IOWA STATE UNIVERSITY Digital Repository

[Graduate Theses and Dissertations](https://lib.dr.iastate.edu/etd?utm_source=lib.dr.iastate.edu%2Fetd%2F14946&utm_medium=PDF&utm_campaign=PDFCoverPages)

[Iowa State University Capstones, Theses and](https://lib.dr.iastate.edu/theses?utm_source=lib.dr.iastate.edu%2Fetd%2F14946&utm_medium=PDF&utm_campaign=PDFCoverPages) **[Dissertations](https://lib.dr.iastate.edu/theses?utm_source=lib.dr.iastate.edu%2Fetd%2F14946&utm_medium=PDF&utm_campaign=PDFCoverPages)**

2015

Laboratory evaluation of freezing-thawing resistance of aggregate for concrete pavement using Iowa Pore Index Tests

Jiaxi Ren *Iowa State University*

Follow this and additional works at: [https://lib.dr.iastate.edu/etd](https://lib.dr.iastate.edu/etd?utm_source=lib.dr.iastate.edu%2Fetd%2F14946&utm_medium=PDF&utm_campaign=PDFCoverPages) Part of the [Civil Engineering Commons](http://network.bepress.com/hgg/discipline/252?utm_source=lib.dr.iastate.edu%2Fetd%2F14946&utm_medium=PDF&utm_campaign=PDFCoverPages)

Recommended Citation

Ren, Jiaxi, "Laboratory evaluation of freezing-thawing resistance of aggregate for concrete pavement using Iowa Pore Index Tests" (2015). *Graduate Theses and Dissertations*. 14946. [https://lib.dr.iastate.edu/etd/14946](https://lib.dr.iastate.edu/etd/14946?utm_source=lib.dr.iastate.edu%2Fetd%2F14946&utm_medium=PDF&utm_campaign=PDFCoverPages)

This Thesis is brought to you for free and open access by the Iowa State University Capstones, Theses and Dissertations at Iowa State University Digital Repository. It has been accepted for inclusion in Graduate Theses and Dissertations by an authorized administrator of Iowa State University Digital Repository. For more information, please contact digirep@iastate.edu.

Laboratory evaluation of freezing-thawing resistance of aggregate for concrete pavement using Iowa Pore Index Tests

by

Jiaxi Ren

A thesis submitted to the graduate faculty

in partial fulfillment of the requirement for the degree of

MASTER OF SCIENCE

Major: Civil Engineering (Civil Engineering Materials)

Program of Study Committee: Kejin Wang, Co-Major Professor Fatih Bektas, Co-Major Professor Amy Froelich

Iowa State University

Ames, Iowa

2015

Copyright © Jiaxi Ren, 2015. All rights reserved

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

ACKNOWLEDGEMENTS

I would like to thank my major professors, Dr. Kejin Wang and Dr. Fatih Bektas for their guidance and supports through this research and my master study. I'm grateful to Dr. Wang for her support and advices on my master study at Iowa State University. And I would like to give thanks to Dr. Fatih Bektas for his guidance, patience, and assistance given to me throughout my study. I sincerely appreciate their patience and contributions in this research work.

I would like to thank Dr. Amy Froelich for her valuable time and efforts serving as a committee member.

The research was supported by the Minnesota Department of Transportation (MnDOT). I would like to thank to them for the advices and assistances.

I would like to thank PCC research laboratory manager, Bob Steffes, for his assistances on this research work. I also want to offer my appreciation to my friends, colleagues, department faculties and staffs for making my time at Iowa State University a wonderful experience.

I want to give my special thanks to my grandparents for their supports, love and encouragement. I also want to give thanks to my friends Jianhua Yu, and Zhao Cheng for their supports and assistances.

ABSTRACT

This study is aimed at evaluating Iowa pore index (IPI) test method for characterizing pores in aggregates that may have significant influence on freezing-thawing (F-T) durability of concrete. The study includes three phases: (a) evaluating the pore indexes of various concrete aggregates from different sources that mostly contain both carbonate and non-carbonate portions; (b) testing volumetric properties (such as specific gravity, absorption and desorption) of the aggregate; and (c) examining the correlations between these test results. Statistical analyses were also applied in the examination of these correlations.

The results of this study suggest that IPI test is a quick and simple test method for aggregate pore structure evaluation. Carbonate aggregates generally have higher absorption, lower specific gravity, higher primary pore index (PPI), and much higher secondary pore index (SPI) (for a given absorption) than non-carbonate aggregates. The PPI and PPI+SPI measurements are closely related to absorption of aggregates (for carbonate, non-carbonate, and bulk aggregates). The correlations of SPI with aggregate absorption are very weak, especially for carbonate aggregate. This may be attributed by other unidentified factors (such as aggregate pore tortuosity and the IPI test pressure and time). A pattern has been observed for the residual of the linear regression model for SPI-absorption correlation, which suggests that a higher order regression may be needed to better describe the correlation between SPI and absorption for carbonate aggregate.

More research should be conducted to further study the correlation between F-T durability and aggregate chemistry, mineralogy, and pore structure. Additional tests should also be done to develop statistical model better correlates the aforementioned testing results and aggregate property.

CHAPTER 1. INTRODUCTION

1.1 Organization of Thesis

This thesis is organized into five chapters: (1) Introduction; (2) Literature reviews; (3) Experimental work; (4) Results and discussion; and (5) Conclusions and recommendations.

Chapter 1 introduces the problems on freezing-thawing (F-T) durability of concrete. And the lack of superior coarse aggregate situation in Minnesota. Great efforts have been made on concrete pavement repairing and maintenance during the winter drive people to maximize the longevity of pavement by seeking for the better performance coarse aggregate.

Chapter 2 summarizes literatures of previous studies on mechanics of F-T deterioration of coarse aggregate and its surrounding paste, causes of aggregate-related F-T deterioration, effects of aggregate properties on concrete soundness, material characterization methods, especially the commonly used test methods in the Midwestern states, and the current specifications for aggregate acceptance in Minnesota.

Chapter 3 summarizes the materials and research methodology adopted in the experimental program of this study. The types, sources, and sampling process of the materials and the test procedures are presented.

Chapter 4 presents the test results and findings. The discussion about the test results is focused on:

- Carbonate content, absorption, and specific gravity,
- Pore index summary and relationship between aggregate properties and pore index, and

Rate of absorption and desorption.

Chapter 5 concludes the research findings. Recommendations for future work are provided to improve the quality of study on IPI performance and concrete durability problems.

1.2 Problem Statement

Highway network is vital to the society and economy of the United States. It has the largest number of users in comparisons to other transportation types, such as railway, air transport, or water. It is also the important method for domestic transport of cargos. Safety of the highway network is important to citizens as well as economy. Based on Federal Highway Administration (FHWA), crashes related to winter weather causes thousands of fatalities and considerable property losses [1].

Figure 1: Snowy Region of United States [1]

A major contributor to unsafe road conditions in the winter season isthe pavement damages associated with F-T deterioration of concrete. F-T resistance of concrete material is affected by

various factors including quality of aggregate, cement type and properties, various additives, mix design, etc. Coarse aggregate property is one of the most important factors that affect the material's ability to withstand F-T damages. Because of its pore characteristics, carbonate aggregate usually been considered as the mainly culprit that cause F-T deterioration of concrete.

Minnesota is facing the issue that quality coarse aggregates are diminishing. Alternative resources of coarse aggregate become more and more interested. The properties of these alternative resources need to be determined include carbonate content, absorption, and F-T durability.

1.3 Significance of This Study

F-T deterioration usually occurs on the aged concrete pavement and under the winter weather environment. The maintenance expenses of fixing pavement damages resulted by F-T are enormous. Use high quality aggregates with favored properties in the design of concrete provides good F-T durability which may significantly reduce the maintenance costs during pavement service life. Carbonate based aggregate requires an elaborate analysis whether it can be used as a substitute alternative of superior coarse aggregate. Most of the aggregate characterization tests for F-T durability need long time period to simulate the F-T deterioration process. A simple, sketchy field test methods will draw rein and time. Small pores less than 500 nanometers in aggregate particle provide channel for water to enter the aggregate particle; however, do not easily accommodate the volume change of water during ice formation. These small pores are closely related to the F-T resistance of aggregate. Iowa Pore Index (IPI) Test measures the amount of such small pores in a timely manner. In comparison to the conventional F-T durability test, this test method measures the F-T durability of aggregate with a relatively quick and indirect approach.

Besides the differences of test methods, the damage associated with F-T deterioration on Portland Cement Concrete pavement is related to a variety of causes such as environment and temperature, pore structures, sulfate soundness, absorption ability, chemical reactions between concrete constituents, etc. These interrelated deleterious effects make the mechanism of F-T deterioration sophisticated. Thus, it is important to provide more information on this topic.

1.4 Objectives of the research

The intent of this is study is to use a fast and convenient test method to evaluate the differences in F-T durability between carbonate and non-carbonate aggregates. The objectives of this study are: 1, Evaluate the IPI Test; 2, study IPI Test's relationship with other pore structure related aggregate properties, such as absorption, desorption, and specific gravity; and 3, establish a new acceptance criterion for aggregate F-T durability for Minnesota DOT.

1.5 Scope of the Research

The scope of the research covers the following:

- Conduct literature search on F-T durability of aggregate and concrete,
- Evaluate pore structure of aggregate using IPI Test,
- Identify the difference in pore structure between carbonate and non-carbonate aggregate,
- Explore relationships among aggregate pore structure parameters (such as IPI), and other properties (such as absorption and rate of absorption), and
- Study acceptance criteria for concrete aggregate.

