, mineralogy, absorption, and adherence of aggregate coatings. Due to the relationship between these properties and the characterization test parameters, the correlation coefficients in Table 6-1 may be used to assess the relative importance of dust and clay coatings to concrete strength and durability. Although the test results of carbonate coatings are not included in these correlations, expected trends can be approximated from the coated vs. washed comparisons discussed in Chapter 5.

6.3.1 Extent of Coating

The nature of aggregate coatings has traditionally been associated with the amount of material adhering to the aggregate surface. Since Goldbeck (1932) reported that each 1% increase in adherent dust can decrease compressive strength by 2%, most specifications have controlled aggregate cleanliness by restricting the percentage of p200 material. Accordingly, the WisDOT specification limits the amount of p200 material in coarse aggregates to 1.5% by mass (Wisconsin 1994). In this study, the importance of such specifications was evaluated from the three groups of coarse aggregates included in the mixing plan: (1) washed aggregates prepared in the lab with p200 percentages less than 0.2%, (2) coated aggregates sampled in the field with p200 percentages ranging from 0.2% to 1.0%, and (3) coated aggregates created in the lab with p200 percentages approaching or exceeding the WisDOT p200 limit. The relative performance of each group is incorporated in the tabulated correlations between the p200 percentage and the hardened concrete properties measured for the Source E aggregate.

The correlations indicate that the p200 percentage is an imperfect predictor of the deleterious nature of aggregate coatings. As shown in Table 6-1, there is only a weak relationship between the p200 percentage and concrete strength, and the relationship between the p200 percentage and concrete durability is even weaker. The weakness of these correlations is best explained by the behavior of the dust coatings and clay coatings created with the Barron Co. and Sauk Co. fines. The manufactured dust coatings were largely innocuous as the adherent dust caused only small changes in concrete strength and durability even when the extent of the coating exceeded 1.5% of the aggregate weight. On the other

hand, the coatings containing a similar percentage of clay material were more deleterious as the addition of the clay material resulted in significant changes in concrete performance. Based on these differences in behavior, it is evident that the effect of aggregate coatings is not solely dependent on the p200 percentage of the aggregate.

The inadequacy of the p200 test to monitor aggregate coatings is even more apparent when the properties of the Source C and Source H concrete batches are considered. For each of these aggregates, increases in the amount of p200 material were associated with increases in both compressive and tensile strength. Although the mechanism of this strength increase was not investigated, previous research has shown that additions of carbonate material can improve the quality of the aggregate-cement paste bond (Grandet and Olivier 1980). Consequently, the nature of aggregate coatings appears to be closely associated with the mineralogy of the adherent material as well as the extent of the coating.

6.3.2 Mineralogy of Coating

Research studying the effects of dispersed fines in mortar sand suggests that additions of dust, clay, and carbonate fines have different effects on the properties of mortar. Typically, increases in dust fines or carbonate fines have little or no effect on strength, but increases in clay fines significantly decrease strength (Pike 1992). Based on these results, it follows that the effects of coarse aggregate coatings should also depend on the mineralogy of the adherent material. This is implied in the coarse aggregate specifications in ASTM C33, which allows the p200 limit to be increased if clay and shale are not present.

In this study, the mineralogy of the p200 material was measured with the methylene blue value. As discussed previously, this parameter is closely related to the absorptive

properties of clay material and is often used to detect the presence of clay minerals. Although the effects of each coating type (dust, clay, and carbonate) were investigated, only the nature of dust and clay coatings can be distinguished from the correlation coefficients because carbonate material was not found in the Source E coatings. The significance of this material must be inferred from the Source C and Source H concrete tests.

Compared to the p200 percentage, the methylene blue value has a much stronger correlation with the properties related to concrete durability. However, like the p200 percentage, the parameter is only weakly related to concrete strength. The distinction between these correlations results from the different properties of the adherent materials. Clay coatings are able to absorb much more water than either dust or carbonate coatings due to their unique internal structure. As a result, water that is expelled during cycles of freezing and thawing durability can increase the critical saturation of the cement paste and impair durability (Newlon and Mitchell 1994). Depending on the type of clay minerals in the coating, the displaced water may affect durability even if the extent of the coating is small. On the other hand, compressive strength likely depends on both the mineralogy of the coating and the p200 percentage because the effects of water absorption and bond strength are only important if the total amount of absorbed water is large enough to decrease the effective water-cement ratio or disrupt aggregate-cement paste bond.

Although mineralogy is an important indicator of deleterious coatings, the extent of the coating must also be considered. As shown in Table 6-1, the effects of aggregate coatings are best predicted by the California cleanness value and the modified methylene blue value. In each of these tests, the p200 measurement accounts for the differences in

particle size and plasticity among dust, clay, and carbonate fines. The California cleanness test is a volumetric measurement of clay-sized particles based on the slow settling times and large absorptions of clay fines in water, while the modified methylene blue value is simply the methylene blue value multiplied by the p200 percentage. Although the correlations for these tests were stronger than the other characterization tests, they are not perfect predictors of the effects of aggregate coatings. Because the correlations ranged between 0.68 and 0.96, it is appears that several parameters, like strength of adherence, may also influence performance.

The carbonate coatings on the Source C and Source H aggregates appeared to improve concrete strength, which supports the claim made in earlier reports that some coatings can improve concrete performance (Schmitt 1990). Based on the evaluation of the carbonate/clay coating on the Source H aggregate, the innocuous behavior of the carbonate fines may even counteract the deleterious behavior of clay fines. Despite a significantly lower cleanness value and higher modified methylene value, the aggregate and coating sampled from Source H outperformed the washed aggregate. The result appears to discount the use of the California cleanness test and methylene blue adsorption test to monitor coatings with large amounts of carbonate material. However, additional testing of more extensive carbonate and clay coatings is required before this can be concluded.

6.3.3 Water Absorption by the Coating

The water absorption of coarse aggregate coatings is usually ignored when concrete mix designs are determined. However, if a coarse aggregate with a p200 percentage of 1.5% is used in a standard WisDOT Grade A concrete mix, a 60% absorption can reduce the net

water-cement ratio by 0.03 and presumably increase concrete strength by 400 psi, if no water additions occur. In Section 4.3, the water absorption of each coating was approximated from the methylene blue values and the water absorptions measured for the Barron Co. and Sauk Co. fines. To further investigate the effect of this property on concrete performance, the correlations for the modified methylene blue value were studied because the total amount of water absorbed by the coating and the resulting change in water-cement ratio depend on both the methylene blue value and the p200 percentage of the aggregate

If the estimated water absorptions were accurate, it appears that the absorption of the coating was detrimental to concrete performance. As shown in Table 6-1, increases in the modified methylene blue value, and hence the absorption of the coating, were associated with decreases in both concrete strength and durability. This result indicates that the positive effect of the net water-cement decrease is counteracted by another mechanism that has a negative effect on hardened concrete properties. One possible theory is that the water adsorbed by the coating increases the thickness of the interfacial water-layer described in Section 2.2.1. While this produces an overall decrease in the effective water-cement ratio of the concrete, the film of water increases the water-cement ratio at the interface of aggregate-cement paste and reduces the strength of the aggregate-cement paste bond.

The same correlations between water absorption and concrete performance breakdown when carbonate/clay coatings are considered. Although the aggregate coating sampled from Source H was characterized with a high modified methylene blue value, the strength and durability of the coated aggregate batch were higher than that of the washed aggregate batch. Again, this behavior appears to verify the effects of aggregate coatings

discussed in Section 2.2.1. Research has shown that interactions between carbonate material and the cement paste can improve bond strength by reducing the thickness of the water layer and increasing mechanical interlocking in the interfacial zone. Both of these effects would explain the slight increases in strength and durability that have been attributed to the carbonate coatings.

6.3.4 Adherence of Coating

Tensile strength is an indicator of the strength of the aggregate-cement paste bond and the tendency for cracking since the property is related to the propagation of cracks at the aggregate-cement paste interface (Popovics 1987). Based on this association, the correlations in Table 6-1 indicate that the adherence of the coating depends on clay content. As shown in the table, the correlation between tensile strength and the cleanness value and modified methylene blue value were stronger than the correlation between tensile strength and the p200 percentage. Note, however, that the petrographic examination of thin-section samples did not provide conclusive evidence that extensive clay coatings disrupt the interfacial zone. As a result, future research work should examine possible test methods to more accurately measure the strength of the aggregate cement-paste bond so that more accurate correlations can be made.

6.4 IMPLEMENTATION OF TEST RESULTS

Because little research has been devoted to the effects of aggregate coatings on hardened concrete properties, few specifications explicitly state which types of coatings are deleterious and which are innocuous. For example, the WisDOT specification requires

coarse aggregates to be clean and free of injurious coatings but does not define the terms *clean* or *injurious*. As a result, the decision to use suspect aggregates is usually based on the opinion of the engineer and not on a standard set of guidelines. Typically, aggregate coatings are controlled by limiting the amount of p200 material adhering to the aggregate. However, the p200 test is only related to the extent of the coating and not to the mineralogy of the adherent material. As discussed in the previous sections, both of these properties need to be considered when distinguishing between innocuous and deleterious coatings.

The results of the aggregate coating and concrete tests indicate that the behavior of the dust/clay coatings prevalent in northern Wisconsin is quite different from the behavior of the carbonate/dust coatings in southern Wisconsin. While the carbonate material appeared to slightly improve concrete strength and durability, the clay material impaired performance by increasing the water requirement and disrupting the aggregate-cement paste bond. Although tests were also conducted to estimate the impact of these two effects, it is unclear whether the absorption or the adherence of the clay coatings is more deleterious to concrete performance. Nevertheless, specifications should explicitly distinguish between these types of coatings either by allowing a higher p200 percentage for carbonate aggregates or specifying another test that better characterizes the adherent dust and clay material on silicate aggregates. In light of the minor changes performance caused by the dust coatings created with the Barron Co. fines, the 1.5% p200 limit specified by the WisDOT may also be relaxed if coatings on silicate aggregates consist predominantly of feldspar minerals.

The correlation coefficients in Table 6-1 suggest that either the California cleanness value or the modified methylene blue value are better suited to characterize the nature of

dust/clay coatings than the p200 percentage. As described in Section 4.4.2 and 4.4.5, these parameters are better predictors of the potential concrete strength and durability changes caused by the coating because they account for both the quantity and mineralogy of the adherent fines. Although each test is a suitable replacement for or supplement to the p200 test, a comparison indicates that the California cleanness test would be the best screening test. Some of the advantages of the California cleanness test over the methylene blue adsorption test are listed below.

1. A standard test procedure and specification limit for the California cleanness test have already been adopted by Caltrans to monitor coarse aggregate coatings. On the other hand, the methylene blue adsorption test is still gaining acceptance within the concrete industry and has only been used to screen fine aggregates used in concrete and asphalt.
2. The California cleanness test could replace the p200 test because it is conducted directly on coarse aggregate samples. However, the methylene blue adsorption test is conducted on samples of the p200 material removed from the aggregate and is only meaningful if it is used as a supplement to the p200 test.
3. The California cleanness test is easy to conduct, while the methylene blue adsorption test is more sophisticated, highly subjective, and time consuming.
4. The stability of the calcium chloride solution used in the California cleanness test does not significantly affect the results of the test; the instability and variable concentration of the methylene blue solution used in the methylene blue adsorption test significantly reduce the precision of the test.