The first part of the research work evaluated IPI of various aggregates with different carbonate content. In this part, natural gravels from 15 different sources were tested for carbonate and non-carbonate portions, and IPI Test were performed on each of the aggregate portion. The second part inspected the pore structure of aggregate from the results of Specific Gravity and Absorption Test. Saturated-surface-dry (SSD) weight in air, apparent specific gravity and absorption of carbonate and non-carbonate aggregates were tested in this part. In the third part, Rate of Absorption/Desorption Test indicate the amount of the pore of the micro-structures of aggregate. And the relationships among the results of tests were analyzed.

CHAPTER 2. LITERATURE REVIEW

Distresses associated with F-T damage in coarse aggregate are concerned for Portland cement concrete (PCC) and one of the common causes to PCC failure. F-T deterioration causes cracking and spalling around the pavement joints or edges by progressive expansion of the cement paste. Such an aggregate-related F-T deterioration is also called D-cracking, which forms a capital "D" along pavement joints. The behavior of water in frost condition also contributes to internal pressures resulting cracking. The effects of frost action on two major components of concrete, cement paste and aggregate, can be different. But, both lead to frost damage and F-T deterioration [2].

This chapter provides a comprehensive review of aggregate properties that influence the concrete F-T durability. The applied testing procedures to qualify or quantify the influence of aggregate on concrete F-T durability were also investigated. The test procedures, applicability, advantages and disadvantages are also discussed.

2.1 F-T Deterioration

Pavement concrete nearby joints was often saturated during a cold weather winter season. As the environment temperature dropped below the freezing point, water in some pores of concrete (both paste and aggregates) became ice, which increased volume by 91% of the volume of the original water [8]. The super cooling water, which was not frozen under the freezing temperature, would been pushed to move from small capillary voids to relatively bigger voids so as to form ice there. Such a water movement, together with increased volume resulting from ice formation, would generate significant stresses in aggregates and paste and caused aggregates and paste to crack. Figure 2 shows the pavement joint and aggregate suffered from F-T deterioration.

(a) D-cracking at a Joint Intersection (b) Fractured Carbonate Aggregate

Figure 2: Frost Damage Associated with Concrete Aggregate [4]

2.1.1 Mechanisms of F-T Deterioration

The investigation of the mechanism of F-T deterioration was started in 1930s after Dcracking was observed as the first time [5]. At the present time, several theories have been proposed to explain the mechanism of the frost action in concrete. Some of these theories can be used to explain the F-T damage in aggregate particles.

Powers and Helmuth [6] indicated distending pressures contributes to some cracking types of concrete. Under the frost environment, water is frozen in the large capillary pores; while the water in the small gel pores is usually unfrozen. Because of the differences in the solute concentrations, the unfrozen water will be attracted to ice and expand.

Verbeck and Landgren [7] developed theory to explain the development of D-cracking. The authors found that: 1, the effect of aggregate size is critical; some of aggregate particles can't afford the pressures developed during freezing process which will lead to fracture and distress of the concrete. The difficulty of expelling excess will increase with the increase of particles size; 2, Some aggregates have elastically expand behavior during a increasing freezing pressure, while the

surrounding mortar usually is not able to accommodate the expansion and will fracture; and 3, Some highly absorptive aggregates will influence the expelling pressures and lead to fracture under high pressure.

Verbeck and Landgren [7] proposed three classes of concrete aggregates:

- Low permeability aggregates Aggregates in this class usually have a porosity less than 0.3%. Because of the low porosity, very little water can be absorbed by this type of aggregate. The stress resulting from water freezing is not sufficient to cause damage to the aggregate structure.
- Intermediate permeability aggregates Aggregates in this class usually have a considerable number of capillary pores in their structure. The capillary pores are typically small with a diameter less than 500 nanometer. These pores are capable of holding large amount of water by capillary force. When the absorbed water is freezing at a certain speed, these small pores do not allow the freezing water to dissipate. The change in volume of the trapped water applies pressure to the aggregate and can cause the aggregate to fracture.
- High permeability aggregates Aggregates in this class generally have considerable pores in larger size in comparisons to those in the other classes. The large pore size allows water to dissipate easily during freezing. Therefore, pressure will not be developed in the pores; and the aggregates are expected to have good F-T performance.

F-T deterioration occurs when the retained water in the pores in aggregate is freezing causing volume change. Such change in volume applies a disruptive pressure which can fracture the aggregate particle and lead to failure of the surrounding mortar.

2.1.2 Factors Affecting Deterioration

Factors that affect F-T deterioration include natural environmental factors and aggregate characteristics. Some other factors like fine aggregate content, design of pavement, and traffic also influence the F-T deterioration.

2.1.2.1 Natural environmental factors

Natural environmental factors include moisture and cyclic F-T.

Moisture

The development of D-cracking needs high moisture level in the concrete. F-T deterioration will only occur when concrete or aggregate is exposed to high moisture content environment. The critical degree of saturation of aggregate particle is usually 91.7% [8]. The critical saturation is easily to be achieved in the snow regions of the United States due to the winter season precipitation and temperature [1]. The movement of moisture within concrete is also considered as a factor which influences the development of F-T deterioration. Pore structure characteristics (the ratio of absorption to permeability and others) will influence the degree of saturation of aggregate particles [9]; and this also affects the frost susceptibility of concrete aggregates.

Cyclic F-T

As discussed previously, repeated cycles of F-T have more serious impacts than a single F-T cycle. This process also increases the moisture content in concrete [8]. From the Scheartz's research [10], 5 to more than 10 years of freezing and thawing are required to be sufficient for Dcracking appearance. It also needs to consider the depth of freezing in pavement. D-cracking usually appears near joints rather than other types of deteriorations which usually initiate from the

bottom of the slab [9]. The freezing temperature is an important factor that influences the F-T deterioration [4]. A temperature below the freezing point results in more ice in the aggregate particle and surrounding mortar. The intensity of F-T ing deterioration also is determined by the freezing rate [8]. In the field, freezing rates usually ranged from 0.8 to 0.9 \textdegree C/hour. An increasing freezing rate may result in a reduction of the F-T durability.

2.1.2.2 Aggregate Properties

The F-T durability of aggregate particles is influenced by particle size, pore structure, absorption, and specific gravity, etc.

Particle Size

Coarse aggregate particle size can influence aggregate susceptibility to D-cracking: the smaller the nominal maximum size, the better the F-T durability [10]. It's because the size affects the length of the flow path of the unfreezing water expelled from particles of aggregates. Smaller particle sizes generally have shorter paths which develop less hydraulic pressure. Based on previous research [11, 12], the durability of concrete pavement was improved when the nominal maximum size of crushed limestone aggregates was reduced. The critical size of an aggregate depends on its physical properties such as porosity and permeability. Strength, degree of saturation and freezing rate also affect critical size. Smaller sized aggregates can accommodate a faster freezing rate/higher stress without being fractured. Aggregates that have a large amount of macroscopic voids seem to have a larger critical size, because the permeability of macroscopic voids is higher [13].

Pore Structure

Pore structure is the most important property influencing the D-cracking resistance of coarse aggregates. Pore structure also affects the strength of material, absorption and permeability of aggregate [14]. The characteristics of aggregate pore structure include porosity, permeability, and the pore size distribution. These characteristics affect the concrete durability by controlling aggregate absorption capacity, absorption rate, internal surface area, and bulk volume occupied by solids, the osmotic and hydraulic pressures developed by freezing and thawing, and their effects on the freezing temperature [15, 16, 17, and 18]. Aggregates containing large pores or small pore diameter (for pore sizes larger than1 mm and not smaller than 4.5nm) usually have lower- F-T durability [18]. The previous research [19] indicated that almost all nondurable aggregates have a large proportion of pore diameters between 0.04 and 0.2 mm. Not only large proportion of pore diameters of aggregates will impact durability, but aggregates have very fine pore size distribution (less than 1 um in diameter) will also related to the D-cracking [20]

Absorption

Aggregate absorptive behavior depends on pore structure. Low absorption is usually an indicator for low permeability. These aggregates are generally expected to have good F-T performance. Small-size pores draw water in through capillary action faster than larger pores [8, 21]. In a natural environment, the capillary action retains water in the small-size pores, resulting in higher moisture condition which may lead to a critically saturated condition.

The rate of absorption is related to the aggregate's grain size and the pore size. Hudec [22] indicated that very fine grain aggregates are capable of absorbing about 35 percent of the aggregate's total absorption in nine minutes. While, medium grained aggregates can only absorb slightly over 25 percent of its absorption potential.

11

Aggregates with a fine pore structure usually reach a critical degree of saturation faster than aggregates with a coarse pore structure; even both sources of aggregates have the same porosity. Therefore, the absorption and the rate of absorption need to be considered synthetically when evaluating aggregate F-T durability.

Specific Gravity

Specific Gravity is usually used as the indicator of F-T durability for its correlation to both aggregate porosity and particle strength. Previous researches [18, 23, and 24] indicate that aggregates with a lower coarse aggregate bulk specific gravity are usually more susceptible to the D-cracking of aggregate. However, specific gravity is not usually used as the single indicator to predict aggregate F-T durability. Specific gravity is often used in combination with other pore structure characteristics for evaluating aggregate F-T durability [25, 26].