The results of the study also suggest that coatings on washed aggregates are not usually important to concrete performance. No effect that would be detected during the mixing or placing of concrete could be attributed to the dust/clay coating and carbonate coating that were evaluated on field washed aggregates. On the other hand, the clay coatings created with the Sauk Co. fines produced noticeable changes in workability of the fresh concrete and significant decreases in durability even though the total amount of p200 material was less than the specified WisDOT limit. The deleterious effect of these coatings indicates that the washing of coarse aggregates should focus on the removal of clay material. Sims and Brown (1992) suggest that aggregates with a high clay content should be washed with log washers or scrubbers before they are mixed in concrete. Adherent dust and carbonate material do not appear to be as important as adherent clay since the manufactured dust coatings were largely innocuous and the unwashed Source H aggregate appeared to improve performance. Although it is possible that aggregates with these types of coatings do not need to be washed even if the 1.5% p200 limit is exceeded, additional research is required to examine the performance of pit-run aggregates in concrete before this can be recommended.

CHAPTER 7 – SUMMARY AND CONCLUSIONS

7.1 SUMMARY

The primary objectives of this study were to identify the types of aggregate coatings prevalent in Wisconsin and to determine their effects on concrete strength and durability. To this end, coarse aggregates containing surface coatings were collected throughout the state and characterized with the California cleanness test, methylene blue adsorption test, and x-ray diffraction. Based on the results of these tests, a subset of the sample aggregates was selected to further study the effects of the coatings on concrete performance. The 10-batch mixing plan tested coarse aggregates from three different sources and in the following three conditions: (1) coated aggregates that were sampled in the field, (2) washed aggregates that were washed in the laboratory to remove the existing coating, and (3) coated aggregates that were manufactured with dust and clay fines to increase the extent of the coating. The effect of each aggregate coating was assessed by comparing the relative changes in compressive strength, tensile strength, drying shrinkage, freeze-thaw durability, and chloride ion penetrability between concrete batches containing the washed and coated aggregates. A summary of the results of the coating characterization and hardened concrete tests is provided in the sections that follow.

7.1.1 Summary of Aggregate Coating Tests

Based on the characterization of the 10 coated aggregates sampled in Wisconsin, the following observations were made regarding the nature of aggregate coatings:

1. Visible surface coatings of silt and clay-sized particles adhere to most coarse aggregates even after they are washed in the field.
2. Coatings in southern Wisconsin consist predominantly of dolomite, quartz, and anorthite minerals, and the coatings in northern Wisconsin consist predominantly of quartz and feldspar minerals. These materials can be classified as stone dust because the mineralogy is consistent with the major rock-forming minerals of Wisconsin aggregates.
3. Coatings in northern Wisconsin also contain minor amounts of chlorite and illite minerals. These clay minerals are likely associated with the deep weathering of aggregate deposits in that part of the state.
4. The cleanness values and methylene blue values measured for the sampled aggregates and coatings were within the existing specifications limits, which suggested that the potential effects of the field coated aggregates would not be significant.
5. Samples of fine material were collected from Barron Co. and Sauk Co. and attached to the Source E aggregate to create severe dust and clay coatings that approached or exceeded the 1.5% WisDOT p200 limit. The Barron Co. fines consisted of quartz and a variety of feldspar minerals. The Sauk Co. fines consisted of quartz and major amounts clay minerals, including a swelling clay.

6. Certain types of clay coatings were shown to have water absorptions as high as 60%. This absorption could decrease the water-cement ratio by 0.03 and significantly reduce workability if the amount of p200 material is 1.5% and the absorbed water is ignored in the mix design.

7.1.2 Summary of Concrete Tests

Based on the washed aggregate vs. coated aggregate comparisons made for each aggregate source, the coatings evaluated in the mixing plan had the following effects:

1. Extensive clay coatings dramatically reduced slump when the water absorption of the fines was ignored. Only a ¼-in. slump was measured for Batch 9, which contained a clay coated aggregate with a p200 percentage of 1.4%, compared to a 2½-in. slump measured for the washed aggregate batch. In contrast, the dust coatings and carbonate coatings did not significantly affect the workability of the concrete even when the p200 percentage approached the WisDOT specification limit.
2. The carbonate coatings slightly increased compressive and tensile strength, while the clay coatings slightly decreased strength. However, these effects could not be distinguished statistically since the changes are approximately equal to the inherent variation of the strength tests (~5%).
3. The dust/clay coatings progressively increased drying shrinkage as the extent and clay content of the coating increased. The shrinkage of each concrete batch containing these coatings could be distinguished from the shrinkage of the washed aggregate batch. The most significant change in shrinkage (40%) was observed for Batch 10, for which water

was added to correct for the absorption of the clay fines. In contrast, the carbonate coatings appeared to have no effect on shrinkage.

4. Only the clay coatings manufactured with Sauk Co. fines impaired the freeze-thaw durability of concrete. For the specimens prepared with these coatings, the degradation of weight (scaling) was 2% and the degradation of stiffness (cracking) was 2.5% after 300 cycles. However, no significant changes in durability were noted for the carbonate or dust coatings. In fact, the dust coatings manufactured with the Barron Co. fines appeared to have no effect even when the p200 percentage of the aggregate was 1.9%
5. Although each of the coating types appeared to increase the penetration of chloride ions by as much as 30%, the increase could not be separated from the large amount of variation introduced by the rapid chloride ion penetrability test.
6. Petrographic examinations of various thin-section samples did not reveal any signs of air voids or microcracks at the aggregate-cement paste interface.

7.2 CONCLUSIONS

The aggregate coatings found in Wisconsin can be grouped into three basic types: dust coatings, clay coatings, and carbonate coatings. Although there is a perception that these coatings can decrease concrete strength and durability by disrupting the aggregate-cement paste bond, this research shows that the coatings that adhere to washed aggregates in the field have only minor effects on concrete performance. However, the test results of the aggregate coatings manufactured in the lab (Coated Aggregate Series - Lab) also indicate that aggregates with clay coatings and a p200 percentage near 1.5% can potentially produce

noticeable changes in slump and durability. This is especially true with drying shrinkage, which increased by approximately 40% when extensive clay coatings were investigated. Despite the fact that these effects may be mitigated by vigorously rewashing the aggregate until the adherent clay is removed, the resulting increases in concrete strength (5%) and durability (3%) do not appear to justify the incremental cost of additional aggregate washing.

Although existing specifications imply that the extent of aggregate coatings determines their effects on concrete performance, the test results show that mineralogy is an even more important indicator of deleterious and innocuous coatings. Currently, coatings are controlled in the field by limiting the amount of p200 material on the aggregate. However, the calculated correlations between the test parameters measured for the dust/clay coatings and the corresponding hardened concrete properties indicate that the California cleanness test is a better predictor of concrete strength and durability. During the study, aggregates containing significant clay coatings passed the WisDOT p200 requirement but failed the Caltrans cleanness value specification. Because the cleanness test measures the extent of the coating while distinguishing clay coatings from dust or carbonate coatings, it is more closely related to the mechanisms that impair concrete performance. As a result, the WisDOT should require the California cleanness test whenever aggregate coatings are suspected of influencing strength or durability during concrete construction.

7.3 FUTURE RESEARCH NEEDS

As this research progressed, it became evident that many of the issues regarding aggregate cleanliness remain unresolved. In order to completely understand the significance

of aggregate coatings, these areas will need to be investigated further in future studies. A list of specific research needs is provided below:

1. A more extensive mixing plan is needed to evaluate the effects of carbonate/clay coatings over a wider range of p200 percentages and clay contents. These additional batches would test the apparent innocuous behavior of carbonate coatings and the interactions between carbonate fines and clay fines.
2. Test that directly measure the bond strength at the aggregate-cement paste interface and at the coating-aggregate interface are needed to verify the mechanisms through which aggregate coatings affect concrete performance. These measurements would confirm that the effect of coatings on the aggregate-cement paste bond has been overestimated in previous research.
3. A more thorough petrographic examination of the interfacial transitional zone is needed to better identify the chemical and physical changes caused by aggregate coatings. The use of scanning electron microscopy could be used to investigate the hydration products and water films that collect in this region.
4. Additional concrete batches mixed with other admixtures are needed to identify feasible methods that can be used to offset deleterious effects of aggregate coatings. These batches would investigate how cementitious materials and water reducers can compensate for the water requirements and hydration changes caused by the coatings.
5. Additional batches mixed with pit-run material or unwashed aggregates are needed to further evaluate the importance of aggregate cleanliness. These batches would better define the required end result of aggregate washing methods.

BIBLIOGRAPHY

Ahn, N., Fowler, D.W., and Hudson, B. (2000). *The Effects of High Fines on the Properties of Cement Mortar*. Presentation at the Transportation Research Board Meeting, January 2000. (unpublished).

Aschenbrenger, T. (1992). *Comparison of Colorado Component Hot Mix Asphalt Materials with Some European Specifications*. Report No. CDOT-DTD-R-92-14. Colorado Department of Transportation.

Benson, P.E. and Ames, W.H. (1975). The Precision of Selected Aggregate Test Methods. *Transportation Research Record*, No. 539. 85-93.

Bernsted, J. (1985). Application of the Methylene Blue Test to Cement Raw Materials. *Journal of Chemical Technology and Biotechnology*, 35A, 181-184.

Bonavetti, V.L. and Irassar, E.F. (1994). The Effect of Stone Dust Content in Sand. *Cement and Concrete Research*, 24(3), 580-590.

Buckely, E.R. (1903). *Highway Construction in Wisconsin*. Wisconsin Geological and Natural History Survey Bulletin No. 10. Madison: State of Wisconsin.

Buth, E., Ivey, D.L., and Hirsch, T.J. (1966). Correlation of Concrete Properties with Tests for Clay Content of Concrete. *Highway Research Record*, No. 124, 1-17.

Buth, E., Ivey, D.L., and Hirsch, T.J. (1968). Dirty Aggregate, What Difference Does it Make? *Highway Research Record*, No. 226, 1-17.

California Department of Transportation (1999). *Standard Specifications*. July 1999 Ed.

Darwin, D. and Slate, F.O. (1970). Effect of Paste-Aggregate Bond Strength on Behavior of Concrete, *Journal of Materials*, 5(1), 86-98.

Das, B.M. (1998). *Principles of Geotechnical Engineering*. Boston: PWS Publishing.

Darwin, D. (1999). Concrete in Compression. *Concrete International: Design and Construction*, 21(8). 82-85

Davis, R.E., Mielenz, R.C., and Polivka, M. (1967). Importance of Petrographic Analysis and Special Tests Not Usually Required in Judging the Quality of Concrete Sand. *Journal of Materials*, 2(3), 461-486

Dolar-Mantuani, L. (1966). Petrographic Examination of Natural Concrete Aggregates. *Highway Research Record*, No. 120, 1-17.

Dolar-Mantuani, L. (1983). *Handbook of Concrete Aggregates: A Petrographic and Technological Evaluation*. Park Ridge, NJ: Noyes Publications.

Forster, S.W. (1994). Soundness, Deleterious Substances, and Coatings. In: P. Kiegler and J. Lamond (Ed.), *Significance of Tests and Properties of Concrete and Concrete-Making Materials*, ASTM 169C, (pp. 415-426). Philadelphia: American Society for Testing and Materials.

Gillot, J.E. (1987). *Clay in Engineering Geology*. Amsterdam: Elsevier Science Publishers.

Graves, R.E., Eades, J.L., and Smith, L.L. (1992). Calcium Hydroxide Treatment of Construction Aggregates for Improved Cementation Properties. In D.D. Walker, T.B. Hardy, D.C. Hoffman, and D.D. Stanley (Ed.), *Innovations and Uses for Lime*, STP 1138, (pp. 65-77). Philadelphia: American Society for Testing and Materials.

Grim, R.E. (1953). *Clay Mineralogy*. New York: McGraw-Hill.

Goldbeck, A.T. (1932). The Nature and Effects of Surface Coatings on Coarse Aggregates, *Proceedings, Highway Research Board*, 12(1), 305-319.

Hills, J.F. and Pettifer, G.S. (1985). The Clay Mineral Content of Various Rock Types Compared with the Methylene Blue Value. *Journal of Chemical Technology and Biotechnology*, 35A, 168-180.

Hosking, J.R. and Pike, D.C. (1985). The Methylene Blue Dye Adsorption Test in Relation to Aggregate Drying Shrinkage. *Journal of Chemical Technology and Biotechnology*, 35A, 185-194.

Hotchkiss, W.O. and Steidtmann, E. (1914). *Limestone Road Materials of Wisconsin*. Wisconsin Geological and Natural History Survey Bulletin No. 34. Madison: State of Wisconsin.

Hover, K.C. (1994). Air Content and Unit Weight of Hardened Concrete. In: P. Kiegler and J. Lamond (Ed.), *Significance of Tests and Properties of Concrete and Concrete-Making Materials*, ASTM 169C, (pp. 296-314). Philadelphia: American Society for Testing and Materials.

Hughes, B.P. and Ash, J.E. (1968). The Effect of Mix Proportions and Aggregate Dust upon the Compressive Strength of Concrete. *Magazine of Concrete Research*, 20(63), 77-84.

Kandhal, P.S. and Parker, F. (1998). *Aggregate Tests Related to Asphalt Concrete Performance in Pavements, Report 405*. Washington D.C.: National Cooperative Research Program.

Kelley, P.L., Bell, R.B., Brainerd, M.L., and Scali, M.J. (2001). Under the Microscope. *Civil Engineering*, 71(6), 66-71

Kosmatka, S.H. and Panarese, W.C. (1988). *Design and Control of Concrete Mixtures*. 13th Ed. Skokie, IL: Portland Cement Association.

Lang, F.C. (1943). Deleterious Substances, *Report on the Significance of Tests of Concrete and Concrete Aggregates*, ASTM SP 22A, 96-101.

Mehta, P. K. (1994). Volume Change. In P. Kiegler and J. Lamond (Ed.), *Significance of Tests and Properties of Concrete and Concrete-Making Materials*, ASTM 169C, (pp. 219-228). Philadelphia: American Society for Testing and Materials.

Mielenz, R.C. (1994). Petrographic Examination of Concrete Aggregates. In: P. Kiegler and J. Lamond (Ed.), *Significance of Tests and Properties of Concrete and Concrete-Making Materials*, ASTM 169C, (pp. 341-366). Philadelphia: American Society for Testing and Materials.

Newlon H. and Mitchell, T.M. (1994). Freezing and Thawing. In: P. Kiegler and J. Lamond (Ed.), *Significance of Tests and Properties of Concrete and Concrete-Making Materials*, ASTM 169C, (pp. 153-163). Philadelphia: American Society for Testing and Materials.

Neville, A.M. (1996). *Properties of Concrete*. New York: John Wiley.

Noble, D.F. (1967). Reactions and Strength Development in Portland Cement/Clay Mixtures. *Highway Research Record*, No. 198, 39-56.

Ozol, M.A. (1994). Alkali-Carbonate Rock Reaction. In: P. Kiegler and J. Lamond (Ed.), *Significance of Tests and Properties of Concrete and Concrete-Making Materials*, ASTM 169C, (pp. 372-387). Philadelphia: American Society for Testing and Materials.

Ozol, M.A. (1979). Shape, Surface Texture, Surface Area, and Coatings. *Significance of Tests and Properties of Concrete and Concrete-Making Materials*, ASTM 169B, (pp. 584-625). Philadelphia: American Society for Testing and Materials.

Patten, J.F. (1973). The Effects of Adhesive Bond Between Coarse Aggregate and Mortar on the Physical Properties of Concrete. *Civil Engineering Transactions*, 15(1,2), 58-62.

Perkins, D. (1998). *Mineralogy*. Upper Saddle River, NJ: Prentice Hall.

Popovics, S. (1998). *Strength and Related Properties of Concrete a Quantitative Approach*. New York: John Wiley.

Popovics, S. (1987). Attempts to Improve the Bond Between Cement Paste and Aggregate. *Materials & Structures*, 20(115), 32-38.

Pike, D.C. (1992). *Methodologies for Assessing the Variability of Fines in Sands Used for Concretes and Mortars*. PhD Thesis. University of Reading.

Schmitt, J.W. (1990). Effects of Mica, Aggregate Coatings, and Water-Soluble Impurities on Concrete. *Concrete International: Design and Construction*, 12(12), 54-58.

Sims, I. and Brown, B.V. (1998). Concrete Aggregates. In: P.C. Hewlett (Ed.), *Lea's The Chemistry of Cement and Concrete*, 4th Ed. London: Arnold Publishing.

Sridharan, A. and Nagaraj, H.B. (1999). Absorption Water Content and Liquid Limit of Soils. *Geotechnical Testing Journal*, 22(2), 121-127.

St. John, D.A., Poole, A.B., and Sims, I. (1998). *Concrete Petrography: A Handbook of Investigative Techniques*. London: Arnold Publishing.

Struble, L., Skalny, J., and Mindess, S. (1980). A Review of the Cement-Aggregate Bond. *Cement and Concrete Research*. 10(2), 277-286.

Tasong, W.A., Lynsdale, C.J., and Cripps, J.C. (1998). Aggregate-Cement Paste Interface. II: Influence of Aggregate Physical Properties. *Cement and Concrete Research*, 28(10), 1453-1465.

Tasong, W.A., Lynsdale, C.J., and Cripps, J.C. (1999). Aggregate-Cement Paste Interface. I: Influence of Aggregate Geochemistry. *Cement and Concrete Research*, 29(7), 1019-1025.

Taylor, R.K. (1985). Cation Exchange in Clays and Mudrocks by Methylene Blue. *Journal of Chemical Technology and Biotechnology*, 35A, 195-207.

Tourenq, C. and Tran, NL. (1997). Assessment of Clays in Aggregates. A Track for Further Research. *Bulletin of the International Association of Engineering Geology*, 56: 97-102.

Unikowski, Z.R. (1982). Influence of Clays on the Properties of Cement. *Laboratoire Central des Ponts et Chaussees*, Report LPC110. (In French)

Wisconsin Department of Transportation. (1996). *State of Wisconsin Standard Specification for Road and Bridge Construction*. 1995 Ed.

White, R. H. (1987). *Introduction to the Principles and Practice of Soil Science*. Oxford: Blackwell Scientific Publishing.

Yool, A.I.G., Lees, T.P., and Fried, A. (1998). Improvements to the Methylene Blue Dye Test for Harmful Aggregates for Concrete and Mortar. *Cement and Concrete Research*, 28(10), 1417-1428

Zimbelmann, R. (1985). A Contribution to the Problem of Cement-Aggregate Bond. *Cement and Concrete Research*, 15(5), 801-808.

Zimbelmann, R. (1987). A Method for Strengthening the Bond Between Cement Stone and Aggregates. *Cement and Concrete Research*, 17(4), 651-660.

STANDARDS

AASHTO TP57-99. *Methylene Blue Value for Clays, Mineral Filler, and Fines*. Standard Specifications for Transportation Materials and Methods of Testing, 22nd Edition.

ASTM C33-00. *Standard Specification for Concrete Aggregates*. Vol 04.02.

ASTM C39-01. *Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens*. Vol 04.02.

ASTM C117-95. *Standard Test Method for Materials Finer than 75 mm (No. 200 Sieve) in Mineral Aggregates by Washing*. Vol 04.02.

ASTM C127-01. *Standard Test Method for Density, Relative Density (Specific Gravity), and Absorption of Coarse Aggregate*. Vol 04.02.

ASTM C128-01. *Standard Test Method for Density, Relative Density (Specific Gravity), and Absorption of Fine Aggregate*. Vol 04.02.

ASTM C143-00. *Standard Test Method for Slump of Hydraulic Cement Concrete*. Vol 04.02.

ASTM C192-00 *Standard Practice for Making and Curing Concrete Specimens in the Laboratory*. Vol 04.02.

ASTM C231-97. *Standard Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method*. Vol 04.02.

ASTM C295-98. *Standard Guide for Petrographic Examination of Aggregates for Concrete*. Vol 04.02.

ASTM C457-98. *Standard Test Method for Microscopial Determination of Parameters of the Air-Void System in Hardened Concrete*. Vol 04.02.

ASTM C490-00. *Standard Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete.* Vol 04.02.

ASTM C496-96. *Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens.* Vol 04.02.

ASTM C566-97. *Standard Test Method for Total Evaporable Moisture Content of Aggregate by Drying.* Vol 04.02.

ASTM C666-97. *Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing.* Vol 04.02.

ASTM C856-95. *Standard Practice for Petrographic Examination of Hardened Concrete.* Vol 04.02.

ASTM C1202-97. *Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration.* Vol 04.02.

ASTM D422-63. *Standard Test Method for Particle-Size Analysis of Soils.* Vol. 04.08

California Test No. 227. (1999). *Method of Test for Evaluating Cleanness of Coarse Aggregates.* Materials Testing Manual, December 1999.

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APPENDIX A – AGGREGATE COATING SURVEY

The WisDOT employees and Wisconsin paving contractors listed below were polled to identify the aggregate deposits that have been susceptible to aggregate coatings. Copies of the survey and its accompanying cover letter are provided in the pages that follow.

Name	Position	Company
Paul Anderson	Technical Services Manager	WisDOT District 6
Greg Bethke	Technical Services Manager	WisDOT District 5
Bill Cape	President	James Cape & Sons, Co.
Will Dorsey	Technical Services Manager	WisDOT District 3
John Geiger	Vice-President	Streu Construction Co.
Tim Hansen	Technical Services Manager	WisDOT District 8
Shau Nong Jea	Technical Services Manager	WisDOT District 2
Dave Kircher	Technical Services Manager	WisDOT District 7
Joe Lacenski	Technical Services Manager	WisDOT District 4
Michael Maples	Vice-President	Vinton Construction Co.
Gene Mueller	Engineer	Trierweiler Construction Co.
John Parisi	President	Parisi Construction Co.
Jim Parry	Test Engineer	WisDOT District 1
Kevin Patrow	Vice-President	Chippewa Concrete
Thomas Ptaschinski	President	Ptaschinski Construction Co.
Bob Serak	Construction Services Supervisor	WisDOT District 2
John Stafford	President	LaLonde-Stanford, Inc.
E.J. Streu	President	Streu Construction Co.
Barbara Voigt	Technical Services Manager	WisDOT District 1

Department of Civil and Environmental Engineering
1415 Engineering Drive
Madison, Wisconsin 53706

February 24, 2000

Dear Survey Recipient:

The attached survey is part of a study led by researchers in University of Wisconsin-Madison Civil Engineering Department. We understand the number of surveys we all encounter today are burdensome, but we truly need your help. You are one of a select few we have chosen to complete this survey and thus your response is critical to this research.