2.1.2.3 Fine Aggregates and Mineralogy

In Klieger's report [27], source of fine aggregate is believed having few effects on the concrete F-T durability even the fine aggregate is made from nondurable coarse aggregate source. However, in 1985, Dubberke reported [28] that when coarse aggregate is treated with salt, with extra dolomite fines will reduce the concrete F-T durability. Stark indicated in his report [29] that aggregates, which are composed of limestone, dolomite, and chert, are generally susceptible to Dcracking. Aggregates which consist of igneous and metamorphic rocks are generally expected to have better performance with regards to F-T deterioration.

2.2 Test Methods

2.2.1 Tests Summary

Commonly used material tests for F-T durability include four different types: environmental simulation tests, pore structure-related tests, mechanical tests, and chemistry/mineralogy tests.

2.2.1.1 Environmental Simulation Tests

Environmental simulation tests usually include sulfate soundness (AASHTO T104), unconfined aggregate F-T (AASHTO T103), rapid F-T (AASHTO T161), Powers slow cool (ASTM C671), single-cycle slow freeze tests, and Washington hydraulic fracture tests. Each test is briefly introduced in the following paragraphs.

Sulfate Soundness Test

The soundness test is performed by following ASTM C88 or AASTHTO T104. Aggregate is immersed in sodium or magnesium sulfate solution at 70 ° F for 16 to 18 hours and dried at 230°F for over 4 hours. Then, this process is repeated for additional four times to simulate F-T cycles. After the required cycles are completed, the amount of weight losses of aggregate is determined. The testing specifications consider aggregates with a weight loss higher than 12 and 18% to be unacceptable for use in concrete pavement for sodium and magnesium sulfate, respectively.

The procedures of this test are easy to perform without requirement for special equipment. The test is time consuming; and can be completed in approximate 5 days. It directly tests the aggregate durability instead of other aggregate properties which have correlations with the F-T performance. However, aggregate tested using the Sulfate Soundness Test is damaged by the pressure resulted from expansion of salt crystallization, which is a different mechanism involving

different functioning pore sizes from those for the F-T damage. Therefore, the use of the Sulfate Soundness Test is generally limited.

Unconfined Aggregate F-T Tests

AASTHTO T103-Unconfined Aggregate F-T Test

The unconfined aggregate F-T test is performed by following AASTHTO T103. The aggregate sample is soaked in water for 24 hours. Then, the sample is placed in an environment chamber for 50 F-T cycles. Twenty five F-T cycles are needed if the sample is vacuum saturated with water. If the sample is vacuum saturated with ethyl alcohol, the required number of F-T cycles can be decreased to 16 cycles. Each F-T cycle requires 2 to 3 hours to complete depending on the type of saturation. This test provides a good indication of F-T performance. However, it is a time consuming procedure which requires more testing time than the sulfate soundness test. Because the variations in the test condition, the testing results are less reproducible compared to other F-T durability tests [30].

CSA A23.2-24A- Resistance of Unconfined Coarse Aggregate to Freezing and Thawing

The procedure of this test is performed by following CSA A23.2-24A. The test sample is prepared by immersing the aggregate in a sodium solution with 3% sodium chloride for 24 hours. The weight loss of the sample after 5 F-T cycles is determined. The test is relatively more reproducible and has better correlation with field performance in comparison to other F-T simulation tests [31, 32].

Rapid F-T Test

Concrete beam made with interested aggregate are subjected to F-T cycling. According to ASTM C666 the beam is treated at a constantly varying temperature from 40° F to 0° F and from

0°F back to 40°F within a 2 to5-hour period. This F-T cycling condition is more extreme in comparison to most field conditions. The change in the specimen dimension before and after the F-T cycles is determined. A durability factor can be calculated from the length change according to ASTM C215, and used as an indicator of the aggregate F-T durability.

The Rapid F-T Test is the most commonly used procedure to evaluate aggregate F-T durability. It directly measures the F-T durability which provides good indication to the field performance. The disadvantage of this test is that the time required for a complete test is 2 to 5 months.

Powers Slow Cool

Test method is performed by following ASTM C682/671, Powers Slow Cool test measures the changes in specimen dimensions with temperature changes. The sample is conditioned at 35°F. The specimen is cooled by immersing in a water-saturated kerosene bath every two weeks. During the cooling process, the temperature is lowered from 35° F to 15° F at a constant rate in 4 hours. Length changes are measured during cooling.

The test is theoretically sound and reproducible. The correlation between the test result and field performance is good. However, the test condition requirements are relatively complex and extensive. The test may be time consuming; and the duration of testing depends on the time for the aggregate to reach its critical dilation length.

Single-Cycle Slow Freeze

Single-cycle Slow Freeze test also measures the specimen dimension change by temperature. The transverse frequency, mass, and dimension of the specimen are measured before

testing. Then, the specimen is conditioned at $0^{\circ}F$ in a freezing apparatus. Length change measurements are made at 5 to 15-minute intervals for 4 hours.

Completing the testing procedures requires three days. The test is accurate in terms of distinguishing between nondurable aggregates and those with very good F-T performance. However, the test is less effective to quantify the durability of questionable aggregates.

Washington Hydraulic Fracture Test

The Washington Hydraulic Fracture Test uses hydraulic pressure to simulate the stress state due to F-T cycle. The test repeatedly apply and release a pressure, which is assumed to be similar as the stress increase and decrease during freezing and thawing. The aggregate sample is prepared by washing, drying, and a surface treatment in order to make the aggregate surface hydrophobic. The aggregate particles are sieved to exclude particles smaller than 12.5mm. The mass and number of particles of the sample are recorded. Then, a pressure chamber filled with water is used to apply the hydraulic pressure to the aggregate and then suddenly release the pressure.

This test is time consuming which requires eight days to complete. The test simulates the stress state in aggregate during F-T process. However, the results are affected by the pressure release rate.

2.2.1.2 Pore Structure-Related Tests

Aggregate Pore Structure - Related Tests include IPI, absorption/desorption, and tube suction tests.

IPI Test

The IPI Test provides a quantitative measure of aggregate pore structure by measuring the amounts of water absorbed by aggregate in different time periods of testing under a pressure. The test procedure was developed by Myers and Dubberke in 1970s as a quick and simple alternative to the traditional freeze-thaw testing of aggregate for evaluating aggregate's susceptibility with regards to D-cracking [33]. During testing, oven dried aggregate sample is saturated with water under a 35 psi pressure. The amounts of water absorbed by the sample in the first one minute and the following 14 minutes are determined as the primary and secondary load, respectively. The primary load is considered as a result of large pores in aggregate particles; however, the secondary load is mainly influenced by capillary pores, which is closely associated with concrete F-T performance.

The test can be performed in a timely manner with simple procedures. However, the results do not directly indicate the aggregate field performance. The correlation between concrete F-T performance and secondary load was studied by Koubaa and Snyder [34]. The investigation indicated that the correlation between the secondary load and concrete D-cracking resistance was fair, however, weaker in comparison to that between the D-cracking performance and the results of the ASTM C666 Procedure B, the Virginia Polytechnic Institute Test, and the Washington Hydraulic Fracture Test. An investigation conducted by Scholer and Shakoor in 1985 evaluated the reproducibility of the IPI test. The results did not draw definite conclusion on the reproducibility of the IPI test. [35] The reproducibility of the IPI test was proved by a later research conducted by the University of Illinois for the State of Illinois Department of Transportation [36]. Another limitation of the IPI test is that the primary and secondary load measures the amount of pores in two categories, the large pores and the small pores. The distribution of pores based on

pore size cannot be determined. Because of these limitations of the IPI test, it is usually used together with other pore-structure tests to evaluate aggregates.

Absorption

The absorption test procedure is performed by following ASTM C128. The test measures aggregate specific gravity and the amount of water needed for the aggregate to reach a surface saturated dry condition.

The absorption indicates aggregate water retaining ability which is primarily influenced by the porosity of aggregate. It does not describe pore structure or the characteristics of capillary pores.

Tube Suction Test

The Suction Test measures the capillary rise of water in a cylinder filled with compacted aggregate and the dielectric constant at aggregate surface. The aim of the test was to evaluate the moisture susceptibility of granular base materials. The test results are affected by the unbound water in the sample which was found to be correlated with the F-T performance [37]. However, the use of the test method is limited; this method is less effective for samples that are not marginal aggregates. In addition, the procedure tests for the dielectric constant of aggregate instead of directly measures the F-T performance. Meanwhile, the test results are sensitive to testing environment, such as pressure and temperature.

2.2.1.3 Mechanical Tests

Los Angeles Abrasion Test

The Los Angeles (L.A.) Abrasion Test performed by following ASTM C131. The test measures toughness of aggregate by placing the sample in a steel drum along with 6-12 steels

spheres [31]. Each steel sphere weighs approximately 420 grams. During the testing, the drum rotates at a rate of 30 to 33 revolutions per minute for 500 revolutions. The change in the sample weight before and after the testing is determined.

The test is widely accepted nationwide to evaluate the abrasion resistance of aggregate. The result is closely related to aggregate mineral and mechanical properties (such as grain size, porosity, strength, and crushability) [31, 38]. The LA Abrasion Test provides a good indication of aggregate durability for impact forces which occur during handing of aggregate and construction, and under traffic load with a simple procedure. However, the result cannot be directly related to the F-T performance of aggregate. In addition, the test cannot be used for aggregates with a smaller size (usually passing No.12 Sieve).

Aggregate Crushing Value

Aggregate crushing value test is a British test method to evaluate the strength of aggregate for high strength concrete. It can be used to evaluate the aggregate F-T resistance. This Test is widely adopted to qualify the strength of graded aggregate.

2.2.1.4 Chemistry/Mineralogy Tests

This section summarized aggregate chemistry /mineralogy tests, which includes petrographic examination, X-Ray Fluorescence (XRF)/X-Ray Diffraction (XRD), and Thermo Gravimetric Analysis (TGA) tests.