The purpose of this study is to identify deleterious aggregate coatings in the state of Wisconsin and determine to what degree they affect concrete performance. Through this survey, we hope to locate and sample, for each Wisconsin DOT district, 1 or 2 coarse aggregate sources with surface coatings suspected of having the greatest effects on portland cement concrete construction.

Simply put, aggregate coatings are films or layers of material that adhere to the surface of an aggregate. Depending on their composition and physical characteristics, aggregate coatings can have no effects, harmful effects, even beneficial effects on concrete properties. One common type of aggregate coating is made up of dust accumulated from aggregate crushing or processing. Dust coatings loosely adhere to the aggregate surface and are generally removed during mixing. Another coating type consists of clay minerals deposited from surface or ground water. This type of coating has a stronger bond to the aggregate surface and can remain attached even after several washings. These coatings are commonly referred to as "scum" and are most pronounced when the aggregate surface is wet.

Please take the few minutes needed to answer the questions that follow. When completed, return the survey in the enclosed addressed envelope by March 25. After reviewing your responses, we may contact you at a later time for further investigation of the nature and effects of the aggregate coatings you identify.

Thank you for your response. We greatly appreciate your cooperation. Please contact me directly if you have any questions or concerns.

Sincerely,

Steven M. Cramer, Ph.D., P.E.

Professor of Civil and Environmental Engineering

UNIVERSITY OF WISCONSIN AGGREGATE COATING SURVEY

1. **In the past 5 years, have you experienced aggregate coatings in your work with concrete pavement or bridge deck construction?**

❑ No. *Thank you for your participation. No further information is needed. Please return the survey in the enclosed envelope.*

❑ Yes. *Please continue survey by answering questions 2-4.*

2. **How do you perceive aggregate coatings to affect concrete pavements (check all that apply):**

❑ Cracking
❑ Popouts
❑ Spalling
❑ Scaling or Low Durability
❑ Low strength
❑ High Air Content
❑ Other (Please Specify): _____
❑ None

3. **Where and when were the suspected coated aggregates observed?**

	Project #1	Project #2	Project #3
County	_____	_____	_____
Highway	_____	_____	_____
Year of Construction	_____	_____	_____

4. **For future information on the aggregates coatings used in the projects above, please contact:**

Name _____

Position _____

Phone _____

E-mail _____

APPENDIX B – MATERIAL DATA

B.2 CEMENT COMPOSITION

Table B-1. Chemical Analysis of Cement

Chemical Compounds	Percentage (%)
Silicon Oxide (SiO_2)	21.02
Aluminium Oxide (Al_2O_3)	4.43
Iron Oxide (Fe_2O_3)	23.05
Calcium Oxide (CaO)	65.14
Magnesium Oxide (MgO)	1.93
Sulfur Trioxide (SO_3)	2.69
Potassium Oxide (K_2O)	0.58
Total Alkalies	0.51
C_3S	63.85
C_3A	6.9
Loss on Ignition	1.98

B.2 COARSE AGGREGATE GRADATIONS

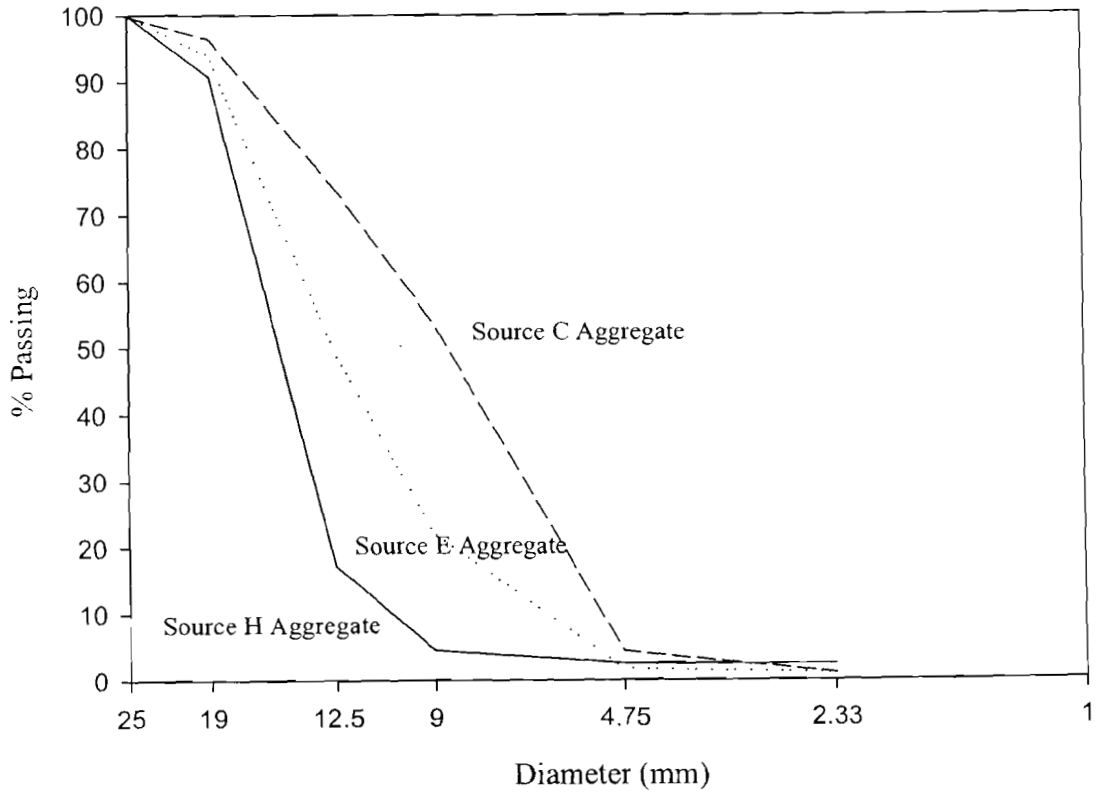

Figure B-1. Coarse Aggregate Gradations

B.3 FINE AGGREGATE GRADATION

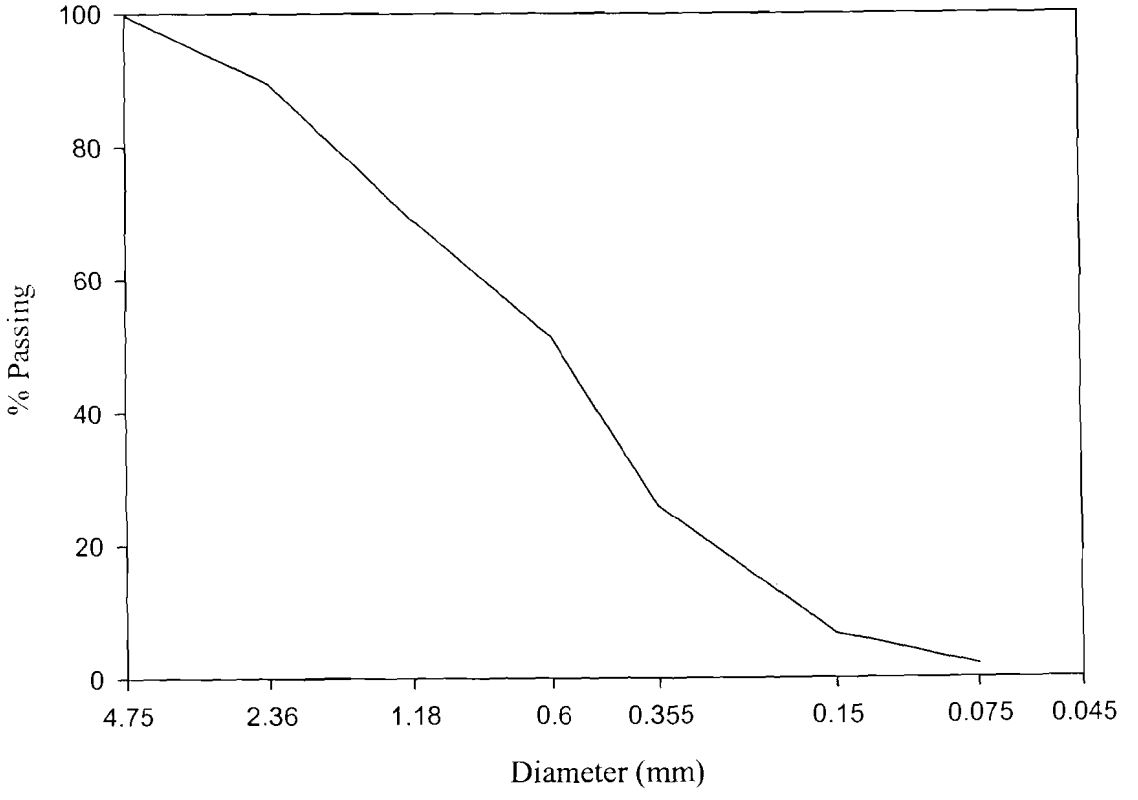

Figure B-2. Fine Aggregate Gradations
(Fineness Modulus = 3.4)

APPENDIX C – BATCH INFORMATION

Table C-1. Test Specimens And Batch Size

Test Specimen	Quantity	Individual Volume (ft^3)	Total Volume (ft^3)
Slump Test	1	0.20	0.20
Unit Weight	1	0.25	0.25
Fresh Air Content	1	0.25	0.25
Compressive Strength	4	0.06	0.23
Tensile Strength	4	0.20	0.79
Air Dry Shrinkage	3	0.11	0.32
Freeze Thaw	3	0.15	0.45
Chloride Ion Resistance (4x8)	2	0.06	0.12
Petrographic Examination	1	0.15	0.15
Air Void Analysis	1	0.15	0.15
10% Additional	1	0.13	0.13
		Total Volume:	**3.00 ft^3**

Table C-2. Batch Quantities

Batch Material	Batch 1	Batch 2	Batch 3	Batch 4	Batch 5A	Batch 5B	Batch 6	Batch 7	Batch 8	Batch 9	Batch 10
Cement	**62.7**	**62.7**	**62.7**	**62.7**	**62.7**	**62.7**	**62.7**	**62.7**	**62.7**	**62.7**	**62.7**
Coarse Aggregate	**211.2**	**211.7**	**211.1**	**209.7**	**212.8**	**211.2**	**212.4**	**211.4**	**211.9**	**211.3**	**212.0**
Absorption (%)	1.34	1.36	1.63	1.34	1.36	1.36	1.63	1.36	1.36	1.36	1.36
Moisture Content (%)	1.39	1.62	1.31	0.65	2.15	1.37	1.93	1.46	1.71	1.44	1.74
Fine Aggregate	**138.9**	**138.9**	**138.9**	**138.9**	**138.9**	**138.9**	**138.9**	**138.9**	**138.9**	**138.9**	**138.9**
Absorption (%)	0.072	0.072	0.072	0.072	0.072	0.072	0.072	0.072	0.072	0.072	0.072
Moisture Content (%)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Water	**29.12**	**28.69**	**29.91**	**30.67**	**27.56**	**29.23**	**28.59**	**29.04**	**28.50**	**29.07**	**31.02**
Net Water	28.23	28.23	28.23	28.23	28.23	28.23	28.23	28.23	28.23	28.23	28.23
Absorbed Water	0.89	0.46	1.68	2.44	-0.67	1.00	0.36	0.81	0.27	0.84	2.79
Air Entrainment (mL)	**29**	**34**	**32**	**30**	**32**	**34**	**34**	**33**	**35**	**40**	**35**

APPENDIX D – X-RAY DIFFRACTION PLOTS

D.1 PHASE II AGGREGATE COATINGS

For each aggregate collected in Phase II, the surface coating was removed by washing over a No. 200 sieve, dried to evaporate the wash water, and tested by x-ray diffraction. Refer to Table 4-1 for the minerals that were identified in each sample and to the map in Figure 3-2 for the locations of each aggregate source.