Petrographic Examination of Aggregates for Concrete

Petrographic examinations are performed on concrete aggregates to characterize the aggregate source and quantify the various rock and mineral constituents present to determine if a

particular aggregate source is suitable for use in concrete [31]. Petrographic examinations of concrete aggregates follow the guidelines of ASTM C295, The test can accurately detect the undesired constituents in aggregate, such as clay or dirt; and estimate the relative amount of each constituent.

The results provide good indications for aggregate durability associated with the Alkali-Aggregate Reactivity (AAR). However, the reliability and precision of the test depend on the operator's skill and experience.

Thermo Gravimetric Analysis

The Thermo Gravimetric Analysis Test measures the durability of aggregate by increasing the temperature of an aggregate sample and record the change in weight. During heating, the chemical structure of non-durable aggregates will decompose which can result in a considerable weight loss during heating.

The test provides a quick approach to determine the content of some specific minerals in aggregate, such as calcite & magnesium and faulty carbonates which are susceptible to salt damage. Special equipment and experienced operator are required for this test method which are usually costly.

X-Ray Fluorescence (XRF)/X-Ray Diffraction (XRD)

The XRF is an elemental analysis method which has been widely accepted to determine the mineral composition of various materials by scanning the sample with X-Ray to measure the spacing between atoms.

The test is accurate in detecting particular compositions in aggregate. The results can be used to identify undesirable constituents. The disadvantages of this test are the requirements for special equipment and operation experiences.

2.3 Acceptance Criteria for F-T Durable Aggregate

McLeod compared the acceptance criteria for coarse aggregate in various states in 2012 [5]. As shown in Table 1, five states use performance history of an aggregate source in concrete pavements as an acceptance criterion. Four of the five states, including Indiana, Minnesota, Nebraska and Wisconsin, determine the acceptance of an aggregate using various laboratory tests and geological properties of the sources. However, Iowa relies on the historical performance of aggregate in PCCP.

KDOT aggregate durability specifications [5] including an initial inspection of the aggregate source and three durability tests, including the Durability Factor Test, the KTMR21 Soundness Test, and the Modified Soundness Test. The initial inspection is performed to a quarry by geologists to determine the natural of the deposit and the mineralogy and lithology of the source. The three material tests are conducted periodically during production to assure the quality of the products. These highway agencies also specify that the on-grade concrete can only be constructed using Class 1 or Class 2 aggregate (Table 2).

Table 1: Aggregate Performance History Evaluation Statements from Midwestern DOTs [5]

Table 2: KDOT Specification Limits for Limestone Aggregate in Concrete [5]

Nebraska Department of Road (NDOR) specifies that all classes of concretes, except for PR1 (Repair) and PR3 (Repair), shall have a durability factor higher than 70 and a mass loss less than 5% after 300 F-T cycles when tested in accordance with ASTM C 666-Procedure A [39].

Table 3 shows the test methods and specification limits used by Wisconsin State Department of Transportation (WisDOT) as aggregate durability indicators:

Table 3: WisDOT Specification Limits for Aggregate in Concrete [31]

Test Method	Specification Limits
Los Angeles abrasion (AASHTO T 96)	Weight loss $\leq 50\%$.
Sodium sulfate soundness (AASHTO T 104)	Weight loss $\leq 12\%$.
Unconfined Aggregates F-T (AASHTO T 103)	Weight loss \leq 18%.
Lightweight Pieces in Aggregate (AASHTO T 113 and CMM 13.22)	Shale, coal 1%, clay lumps 0.3%, soft fragments 5.0%, thin/elongated piece 15%, fines 1.5% , chert 5%

Minnesota Department of Transportation (MnDOT) adopted the following acceptance criteria for concrete pavement coarse aggregate [4]:

- Class B aggregate (crushed quarry or mine trap rock other than Class A: i.e., carbonates, rhyolite, schist) must have a maximum absorption of 1.75%
- Class C aggregate (natural or party crushed natural gravel) must have a maximum carbonate content of 30% (by weight)

These criteria have been implemented by the state highway agencies to assure the performance of aggregate used for concrete with regards to F-T deterioration. However, they are very restrictive that it possibly rejects an aggregate with acceptable field performance.

2.4 Summary of Literature Review

This chapter of the thesis provides a comprehensive review to the mechanism of aggregate F-T deterioration and test methods adopted to evaluate the aggregate F-T performance. Throughout the literature review, it was found that the D-cracking is primarily caused by the F-T damage of aggregate. Traditional test methods simulate the F-T cycles in a controlled environment. However, these tests are usually time consuming and costly. The resistance of F-T damage for an aggregate is influenced by aggregate pore structures, which provide an alternative approach to evaluate the F-T durability of aggregate. Various test methods have been developed to directly or indirectly measure the pore structures. The advantages and disadvantages of these tests were discussed in this chapter. The acceptance criteria for aggregate F-T durability adopted by various state highway agencies were also investigated. Literature shows that no single existing method is good enough to be used to accept or reject concrete aggregate properly. Iowa DOT approach indicates that combination of physical and chemistry/mineralogy test methods may provide a better evaluation for aggregates.

CHAPTER 3. EXPERIMENTAL WORK

The following tests were performed in this study: 1, the IPI Test; 2, the Specific Gravity and Absorption Test, and 3, the Rate of Absorption/Desorption Test.

The durability of coarse aggregate used in concrete is related to the IPI Test results. The IPI Test was conducted for aggregates from various sources as well as the carbonate and noncarbonated particles from each source. The main goal of the IPI Test was to determine the differences between the characteristics of the pores in carbonate and non-carbonate aggregate.

The specific gravity and absorption were evaluated based on ASTM C127 of standard test method for density, relative density (specific gravity), and absorption of coarse aggregate. Results from this test was found to support the IPI Test, and provided detailed information for the pore characteristics.

To evaluate the water absorbability which is related to the characteristics (such as size and distribution) of capillary pores in the aggregate, the Rate of Absorption/Desorption Test is necessary. The pore structure was evaluated by measuring the amount of water absorbed by aggregate and drawn out from the aggregate in a SSD condition. The rate of absorption and desorption at incremental time periods were tested.

3.1 Materials

Aggregates from 15 sources (Table 4) in Minnesota were evaluated in this study. Among the 15 aggregates, 3 of them have a carbonate content of 100%; and the carbonate content of the other aggregates are below 50%.

Table 4: Aggregate Sources and Source Numbers

* 100% carbonate aggregate.

Figure 3 shows the location of the sources of the coarse aggregates.

Figure 3: Location of the Sources of the Coarse Aggregates

3.2 Aggregate Preparation

The IPI Test samples were prepared using the following steps:

- 1. Sieve all aggregates according to ASTM C136. Retain the aggregates passing the $\frac{3}{4}$ " sieve and retained on the $\frac{1}{2}$ " sieve for the experiments;
- 2. Wash the aggregates in order to remove the dust and impurities;
- 3. Dry the aggregates at 105° C for 24 hours; and
- 4. Sort the aggregates into carbonate and noncarbonated based on the carbonate content of the aggregates.

3.2.1 Sorting of Aggregate

Aggregate from each source was sorted as carbonate particles and non-carbonate particles before the F-T durability tests were performed. The sorting process includes two steps: 1. sort the aggregate particles by aesthetic and hardness differences; and 2. sort the aggregate particles using 10% hydrochloric acid.

Carbonate aggregate usually has a light-colored surface and relatively rough surface texture in comparison to noncarbonated aggregate. Figure 5 shows the differences in the appearance of carbonate and non-carbonate particles. The hardness of carbonate aggregate is usually lower than that of non-carbonate aggregate. During sorting, a steel knife was used to scrape the surface of the aggregate particles. If some fine powders can be scraped off from the particle surface, this would be an indication of carbonate aggregate. The steel knife used for sorting is shown in figure 4.

Figure 4: Steel knife Used to Scrape the Surface of Aggregate

(a) Carbonate (b) In Between (c) Non-Carbonate Figure 5: divided Carbonate, In Between and Non-Carbonate aggregates

Some aggregate particles have light-colored surface and high hardness. The visual observation and hardness test may not definitely categorize these aggregate particles. Such aggregates were marked as "in-between" particles (Figure 5 (b)); and were tested for their chemical composition using a chemical sorting method.

Figure 6: Carbonate Aggregate Surface React with Acid Solution

Carbonate aggregate reacts violently with acid solution. Therefore, a solution containing 10% hydrochloric acid was used to separate the carbonate and non-carbonate aggregates. Aggregates which rapidly released large amount of gas when tested with the acid were classified as carbonate aggregate. Figure 6 shows a carbonate aggregate reacts with the acid solution. The bubbles in the figure indicate the violent reaction process.

Based on the sorting measurement, the raw aggregates can be divided into carbonate and non-carbonate aggregate. The bulk aggregates were kept for future tests. The carbonate contents of all of the aggregate sources can be obtained through the process. The results of carbonate content of each source can be used for analysis, and to determine whether the carbonate content of certain source of aggregate influences the aggregate F-T durability.

3.3 Test Methods

The IPI Test, the Specific Gravity and Absorption Test, and the Rate of Absorption/Desorption Test were performed for the carbonate, non-carbonate, and bulk aggregate particles from each source. Table 5 summarizes the standards of test methods employed in this research; and briefly explains the significance of each work. The devices used for sieving aggregate, the IPI Test, the Specific Gravity and Absorption, and the heating oven are shown in Figure 7, 8, 9 and 10, respectively. Figure 8illustrats the measurements of the IPI Test. It should be noticed that the same oven were used in the three tests. All of the samples prepared for the three tests were dried with oven before testing at 105°C.