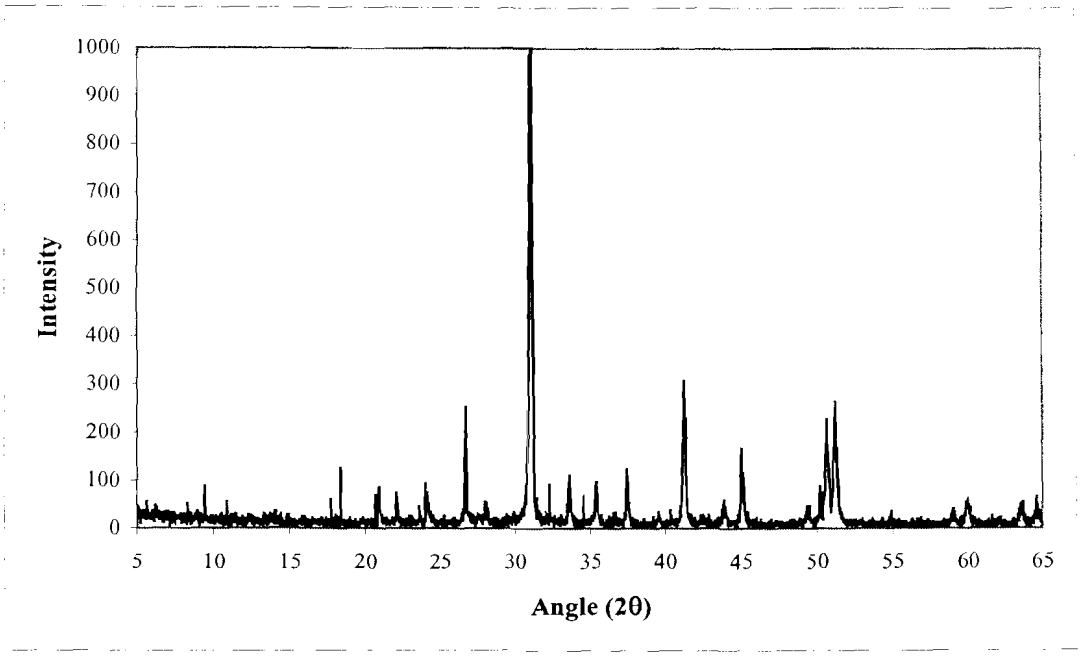

Figure D-1. Source A Aggregate Coating

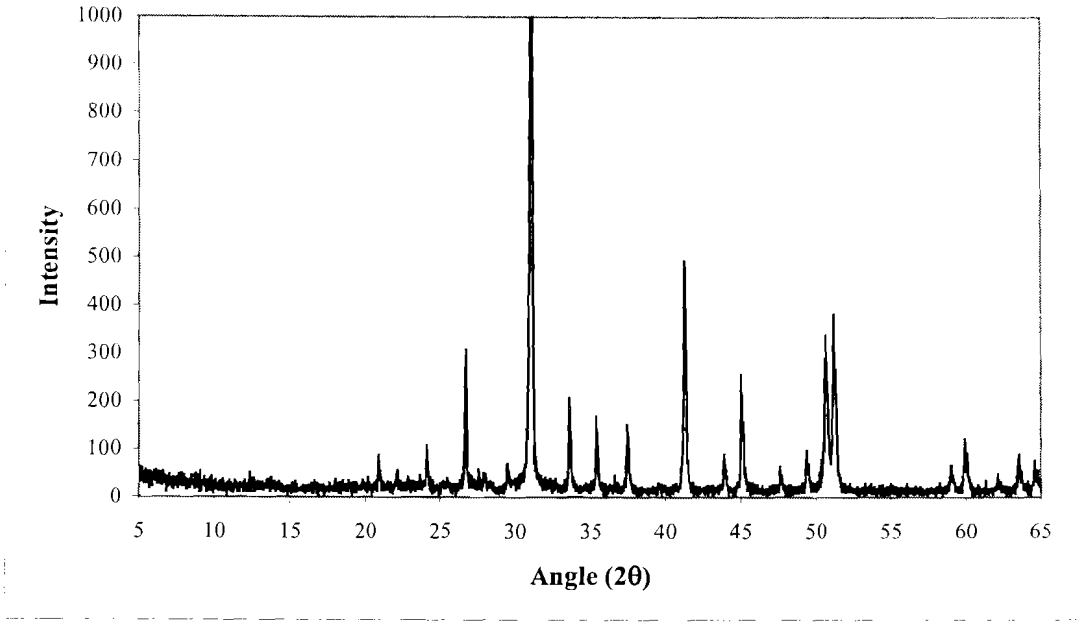

Figure D-2. Source B Aggregate Coating

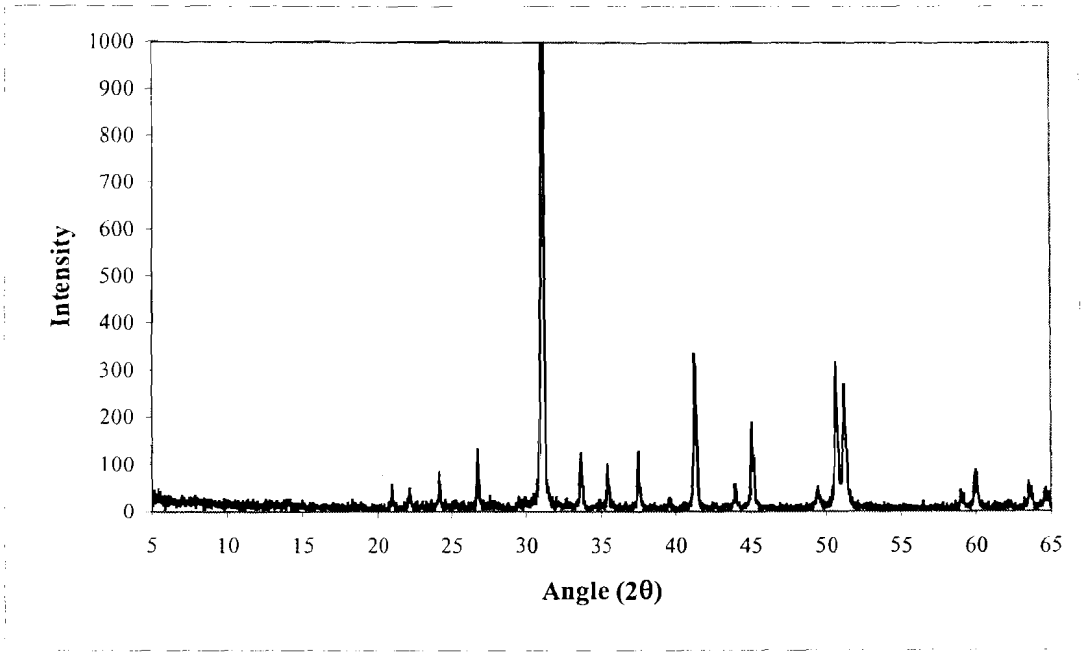

Figure D-3. Source C Aggregate Coating

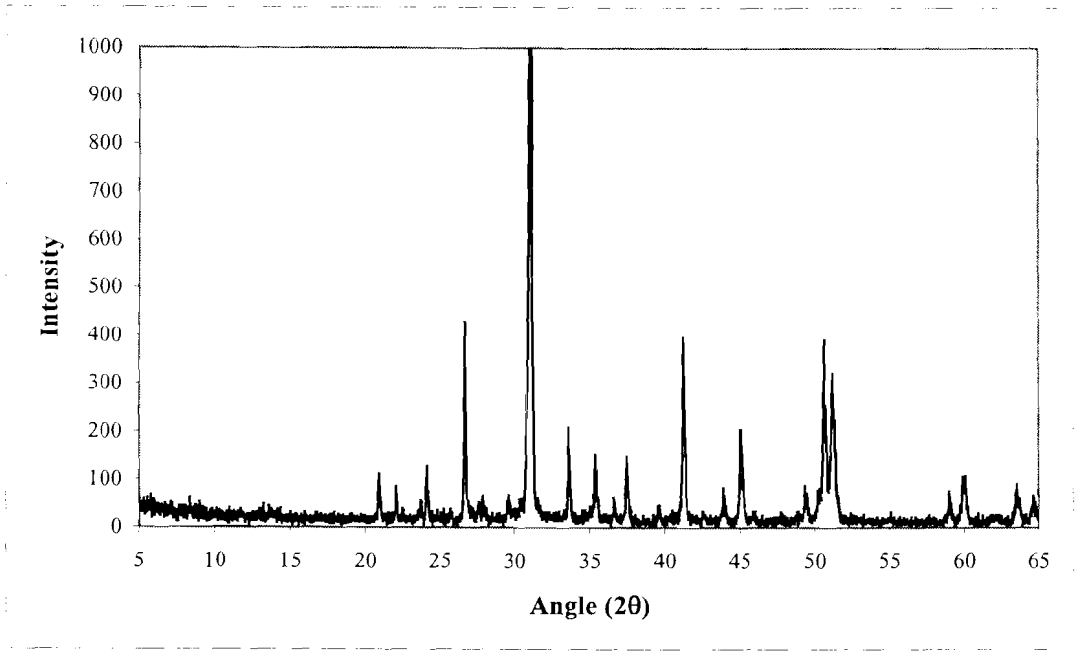

Figure D-4. Source D Aggregate Coating

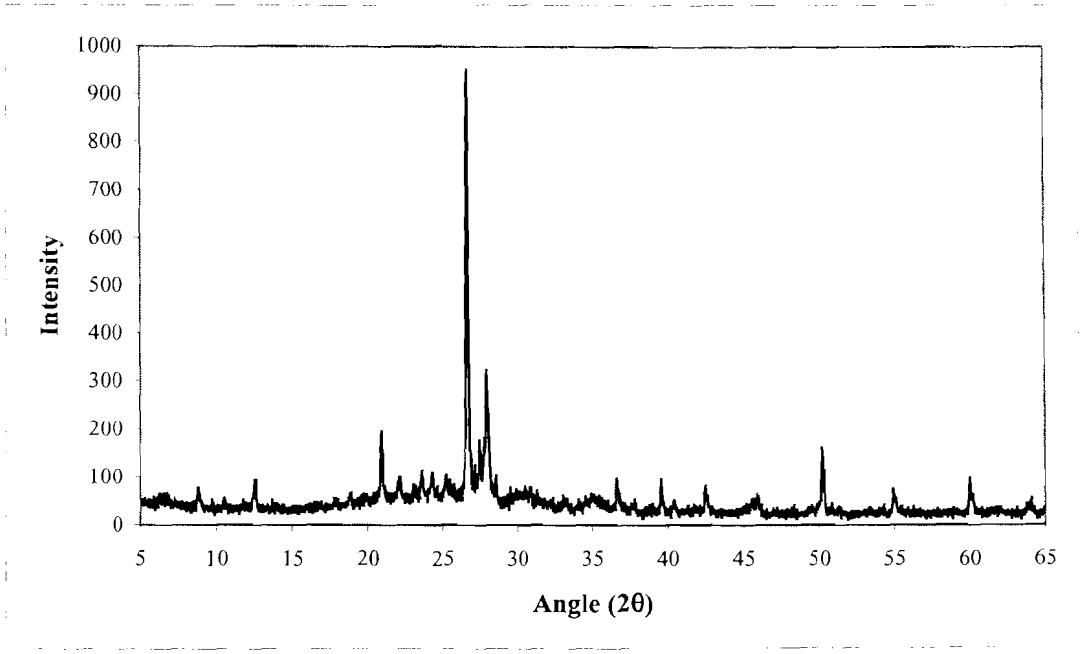

Figure D-5. Source E Aggregate Coating

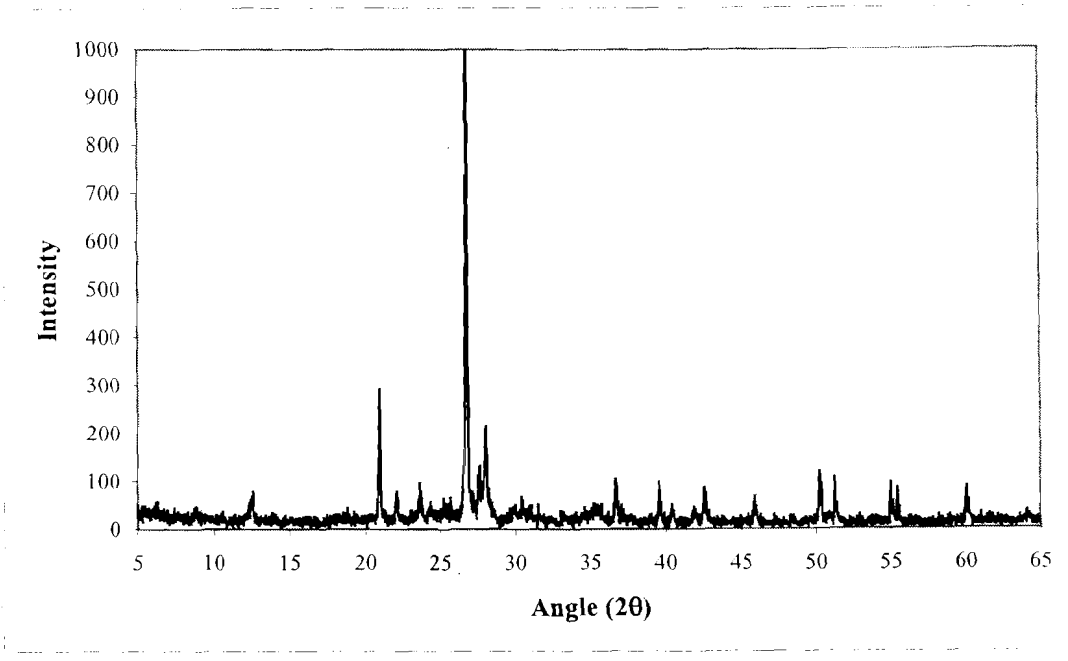

Figure D-6. Source F Aggregate Coating

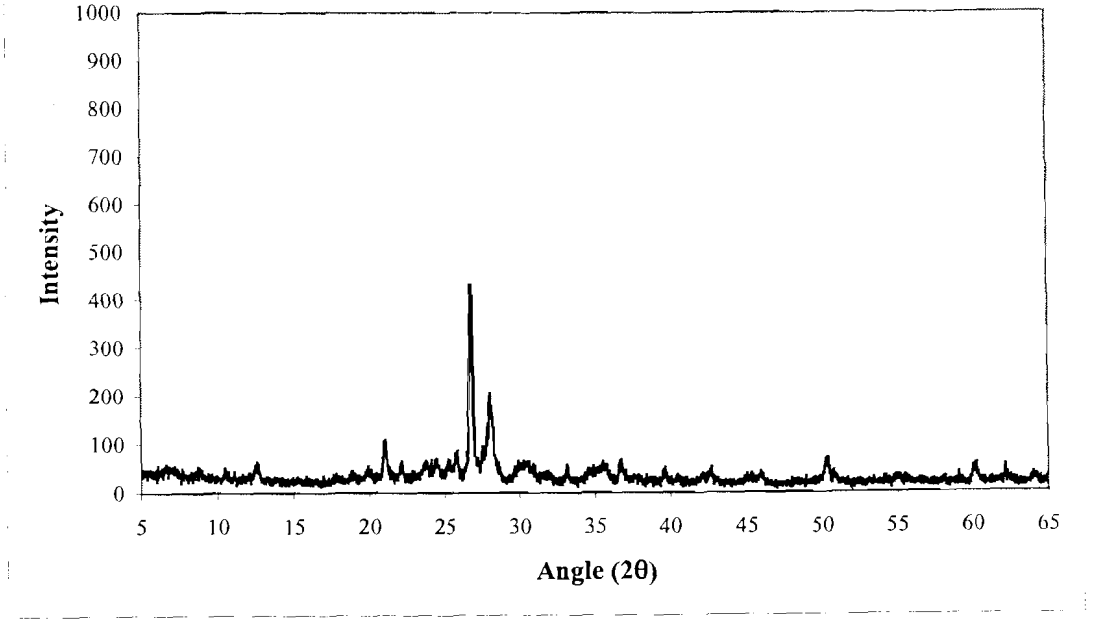

Figure D-7. Source G Aggregate Coating

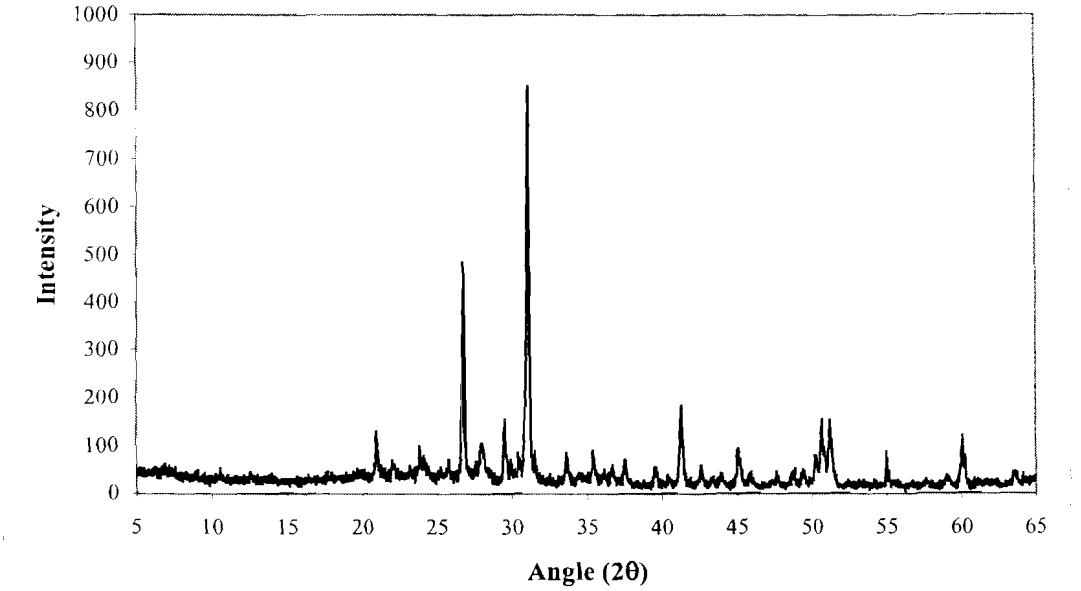