Figure 7: The Aggregate Sieving Device

(a) IPI Device and Control Panel

32

(b) Illustration of PPI Measurement (c) Illustration of SPI Measurement

Figure 8: IPI Apparatus and Illustration of Measurements

Figure 8 shows the device used for the IPI Test. The test equipment consists of two persistent parts: the primary index reading and the secondary index reading. The secondary pore index reading is procedural after the primary index reading. The detailed procedures are discussed in the later sections.

Figure 9: Specific Gravity and Absorption Device

Figure 10: Oven Used in the Tests

3.3.1 IPI Test

The IPI Test was performed by following Iowa 219-D. This method evaluates aggregate pore system. This test determines the amount of the large and capillary pores in coarse aggregate by measuring the amounts of water absorbed by the aggregate at different times. During the testing, the aggregate sample is placed in a special container filled with water. A pressure of 35 psi is applied to force water to fill the permeable voids in the aggregate. The time for water to fill a pore is related to the pore size. The amounts of water absorbed by the sample in a short and long time periods can be used to evaluate the amounts of large and small pores in the aggregate, respectively. The amount of water absorbed in the first 60 seconds provides an indication of the large pores, which allow water to quickly dissipate during freezing, and is the "primary load". The water absorbed in the next 14 minutes reflects the small pores, which do not allow water to dissipate easily or provide enough space to accommodate the volume change during freezing, and is termed as the "secondary load". [33].The total test duration is 30 minutes. The rest time after obtaining

the secondary load is for the safety check, the safety check value will be obtained on the third tube at the 30 minutes time from test start.

Test the aggregate for IPI to obtain PPI, which is considered to be related to the amount of large pores in the aggregate, and SPI, which is considered to be related to the amount of small pores in the aggregate. A high SPI value implies poor F-T resistance of the aggregate. Equations in Table 6 show the calculations of IPI from device reading.

To determine the pot expansion, run the test without adding an aggregate sample. After the primary load indicator light switches off, the primary load value was recorded as the pot expansion value. During the testing, the range of pot expansion were obtained from 13 to15 ml in most of the tests. In order to assure the consistency of the results, most pore index calculated with pot expansion were 14 ml.

Table 6: IPI Calculation Equations

Primary pore index (PPI) = (1 minute reading - pot expansion) x (9000/sample weight)

Secondary pore index $(SPI) = (14 \text{ minute reading})x(9000/\text{sample weight})$

3.3.2 Specific Gravity and Absorption

The specific gravity and absorption of the aggregate were examined according to ASTM C127. The purpose of this test is to determine the relationship between the estimated water content (SSD, absorption) and actual water content (moisture contents).

A sample of aggregate was immersed in water for 24 (± 4) hours. Then, the sample was removed from the water and dried with a towel to achieve a SSD condition. The sample in SSD condition is oven-dried to determine its dry weight. The density, specific gravity and volume of voids can be calculated.

3.3.3 Rate of Absorption/Desorption Test

The results from the IPI Test provide information on the pore characteristics that related to F-T durability performance of the aggregate. However, the pore structure of aggregate cannot be evaluated by the IPI Test. Other pore structure related tests need to be performed in order to accurately determine the F-T durability of the aggregate. A Rate of Absorption/Desorption Test was developed for this study as a supplementary testing procedure for aggregate pore structure. The rate of absorption (sorptivity) by coarse aggregates was measured by measuring the increase in the sample weight during absorption. During desorption, the rate of dehydration was measured by the decrease in the sample weight.

Figure 11 shows the absorption and desorption process. During the absorption, the sample was simply soaked into water. Before measuring the sample weight, the excessive moisture on the aggregate surface was dried by a towel.

(a) Aggregates Soaked in the Water (b) Sample Dried by Towel and Weighted Figure 11: Aggregates Treated Illustration in Rate of Absorption/Desorption Test Process

The Rate of Absorption Test is used to measure the amount of water that is absorbed during the drying process within each time period. The aggregate is weighed every 15 minutes to 10 hours according to the schedule showed in Table 7.

Schedule for Sample Weight Measurement				
15 minutes	6 hours			
30 minutes	8 hours			
1 hour	18 hours			
2 hours	24 hours			
4 hours				

Table 7: Time Schedule of Testing of Rate of Absorption Test

The Rate of Desorption Test is used to measure the amount of water that is drawn out from saturated surface dry (SSD) aggregate within each time period. The aggregate is weighed every 20 minutes to 10 hours according to the schedule showed in Table 8.

Schedule for Sample Weight Measurement						
20 minutes	2 hour 40 minutes					
40 minutes	3 hours					
1 hour	4 hours					
1 hour 20 minutes	6 hours					
1 hour 40 minutes	8 hours					
2 hours	18 hours					
2 hour 20 minutes	24 hours					

Table 8: Time Schedule of Testing of Rate of Desorption Test

CHAPTER 4. RESULTS, ANALYSIS, AND DISCUSSION

As mentioned in the previous part, IPI Test, specific gravity and absorption and rate of absorption/desorption were performed during the study. Figure 12 below summarizes some key test results of the different sources of aggregates tested in the experiment. Detail information is presented and discussed in the chapter.

Figure 12: Location of the Sources of the Coarse Aggregates with Tests Results Summary

4.1 Carbonate Content, Absorption and Specific Gravity

Table 9 shows the summary of key results from the carbonate content calculation and Specific Gravity and Absorption Test. The detail discussion will provided in the below sections.

Aggregate ID	Type	Carb. content, %	Specific Gravity	Absorption, %	Aggregate ID	Type	Carb. content, %	Specific Gravity	Absorption, %
	Carb.		2.55	2.66		Carb.		2.56	2.54
19109	Non-Carb.	13.50%	2.67	1.04	67001	Non-Carb.	25.30%	2.63	1.38
	Bulk		2.66	1.30		Bulk		2.61	1.64
	Carb.		2.61	2.28		Carb.		2.56	2.49
82001	Non-Carb.	14.20%	2.61	0.89	86001	Non-Carb.	28.52%	2.68	1.00
	Bulk		2.68	1.14		Bulk		2.65	1.37
	Carb.		2.56	2.70		Carb.		2.55	2.35
19001	Non-Carb.	20.00%	2.69	1.04	34002	Non-Carb.	40.71%	2.65	0.74
	Bulk		2.65	1.30		Bulk		2.66	1.47
	Carb.		2.59	2.07		Carb.		2.63	1.91
70008	Non-Carb.	21.30%	2.70	0.87	56192	Non-Carb.	44.20%	2.70	0.51
	Bulk		2.67	1.04		Bulk		2.68	1.25
	Carb.		2.58	1.87		Carb.		2.73	1.02
03081	Non-Carb.	22.50%	2.66	0.45	79091	Non-Carb.	100%		
	Bulk		2.63	0.65		Bulk		\sim	\overline{a}
	Carb.		2.57	1.85		Carb.		2.54	3.2
56003	Non-Carb.	22.70%	2.64	0.72	70006	Non-Carb.	100%	\blacksquare	\blacksquare
	Bulk		2.65	0.91		Bulk			
	Carb.		2.64	1.89		Carb.		2.7	1.47
03090	Non-Carb.	23.30%	2.73	0.52	82002	Non-Carb.	100%	\blacksquare	$\overline{}$
	Bulk		2.72	0.82		Bulk		\blacksquare	\blacksquare
	Carb.		2.61	1.84					
14074	Non-Carb.	24.70%	2.67	0.54					
	Bulk		2.65	0.80					

Table 9: Summary of Aggregate Carbonate Content, Specific Gravity and Absorption

4.1.1 Test Results Summary

Figure 13 shows the summary of carbonate content of each source used in the study. From the figure, there indicates three of them were 100% carbonate aggregate. And most of rest sources aggregates have carbonate content below 40%. Aggregate ID 34002 and 56192 have more than 40% of carbonate content. The relationship between carbonate content and other test results will be discussed in the later sections.

Figure 13: Carbonate Content Summary of Each Source

The specific gravity of an aggregate is a property that is related to the mineralogy, grain size, and pore size. With similar mineralogical aggregates, a smaller specific gravity suggests that the aggregate has a higher pore volume compared to an aggregate of higher specific gravity.

Aggregates with a low specific gravity were trend to have higher absorptions than aggregates with high specific gravity [22].

Figure 14: Specific Gravity Results Summary

Figure 14 shows the specific gravity summary of all the aggregates in three classifications of aggregate. From the figure, it indicated that carbonate aggregates usually have lower specific gravity which related to higher pore volume than non-carbonate aggregates and bulk aggregates. Source ID 70006 have the lowest specific gravity which means it will have high ability of absorption.

Figure 15: Absorption Results Summary

Figure 15 summarizes the absorption value from the specific gravity and absorption, with the order of carbonate content of the aggregates is increasing from left to right. From the results there can be observed that absorption value have the opposite trend with the specific gravity value; there have obvious difference among three different classifications of aggregates. The absorption value of carbonate aggregate is the highest in three classifications comparison of each source. And non-carbonate aggregate have the lowest absorption value. Source ID 70006 shows the especially high value than other sources, and this will also been found in the SPI results summary.

The Specific Gravity and Absorption Test can reflect the amount of pores in aggregate to certain degree. Figure 16 shows that carbonate aggregate has a higher absorption in comparison to

non-carbonate aggregate. This suggests that the carbonate aggregate has more pores in comparison to non-carbonate aggregate.