Figure D-8. Source H Aggregate Coating

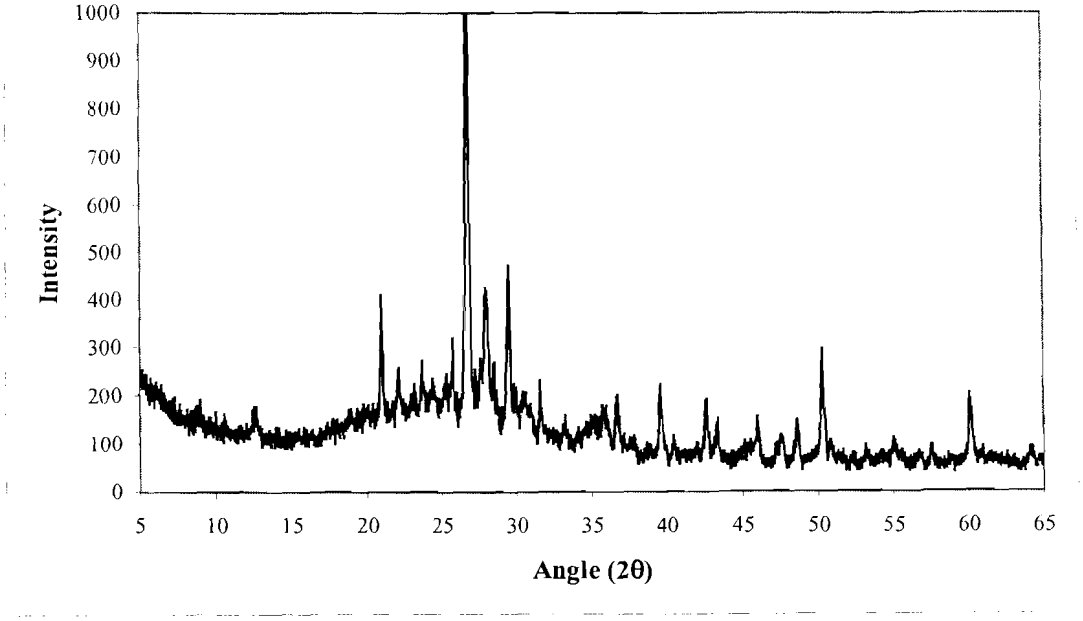

Figure D-9. Source I Aggregate Coating

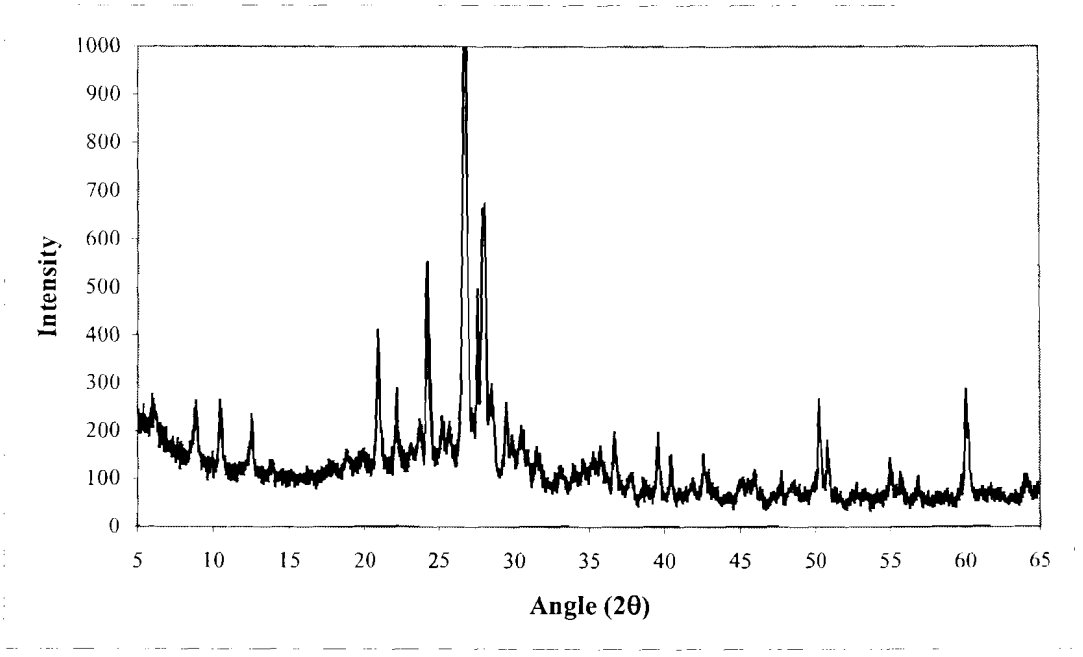

Figure D-10. Source J Aggregate Coating

D.2 PHASE II AGGREGATE COATINGS

For the aggregates tested in the Coated Aggregate Series-Lab during Phase III, coatings were created by artificially attaching fine material collected from sites in Barron Co. and Sauk Co. to the Source E aggregate. In order to classify the type of coatings created, each sample was tested by x-ray diffraction prior to mixing. Refer to Table 4-2 for the minerals that were identified.