Figure 16: Average Absorption Comparison for Aggregates with Various Carbonate Contents

The Minnesota aggregate acceptance specifications require the absorption of crushed carbonate rock less than 1.75%; and carbonate aggregate content for natural aggregate is less than 30%. The specifications are primarily established for minimizing the aggregates that are susceptible to F-T damages.

Figure 12 shows the important properties of alternative resources distribution based on their location. From the map, each source's SPI, carbonate content and absorption can be located easily. For those alternative resources cated very close, some of them have the similar properties like source ID 03090 and ID 03081, but there also have some have the different properties like source ID 70006 and ID 70008. So the location of resource may be can't considered as the indicator to influence the durability performance of aggregates.

Figure 17: Carbonate Content Summary of Each Bulk Aggregate.

Figure 17 shows the carbonate content summary of the alternative resources. From figure, only two resources (ID 34002, 56192) are not meeting the Minnesota criteria. Aggregates have 100% carbonate content using a different acceptable criteria of absorption shown in the Figure 18 for the property of the aggregate. From the observation, there is only one resource of ID 70006 (crushed or manufactured carbonate aggregate) doesn't meet the requirement.

Figure 18: Absorption Results of Each Bulk Aggregate

الاستشارات **(2**

4.2 IPI Test

Table 10 shows all IPI Test results. 3-5 specimens have been tested for each source. Bulk aggregate only test once.

Table 10: IPI Test Results Summary

4.2.1 IPI Test Results Summary

Figure 19 summarizes the PPI values from the IPI Test, with the order of carbonate content of the aggregates is increasing from left to right. From the results it can be observed that PPI values are obvious different for the aggregates with various carbonate contents(carbonate, non-carbonate, and bulk). The PPI value of carbonate aggregate is the higher than that of the non-carbonate and bulk aggregates. Non-carbonate aggregates have the lowest PPI value. The similar trend was also observed in the SPI results as shown in Figure 20.

Figure 19: PPI Results Summary

Figure 20: SPI Results Summary

As shown in Figure 20, similar to what have been observed for the PPI values, the SPI values of carbonate aggregates except aggregate with 100% carbonate content are higher than the SPI values of non-carbonate and bulk aggregate samples for each source. Aggregate ID: 70006 has particularly high SPI value; while the results are not statistically different from other sources. This indicates there have more relatively small pores in the pore structure of this kind of aggregate.

(a) Average PPI

(b) Average SPI

(c) Average Total Pore Index

Figure 21: Average IPI Results of Three Classifications of Aggregates of all 15 sources

Figure 21 shows the comparison of PPI/SPI values of different chemical classifications of aggregates. As mentioned previously, the PPI is correlated to the amount of relatively large pores in the aggregates. The average PPI of carbonate aggregate is 108, which is 1.8 times as that of non-carbonate aggregates. The average SPI of carbonate aggregates is 30, which is 2.5 times as that of non-carbonate aggregates. The total value of average PPI plus SPI of carbonate aggregates is 1.9 times as that of non-carbonate aggregates. This indicates that overall, carbonate aggregates has much higher total pore volume (PPI +SPI) than non-carbonate aggregates. Especially, the volume of small voids (F-T related) is higher.

SPI calculated from late observation of IPI Test was considered associated with the amount of relatively small pores in the aggregate. Figure 21 shows similar trend among three classifications of aggregates SPI value as the PPI value.

Figure 22: PPI versus SPI Plot

The relationship of PPI and SPI is shown in Figure 22. A strong linear correlation with an R Square value of 0.66 was observed between PPI and SPI for all aggregates. However, R Square values of carbonate and non-carbonate individually were 0.56 and 0.10, respectively. The observation indicated non-carbonate had a stronger relationship of PPI and SPI than carbonate aggregates. The resource ID 70006 is especially jarring because it has both high PPI and SPI value. The relationship indicates that higher SPI values are usually obtained when aggregate have a higher PPI value. Such correlation can be used to estimate the amount of smaller pores (SPI), which is related to F-T resistance, in an aggregate by testing for the larger pores (PPI) which is relatively easier to measure.

4.2.2 Statistical Analysis of IPI Test Results

Statistical analyses are conducted for comparing the PPI and SPI values for different aggregate chemical classifications and correlation between the IPI Test results and aggregate carbonate content. Statistical software, JMP, was used to assist the analyses.

The PPI and SPI values were first analyzed considering the aggregates with various carbonate content (carbonate, non-carbonate, bulk) as the only influencing factor. Table 11 summarizes the analysis of variance (ANOVA) results. A p-value of less than 0.0001 was found for the influence of aggregate chemical classification factor on the PPI values which indicates that the chemical classification is a significant factor of PPI. Table 12 shows the Student's t-test results of the comparisons for different aggregate classifications. The t-tests show that the PPI values for carbonate, non-carbonate, and bulk aggregates are all significantly different from each other.

Table 11: Analysis of Variance between PPI and Aggregate Classifications

Source	DF	Sum of Squares	Mean Square F Ratio		Prob > F
Classification of Aggregates		56735.86	28367.95	112.45	< 0001
Error	112	28255.31	252.33		
C. Total	14	84991.17		-	

Table 12: Student's T-Test for Aggregate with Various Carbonate Content According to PPI

* Levels connected by the same letter indicate the difference is not statistically significant.

Same analyses are performed for the SPI results. A summary of the SPI statistical analysis is presented in Table 13. The analysis indicates that aggregates with various carbonate content

(carbonate, non-carbonate, bulk) is also a significant factor of the SPI values; and the SPI values of the three aggregate classifications are all significantly different from each other. Table 14 shows the Student's t-test results of the comparisons for different aggregate classifications according to SPI. The t-tests show that the SPI values for carbonate, non-carbonate, and bulk aggregates are all significantly different from each other.

Table 13: Analysis of Variance between SPI and Aggregate with Various Carbonate Content

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Classification of Aggregates		8019.73	4009.86	83.00	< .0001
Error	112	5410.59	48.31		
C. Total	114	13430.32			

Table 14: Student's T-Test for Aggregate with Various Carbonate Content According to SPI

* Levels connected by the same letter indicate the difference is not statistically significant.

In order to correlate the IPI Test results with aggregate carbonate content, linear models were established for PPI and SPI, respectively. The model has an r square value of 0.18. The ANOVA (Table 15) of the linear model has a p-value of 0.11 which is greater than 0.05 associated with 95% confidence level. This implies that the correlation between carbonate content and PPI values is not significantly different from zero. The regression plot is presented in Figure 23.

Table 15: Variance Analysis of PPI and Carbonate Content

Source	DF	Sum of Squares	Mean Square	∣ F Ratio	Prob > F
carbonate content		904.90	904.90	2.88	0.11
Error	13	4085.66	314.28	-	-
C.Total	14	4990.56		-	-

Figure 23: Regression Plot of PPI and Carbonate Content

The linear model for SPI values exhibits a higher R-Square values and suggests a stronger correlation between carbonate content and SPI value. The expression of the linear model for SPI is shown in Equation 1. The p-value of the model is 0.03, which indicates a statistically significant correlation between the two variables (Table 16). The regression plot is shown in Figure 24. The residual plot (Figure 24 (b)) shows that the residual errors of the model have a certain pattern as the carbonate content increases. The residue errors are more concentrated for smaller carbonate contents. This suggests that a higher order regression model may be needed.

 $SPI = 14.05 + 16.17 \times$ Carbonate Content Equation 1

Source	DF	Sum of Squares	Mean Square F Ratio Prob > F		
carbonate content		376.54	376.54	5.98	0.03
Error	13	818.62	62.97		
C.Total	14	1195.17		0.03	

Table 16: Variance Analysis of SPI and Carbonate Content

(a) Actual by Predicted Plot (b) Residual by Predicted Plot

Figure 24: Regression Plots of SPI and Carbonate Content

The previous analyses did not consider the influences of different aggregate sources for the convenience of statistical analysis. Some of the sources do not have aggregates that are noncarbonate. Including the effects of source for these aggregates can result in loss of degree of freedom. The effects of source are analyzed for the sources that contain both carbonate and noncarbonate aggregates. And, 100% carbonate aggregates were not included in the analysis. The factors considered in ANOVA include aggregate source, aggregates with various carbonate content (carbonate, non-carbonate, bulk), and the confounding effects of classification and source. The ANOVA tables are shown in Table 17 and Table 18 for PPI and SPI, respectively. The results indicate that all three factors are statistically significant factors of PPI and SPI. However, the F ratio for aggregate classification is much higher than that of aggregate source. Thus, the IPI Test result of an aggregate is predominantly influenced by aggregate carbonate content.

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Source	11	13924.01	1265.82	122.10	${<}0001$
Classification of Aggregates		52538.28	52538.28	5067.73	< .0001
Classification of Aggregates*Source	11	2072.17	188.38	18.17	${<}0001$
Error	64	663.50	10.37		
C. Total	87	71187.62			

Table 17: Analysis of Variance between PPI and Source or Source-Classification Associated

Table 18: Analysis of Variance between SPI and Source or Source-Classification Associated

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Source	11	330.47	30.04	16.28	< .0001
Classification of Aggregates		6316.29	6316.29	3423.40	< .0001
Classification of Aggregates*Source	-11	1167.49	106.14	57.53	< .0001
Error	64	118.08	1.845		
C. Total	87	8003.69			

4.2.3 IPI and Absorption Correlation

Absorption ability indicates how much water can be absorbed for certain amount of aggregate. Since the IPI Test measures the amount of pores in different sizes by measuring the amount of water, the results of absorption and IPI Tests are expected to be highly correlated. Figure 25 (a) and (b) show the relationship between PPI and SPI with absorption, respectively. The linear correlations of PPI and SPI with absorption have r square values of 0.93 and 0.77, respectively. The higher r square value for PPI and absorption correlation may because that water is primarily absorbed by larger size pores.

(a) Absorption Value versus PPI Plot

(b) Absorption Value versus SPI Plot

Figure 25: Relationship between Absorption and IPI Test Results

Figure 25 (c) shows the relationship between absorption and PPI plus SPI. According to the definitions of IPI value, the sum of PPI and SPI indicate the total amount of water absorbed by aggregate. The testing results indicated that absorption is well correlated to the primary load (i.e., PPI) and the total load (i.e., PPI+SPI). The correlation is relatively poor for the secondary load (i.e., SPI) as indicated by Figure 25 in comparison to that for the primary load. The r-square values for the correlations of absorption with total load for non-carbonate and carbonate aggregates are 0.87 and 0.31, respectively. The primary load primarily depends on the large pores in aggregate. Therefore, the absorption of carbonate aggregate is more influenced by the large pores in aggregate in comparison to the small pores. The lower coefficient of correlation for carbonate aggregate may suggest other unidentified factors are influencing SPI and/or absorption measurements. The pore distribution characteristics (e.g. tortuosity) and test procedures may contribute to such differences in the coefficients of correlation. For example, aggregate from one source might contain a certain amount of pores that water did not reach during IPI Test. Longer testing time may allow more water get into smaller pores. To have a better analysis, future studies conducted to prove the relationship between PPI and SPI with pore size distribution are needed.

The residuals of the linear regression for carbonate aggregate are shown in Figure 26. A pattern can be observed for the residuals in the figure. The residuals tend to be greater for smaller and greater SPI values and smaller for SPI values close to 35. This pattern suggests that additional variables may be needed to better describe the correlation between SPI and absorption for carbonate aggregates.

Figure 26: Residual by Predicted Plot of SPI and Absorption (Carbonate Aggregates)

4.2.4 Iowa DOT Specification

The Iowa DOT uses the Salt Susceptibility Test and the IPI Test to evaluate the F-T durability of aggregate (Iowa DOT IM409). Based on the tests results, aggregate can be classified into three categories according to Table 19. The expected performance of each class of aggregate is summarized in the following paragraphs:

- Class 2: aggregates will produce no deterioration of pavements of the non-Interstate segments of the road system after 15 years and only minimal deterioration in pavements after 20 years of age.
- Class 3: aggregates will produce no deterioration of pavements of non-Interstate segments of the road system after 20 years of age and less than 5% deterioration of the joints after 25 years.
- Class 3i: durability aggregates will produce no deterioration of the interstate road system after 30 years of service and less than 5% deterioration of the joints after 35 years.

Durability Class	Quality	Test limits	Test Method
Class 2	Salt susceptibility quality	Max. 4.5	Iowa 223
	SPI	Max. 30	Iowa 219-IPI Test
Class 3	Salt susceptibility quality	Max. 1.5	Iowa 223
	SPI	Max. 25	Iowa 219-IPI Test
	Salt susceptibility quality	Max. 1.0	Iowa 223
Class 3i	SPI	Max. 20	Iowa 219-IPI Test

Table 19: Iowa Durability Approvals by Chemical & Physical Testing (Iowa DOT IM409)

The aggregates used in this investigation were evaluated using the Iowa DOT method. The results (Figure 27) show that the aggregate from Source 70006 is unacceptable for use in concrete due to its poor performance with regards to F-T damage; however, the aggregates from the other sources are in the Class 3i and Class 3, which suggest these aggregates have satisfactory F-T resistance. However, aggregates from Sources 34002 and 56192, which meet the Iowa DOT criteria for F-T durability, failed to meet the Minnesota DOT specifications for aggregate F-T resistance because of their high carbonate content. Carbonate content of aggregate does not directly related to aggregate pore structures and the F-T durability. Therefore, the current Minnesota DOT specifications are suggested to change for using more direct approach to measure the aggregate pore structure.

Figure 27: SPI of Bulk Aggregate

4.3 Rate of absorption/desorption test

4.3.1 Rate of Absorption/Desorption Test Summary

Figure 28 shows the time-dependent absorption behavior of three example sources. The rates of absorption of all aggregates during the first 15 minutes after the aggregates are soaked into water were very high. While, the rates of absorption after soaking for 30 minutes were very small. This observation indicated the transition between the above two stages occurs in a very short time.

(a) Carbonate Aggregate Illustration

(b) Non-Carbonate Aggregate Illustration

Figure 28: Time-Dependent Absorption of Aggregates

Figure 29 shows the time-dependent desorption behavior of the three example sources. The rates of desorption of all aggregates during the first 140-240 minutes range after the aggregates were soaked were very high. However, the rates of absorption of all aggregates after 240 minutes

soaking were very low. There are clear transitions between the above two stages for all aggregate tested.

(a) Carbonate Aggregate Illustration

(b) Non-Carbonate Aggregate Illustration

Figure 29: Time-Dependent Desorption of Aggregates

All absorption/desorption time-dependent curves listed in the Appendix.

4.3.2 Absorption and Desorption Analysis

For the purpose of analysis, the amount of water absorbed in the certain time period been observed as the indicator of how fast water been absorbed.

Because the absorption in the first time period was considerably high, the amount of absorbed water in the first time period (0 to 15 minutes) is potentially related to intermediate size of pores in the aggregate. A short-term and a relatively longer-term test period, 15 to 30 minutes and 30 minutes to 24 hours, was recorded after the first test period was completed. In these two periods, the amounts of water absorbed were smaller than it in the first period. Figure 30 and table 20 below show the summary of absorption/desorption in the certain time period.

Figure 30: Absorption/Desorption Results Summary at 24 Hours

Table 20: Water Absorbed Value Summary

المنسارات

Table 20 summarizes the absorption/desorption in certain time period of all sources of aggregates.

The results in Figure 30 show that the amount of water absorbed by carbonate aggregates within 24 hours test period was noticeably higher compared to the water absorbed by noncarbonate aggregates. This suggests that carbonate aggregate has larger absorption/desorption than non-carbonate aggregate which agrees with the conclusions drawn from the IPI Test and the volumetric tests. It was also noticed that a greater disagreement between water absorption and desorption in aggregates from Source 19109, 82001, and 82002, than that in aggregates from the other sources. Such disagreement was more considerable for non-carbonate aggregates than for carbonate aggregates. These three sources are geographically close and share similar terrain and geological features which are different from the other sources with exceptions of Source19001 and 70008. The absorption was measured on change of weight from dry condition to SSD condition. The SSD aggregate was achieved by using a towel dry surface moisture; while, the desorption was measured using an oven dry process which is more precise and accurate. The water retention on aggregate surface can considerably influence the absorption measurements. The differences in the geological features may result in differences in lithology, mineralogy, and pore structures which may lead to different surface water retaining behaviors and the observed large differences between water absorption and desorption for aggregates from these sources.

In order to examine the percentage of absorption and desorption in each test period, figure 31 is designed to show the detailed absorption and desorption process of all sources of aggregates. Carbonate and non-carbonate aggregates of each source have been compared.

(b) Water Desorbed in Certain Time Period, ∆t

Figure 31: Water Absorbed/Desorbed in Certain Time Period, ∆t

Figure 31 shows the percent absorption and desorption occurred in each test period. The majority of absorption occurred in the first 15 minutes of testing. The absorption occurred after 15 minutes were only 11% to 21% of the water absorbed during the entire testing period. However, the rate of desorption was more constant compared with the rate of absorption. The water discharged by carbonate aggregates in 15 minutes, 15 minutes to 30 minutes, and 30 minutes to 24 hours test period was 1.5 to 15.2%, 6.3 to 22%, and 65.7 to 88.9% of the total water desorption, respectively. The results exhibited a strong correlation between the percent of water desorption in each test period and the duration of the test period. For non-carbonate aggregate, the desorption was much faster in the first 15 minutes compared to the carbonate aggregate. Approximately 4.4 to 37.3% of the total water desorption occurred in the first 15 minutes for non-carbonate aggregates.

(a) Water Absorbed within 0-15 minutess vs. PPI

(b) Water Absorbed within 0-30 minutes vs. PPI

Figure 32: Relationship between Water Absorbed and PPI

Figure 32 exhibits strong correlation between PPI and the amount of absorbed water. The PPI increases as the absorbed water increases for both carbonate and non-carbonate aggregate. The absorption testing results in the three test periods are showing the same increasing trend for PPI and similar levels of correlation. Figure 33 shown the relationship of water absorbed and SPI, which both considered can reflect the intermediate and small pores in the aggregate. The SPI also exhibited a positive correlation between the pore test results and the absorption test results. However, the level of correlation between SPI and absorbed water is considerably weaker than that between PPI and absorbed water. With r square values equal to 0.71 in figure (c), an acceptable linear relationship can be indicated. However, because of lower r square, the relationship between carbonate or non-carbonate aggregates was considerably weaker.

The IPI Test can accurately reflect the absorption ability in the early period when large pores acting the leading role. Some micro tests of pore structure may be need in the future study.