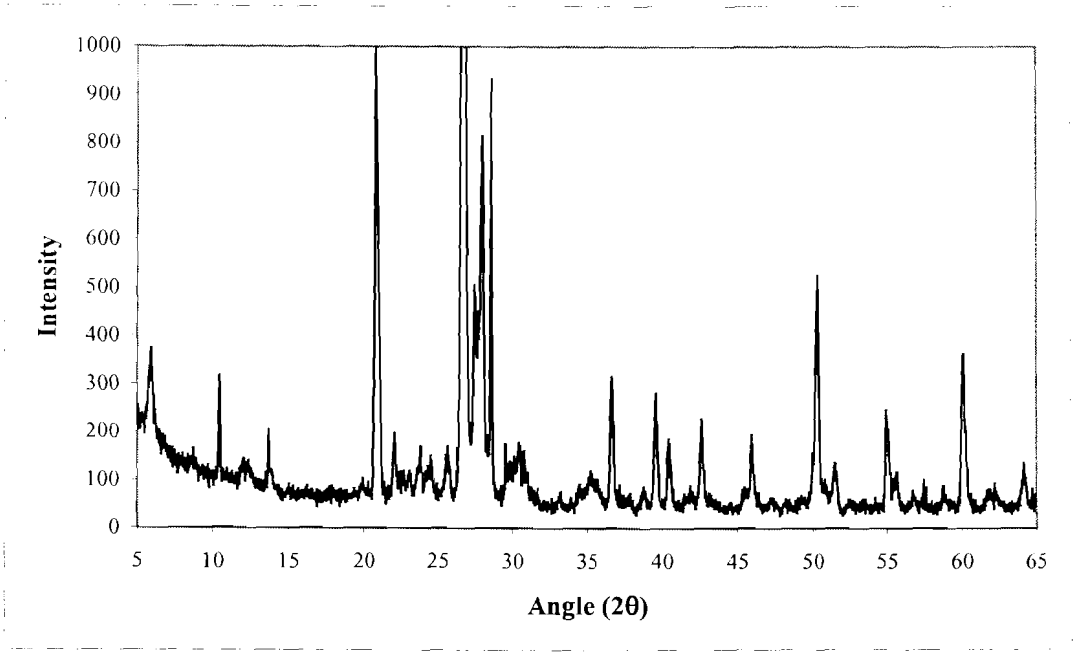

Figure D-11. Barron Co. Fines

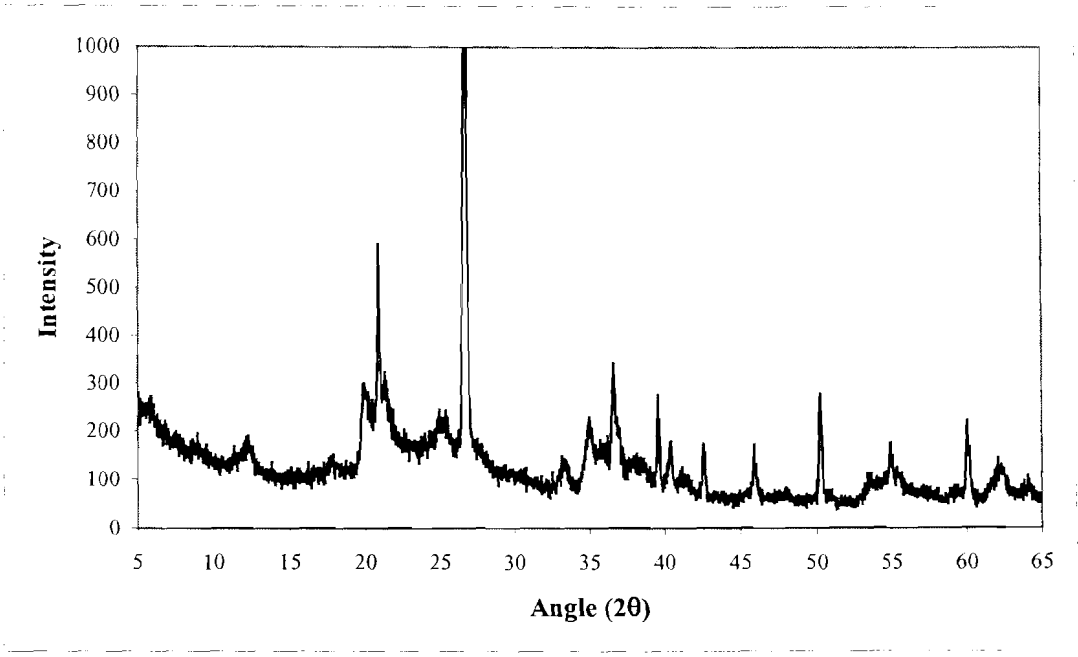

Figure D-12. Sauk Co. Fines

APPENDIX E – ANOVA RESULTS

E.1 Compressive Strength

Table E-1. ANOVA of Source C Aggregates on Compressive Strength

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
1 (Coated - Field)	3	13485	4495	25822
4 (Washed - Lab)	4	17786	4447	73664

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	4020	1	4020	0.074	6.61
Within Batch	272638	5	54528		
Total	276658	6			

Table E-2. ANOVA of Source E Aggregates on Compressive Strength

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
2 (Coated - Field	4	19174	4793	25779
5B (Washed - Lab)	4	19278	4820	16282
7 (Coated - Lab)	4	18517	4629	18459
8 (Coated -Lab)	4	18858	4714	2569
9 (Coated - Lab)	4	18397	4599	13176
10 (Coated - Lab)	4	18390	4598	2530

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	41708873	5	8341775	1.942	2.77
Within Batch	77301423	18	4294524		
Total	119010296	23			

Table E-3. ANOVA of Source H Aggregates on Compressive Strength

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
3 (Coated - Field)	4	19116	4779	79586
6 (Washed - Lab)	4	18122	4531	20287

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	123415	1	123415	2.47	5.99
Within Batch	299621	6	49937		
Total	423036	7			

E.2 TENSILE STRENGTH

Table E-4. ANOVA of Source C Aggregates on Tensile Strength

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
1 (Coated - Field)	4	1634	408	390
4 (Washed - Lab)	4	1378	344	971

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	8195	1	8195	12.05	5.99
Within Batch	4082	6	680		
Total	12277	7			

Table E-5. ANOVA of Source E Aggregates on Tensile Strength

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
2 (Coated - Field	4	1713	428	349
5B (Washed - Lab)	4	1632	408	1125
7 (Coated - Lab)	4	1517	379	707
8 (Coated -Lab)	4	1589	397	3319
9 (Coated - Lab)	4	1881	470	3799
10 (Coated - Lab)	4	1463	366	480

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	28033	5	5607	3.44	2.77
Within Batch	29336	18	1630		
Total	57369	23			

Table E-6. ANOVA of Source H Aggregates on Tensile Strength

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
3 (Coated - Field)	4	1591	398	3973
6 (Washed - Lab)	4	1454	364	3148

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	2339	1	2339	0.657	5.99
Within Batch	21364	6	3561		
Total	23704	7			

E.3 DRYING SHRINKAGE

Table E-7. ANOVA of Source C Aggregates on Drying Shrinkage

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
1 (Coated - Field)	3	-0.1155	-0.0385	1.69E-04
4 (Washed - Lab)	3	-0.1162	-0.0387	1.24E-05

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	9.44E-08	1	9.4E-08	0.001	7.71
Within Batch	3.63E-04	4	9.1E-05		
Total	3.63E-04	5			

Table E-8. ANOVA of Source E Aggregates on Drying Shrinkage

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
2 (Coated - Field	3	-0.130	-0.043	4.91E-05
5B (Washed - Lab)	3	-0.103	-0.034	2.33E-06
7 (Coated - Lab)	3	-0.126	-0.042	3.00E-06
8 (Coated -Lab)	3	-0.139	-0.046	1.03E-05
9 (Coated - Lab)	3	-0.146	-0.049	9.33E-06
10 (Coated - Lab)	3	-0.167	-0.056	3.33E-07

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	7.65E-04	5	1.53E-04	12.335	2.85
Within Batch	1.49E-04	12	1.24E-05		
Total	9.14E-04	17			

Table E-9. ANOVA of Source H Aggregates on Drying Shrinkage

Effect of Carbonate/Clay Coating on Source H Aggregate

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
3 (Coated - Field)	3	-0.115	-0.038	1.50E-03
6 (Washed - Lab)	3	-0.120	-0.040	1.61E-03

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	5.37E-06	1	5.4E-06	0.16	7.71
Within Batch	1.35E-04	4	3.4E-05		
Total	1.40E-04	5			

E.4 FREEZE-THAW DURABILITY

E.4.1 Freeze-Thaw Durability at 100 Cycles

Table E-10. ANOVA of Source C Aggregates on Stiffness Durability (100 cycles)

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
1 (Coated - Field)	3	258.6	86.2	133.2
4 (Washed - Lab)	3	275.3	91.8	57.7

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	46.6	1	46.62	0.488	7.71
Within Batch	381.8	4	95.46		
Total	428.4	5			

Table E-11. ANOVA of Source C Aggregates on Weight Durability (100 cycles)

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
1 (Coated - Field)	3	300.0	100.0	0.002
4 (Washed - Lab)	3	300.4	100.1	0.086

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	0.038	1	0.038	0.872	7.71
Within Batch	0.177	4	0.044		
Total	0.215	5			

Table E-12. ANOVA of Source E Aggregates on Stiffness Durability (100 cycles)

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
2 (Coated - Field	3	297.1	99.0	0.6
5B (Washed - Lab)	3	300.0	100.0	0.4
7 (Coated - Lab)	3	301.8	100.6	0.2
8 (Coated -Lab)	3	301.2	100.4	0.2
9 (Coated - Lab)	3	300.7	100.2	0.2
10 (Coated - Lab)	3	296.3	98.8	1.1

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	8.63	5	1.73	3.879	2.85
Within Batch	5.34	12	0.45		
Total	13.97	17			

Table E-13. ANOVA of Source E Aggregates on Weight Durability (100 cycles)

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
2 (Coated - Field	3	300.5	100.2	0.014
5B (Washed - Lab)	3	300.2	100.1	0.013
7 (Coated - Lab)	3	299.9	100.0	0.044
8 (Coated -Lab)	3	299.9	100.0	0.031
9 (Coated - Lab)	3	300.1	100.0	0.001
10 (Coated - Lab)	3	299.8	99.9	0.030

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	0.112	5	0.022	1.015	2.85
Within Batch	0.265	12	0.022		
Total	0.377	17			

Table E-14. ANOVA of Source H Aggregates on Stiffness Durability (100 cycles)

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
3 (Coated - Field)	3	273.4	91.1	7.60
6 (Washed - Lab)	3	257.1	85.7	35.5

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	44.5	1	44.5	2.07	7.71
Within Batch	86.1	4	21.5		
Total	130.7	5			

Table E-15. ANOVA of Source H Aggregates on Weight Durability (100 cycles)

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
3 (Coated - Field)	3	299.0	99.7	0.050
6 (Washed - Lab)	3	299.8	99.9	0.006

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	0.111	1	0.111	4.00	7.71
Within Batch	0.111	4	0.028		
Total	0.22	5			

E.4.2 Freeze-Thaw Durability at 300 Cycles

Table E-16. ANOVA of Source C Aggregates on Stiffness Durability (300 cycles)

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
1 (Coated - Field)	3	251.0	83.7	274
4 (Washed - Lab)	3	260.0	86.7	83

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	13.5	1	13.5	0.076	7.71
Within Batch	714.9	4	178.7		
Total	728.4	5			

Table E-17. ANOVA of Source C Aggregates on Weight Durability (300 cycles)

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
1 (Coated - Field)	3	300.0	100.0	0.002
4 (Washed - Lab)	3	300.4	100.1	0.086

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	0.038	1	0.038	0.872	7.71
Within Batch	0.177	4	0.044		
Total	0.215	5			

Table E-18. ANOVA of Source E Aggregates on Stiffness Durability (300 cycles)

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
2 (Coated - Field	3	296.9	99.0	0.10
5B (Washed - Lab)	3	303.0	101.0	0.73
7 (Coated - Lab)	3	306.1	102.0	0.07
8 (Coated -Lab)	3	299.8	99.9	2.03
9 (Coated - Lab)	3	297.8	99.3	0.33
10 (Coated - Lab)	3	292.6	97.5	18.50

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	37.4	5	7.47	2.06	2.85
Within Batch	43.5	12	3.63		
Total	80.9	17			

Table E-19. ANOVA of Source E Aggregates on Weight Durability (300 cycles)

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
2 (Coated - Field	3	299.9	100.0	0.013
5B (Washed - Lab)	3	298.8	99.6	0.016
7 (Coated - Lab)	3	299.4	99.8	0.087
8 (Coated -Lab)	3	298.6	99.5	0.039
9 (Coated - Lab)	3	294.3	98.1	0.055
10 (Coated - Lab)	3	294.2	98.1	2.50

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	11.1	5	2.22	4.910	2.85
Within Batch	5.42	12	0.45		
Total	16.5	17			

Table E-20. ANOVA of Source H Aggregates on Stiffness Durability (300 cycles)

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
3 (Coated - Field)	3	245.6	81.9	39.2
6 (Washed - Lab)	3	222.9	74.3	80.4

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	85.8	1	85.8	1.43	7.71
Within Batch	239.2	4	59.8		
Total	325.0	5			

Table E-21. ANOVA of Source H Aggregates on Weight Durability (300 cycles)

Effect of Carbonate/Clay Coatings on Source H Aggregate

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
3 (Coated - Field)	3	296.8	98.9	0.124
6 (Washed - Lab)	3	292.7	97.6	10.3

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	2.79	1	2.79	0.535	7.71
Within Batch	20.9	4	5.21		
Total	23.6	5			

E.5 CHLORIDE ION PENETABILITY

Table E-22. ANOVA of Source C Aggregates on Chloride Ion Penetration

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
1 (Coated - Field)	2	8865	4433	136294
4 (Washed - Lab)	2	7079	3540	97726

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	797449	1	797449	6.82	18.50
Within Batch	234020	2	117010.2		
Total	1031469	3			

Table E-23. ANOVA of Source E Aggregates on Chloride Ion Penetration

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
2 (Coated - Field	2	6654	3327	60309
5B (Washed - Lab)	2	6778	3389	140185
7 (Coated - Lab)	2	7420	3710	874
8 (Coated -Lab)	2	6495	3247	30233
9 (Coated - Lab)	2	7596	3798	7188
10 (Coated - Lab)	2	10018	5009	41732

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	4301773	5	860355	18.40	3.87
Within Batch	280520	6	46753		
Total	4582293	11			

Table E-24. ANOVA of Source H Aggregates on Chloride Ion Penetration

SUMMARY

Batch (Series)	*Count*	*Sum*	*Average*	*Variance*
3 (Coated - Field)	2	9128	4564	236053
6 (Washed - Lab)	2	6926	3463	12372

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*F crit*
Between Batches	1211761	1	1211761	9.76	18.5
Within Batch	248425	2	124212		
Total	1460185	3			

APPENDIX F – CONCRETE PETROGRAPHY

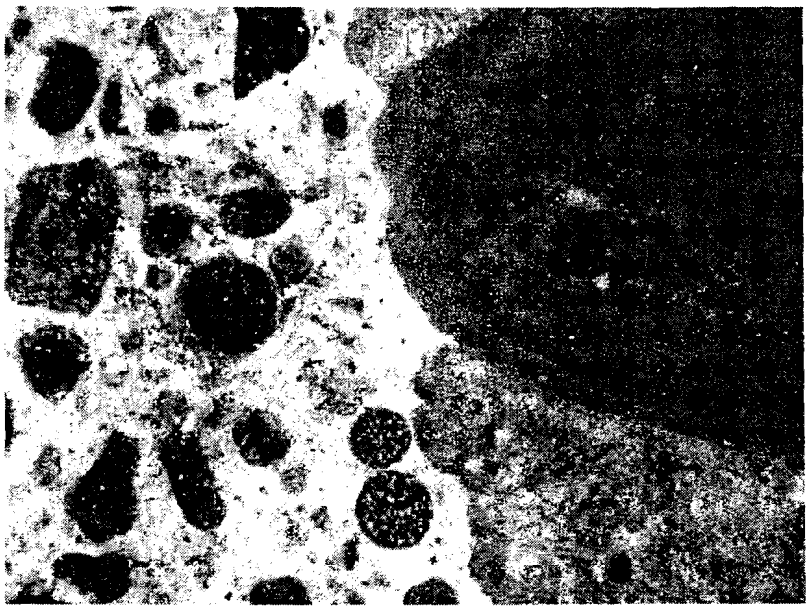

Figure F-1. Photomicrograph of Batch 2 Thin-Section (Magnification = 200x)

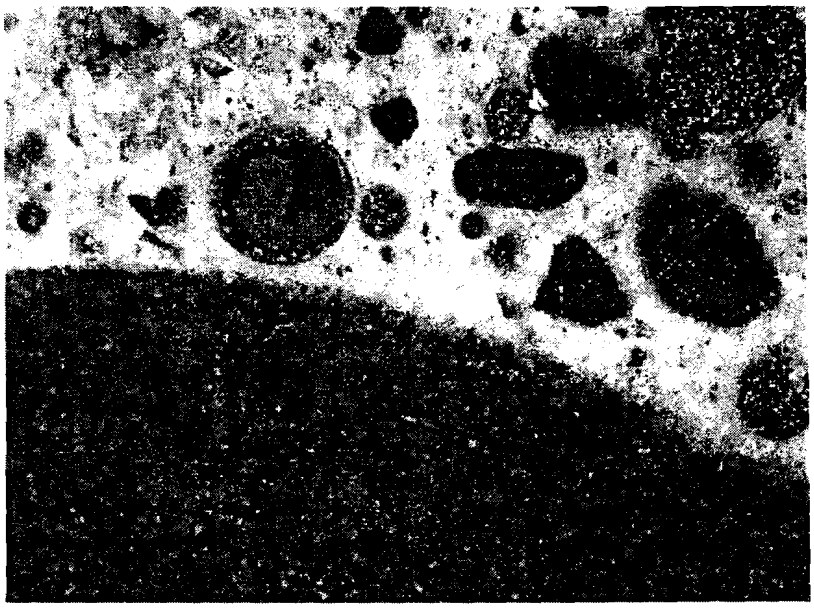

Figure F-2. Photomicrograph of Batch 5B Thin-Section (Magnification = 200x)

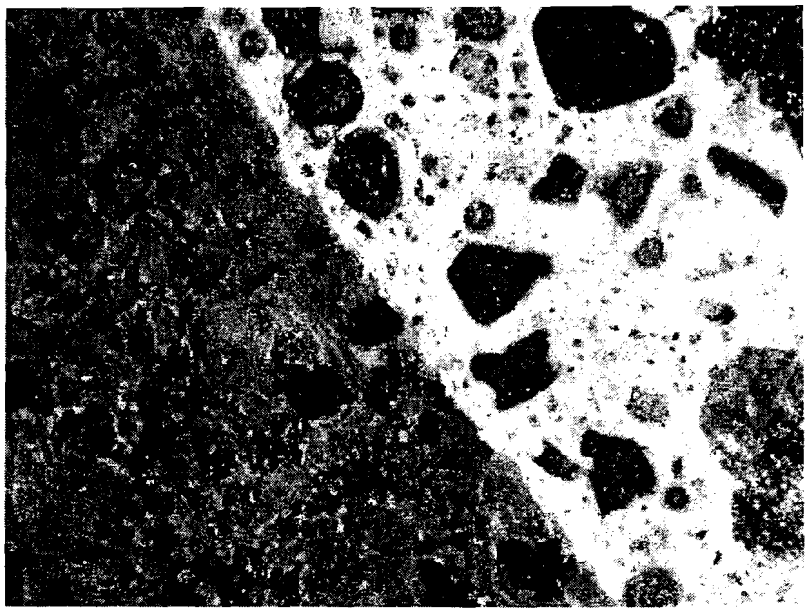

Figure F-3. Photomicrograph of Batch 8 Thin-Section (Magnification = 200x)

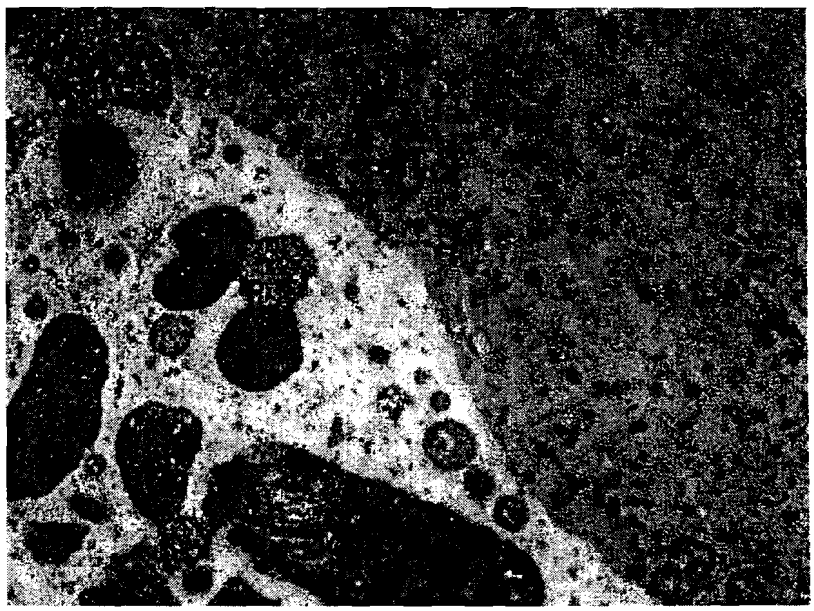

Figure F-4. Photomicrograph of Batch 10 Thin-Section (Magnification = 200x)