(a) Water Absorbed within 15-30 minutes. Vs. SPI

(b) Water Absorbed within 15minutes-24 hours. Vs. SPI

(c) Water Absorbed within $0 - 24$ hours vs. SPI

Figure 33: Relationship between Water Absorbed and SPI

CHAPTER 5. CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The assessment of the aggregate durability in this study has focused on the following aspects: (1) Pore index performance as an indicator of coarse aggregate durability; (2) Absorption and carbonate content relations to pore index; and (3) Relationship between pore structure and pore indexes. The following conclusions are drawn based on the experimental work and analyses conducted:

- 1. IPI Test is a simple, quick and indirect test for aggregate pore structure evaluation. It measures the amount of water enters pores under a pressure in a given time (15 minutes), not directly measuring pore diameter. The PPI, resulting from the amount of water enter pores (believed as large pores) during the first 1 minute of the test, and SPI, resulting from the amount of water enter pores (believed as small pores) during the rest 14 minutes of the test, are easily to measure.
- 2. Carbonate aggregates generally have higher absorption, lower specific gravity, higher PPI, and much higher SPI (for a given absorption) than non-carbonate aggregates.
- 3. The PPI and PPI+SPI measurements are closely related to absorption of aggregates (for carbonate, non-carbonate, and bulk aggregates).
- 4. The correlations of SPI with aggregate absorption are very weak, especially for carbonate aggregate. This may be attributed by other unidentified factors (such as aggregate pore tortuosity and the IPI Test pressure and time).

- 5. A pattern can be observed for the residues of the linear regression model for SPI-absorption correlation. This pattern suggests that a higher order regression may be needed to better describe the correlation between SPI and absorption for carbonate aggregate.
- 6. PPI contributes mainly to the ability of absorption. A correlation between PPI and water absorption/desorbed in the first 15 minutes was found which indicates that the majority of the absorption occurs in the early absorption/desorption period and by the larger pores in the aggregate structure; and the later absorption/desorption may be related to small pores in aggregate. As a result, SPI measurements might be sensitive to the testing pressure and time.
- 7. IPI Test can be considered as a simple, rapid screening test for aggregate field performance if the results can be used in combination with other durability test results. However, understanding the aggregate chemistry and mineralogy are important for accurate field performance prediction.

5.2 Recommendations for Future Study

The experimental work of this study has provided a comprehensive evaluation of IPI Test as well as the information relating the PPI and SPI to volumetric properties of aggregates. The followings recommendations are proposed for implementation of the research results and for a further study:

- 1. Higher order regression model are recommended for the future study as to explain the relationship between IPI Test results and carbonate content/absorption.
- 2. Multiple observations of the rate of absorption and desorption in the first 15 minutes of testing are recommended to record more accurate change of weight, which can be used to

examine the correlation between pore index and characteristics (such as size and distribution) of capillary pores.

- 3. Pore size distributions in aggregate shall be tested using other methods. (e.g. Mercury Intrusion Porosimetry (MIP)); and the results shall be compared with IPI values.
- 4. The relationship between IPI and F-T durability shall be evaluated as well as the effects of aggregate chemistry and mineralogy on the aggregate. Pore structure and F-T durability shall be investigated.

REFERENCES

- 1. Federal Highway Administration, U. D. (2013). ROAD WEATHER MANAGEMENT PROGRAM. Weather Events, Snow and Ice.
- 2. Mehta, P. (1993). Concrete: Structure, Proerties, and Methods. Emglewood Cliffs, NJ: Prentice Hall.
- 3. G.G.Litvan. K.Sakai. (August, 1991). *Low Temp Effects on Concrete.* Ottawa: Second Canada/Japan Workshop.
- 4. Koubaa, A., Snyder, M. B., and Peterson, M. K. (1997). Mitigating Concrete Aggregate Problems in Minnesota. Minnesota Department of Transportation, MN/RC 2004-46.
- 5. McLeod, H. (2012). *D-Cracking Field Performance of Portland Cement Concrete Pavements Containing Limestone in Kansas.* Phase 1 Report, Bureau of Materials and Research & KDOT.
- 6. Powers, T.C., and P.A. Helmuth. (1953). *Theory of Volume Changes in Hardened Portland Cement Paste During Freezing.* Vol. 32. Highway Research Board, National Research Council. Washington, D.C. pp. 285-297
- 7. Verbeck, G. and R. Landgren. (1960). *Influence of Physical Characteristics of Aggregates on Frost*. Vol. 60. American Society for Testing and Materials. Conshohocken, PA pp. 1063-1079.
- 8. Vanderhorst, N.M., and D.J. Janssen. (1990). *The Freezing and Thawing Environment: What Is Severe?* Farmington Hills, Michigan: Paul Klieger Symposium, ACI SP 122. American Concrete Institute. pp. 181-200.
- 9. Stark, D. (1970). *Field and Laboratory Studies of the Effects of Subbase Type on the Development of D-Cracking*. Highway Research Record 342. Highway Research Board. Washington D.C
- 10. Schwartz, D. (1987). *D-Cracking of Concrete Pavements*. Washington, D.C: NCHRP Synthesis Practice 134. Transportation Research Board, National Research Council.
- 11. Janssen, D.J. and Snyder, M.B. (1994). *Resistance of Concrete to Freezing and Thawing*. Strategic Highway Research Program.
- 12. Stark, D., and P. Klieger. (1973). *Effects of Maximum Size of Coarse Aggregate on D-Cracking in Concrete Pavements*. Highway Research Record No. 441. Highway Research Board. Washington, D.C.
- 13. Powers, T. C. (1955). *Basic Considerations Pertaining to Freezing-and-Thawing.* American Society for Testing and Materials Proceedings, V. 55, pp. 1132-1155.

- 14. Rhoades, R. and Mielenz, R.C. (1946). *Petrography of Concrete Aggregates. Proceedings,* American Concrete Institute Journal, No.6, Vol. 42, p. 581.
- 15. Lewis, D.W., W.L. Dolch, and K.B. Woods. (1953). *Porosity Determination and the Significance of Pore Characteristics of Aggregates*. Proceedings, Volume 53. American Society for Testing and Materials. Conshohocken, Pennsylvania. pp.449.
- 16. Dolch, W. (1956). *Permeability and Absorptivity of Indiana Limestone Coarse Aggregates*. Ph.D.Thesis. Purdue University. West Lafayette, Indiana.
- 17. Kaneuji, M. (1978). *Correlation Between the Pore-Size Distributions and F-T Durability of Coarse Aggregates in Concrete*. Ph.D. Thesis. Purdue University. West Lafayette, Indiana.
- 18. Salcedo, M. (1985). *Identification of Frost Susceptible Aggregates and Their Use in Concrete or Bituminous Pavements*. Ph.D. Thesis. Purdue University. West Lafayette, Indiana.
- 19. Marks, V.J., and W. Dubberke. (1982). *Durability of Concrete and the Iowa Pore Index Test*. Transportation Research Record 853. Transportation Research Board. Washington, D.C
- 20. Mehta, P.K., and P.J.M. Montiero. (1993). *Concrete: Structure, Properties, and Materials*. Prentice Hall. Englewood Cliffs, New Jersey
- 21. Korhonen, C. and Charest, B. (1995). *Assessing Cryogenic Testing of Aggregates for Concrete Pavements*. Special Report 95-4, US Army Corps of Engineers, Cold Regions Research and Engineering Laboratory.
- 22. Hudec, P. P. (1989). *Durability of Rock as Function of Grain Size, Pore Size, and Rate of Capillary Absorption of Water*. Journal of Materials in Civil Engineering, V. 1, No. 1, pp. 3-9.
- 23. Girard, R.J., E.W. Myers, G.D. Manchester, and W.L. Trimm. (1982). *D-Cracking: Pavement Design and Construction Variables*. Transportation Research Record 853. Transportation Research Board. pp. 25-30. Washington, D.C.
- 24. Shakoor, A. (1982). *Evaluation Durability Characteristics of Argillaceous Carbonate Aggregates for Highway Pavements*. Ph.D. Thesis. Purdue University. West Lafayetee, Indiana.
- 25. Meininger, R.C., J.F. Fox, and H.A. Lepper. (1965). *A Single Particle Freezing Test for Concrete Aggregate.* Proceedings, Volume 65. American Society for Testing and Materials. pp. 801-808. Conshohocken, Pennsylvania.
- 26. Bukovatz, J.E., and C.F. Crumpton. (1981). *Study of D-Cracking in Portland Cement Concrete Pavements*, Vol 2: Laboratory Phase. Report FHWA-KS-81-2. Kansas Department of Transportation. Topeka, Kansas.

- 27. Klieger, P., G. Monfore, D. Stark, and W. Teske. (1974). *D-Cracking of Concrete Pavements in Ohio*. Final Report: Ohio-DOT-11-74. Ohio Department of Transportation. Columbus, Ohio
- 28. Dubberke, W. and V.J. Marks. (1985). *The Effect of Salt on Aggregate Durability*. Transportation Research Record 1031. Transportation Research Board, National Research Council. pp. 27-34. Washington D.C.
- 29. Stark, D. (1976). *Characteristics and Utilization of Coarse Aggregates Associated with D-Cracking*. Skokie, IL.: Bulletin RD047.01P, Portland Cement Association.
- 30. Boucher, D. L., and Selig, E. T. (1987). *Application of Petrographic Analysis to Ballast Performance Evaluation*. Transportation Research Record, No. 1131, p. 15-25.
- 31. Williamson, G. S. (2005), *Investigation of Testing Methods to Determine Long-Term Durability of Wisconsin Aggregate Resources Including Natural Materials, Industrial By-Products, and Recycled/Reclaimed Materials*, MS Thesis, Virginia Polytechnic Institute and State University.
- 32. Mummaneni, S. K., & Riding, K. A. (June 2012). *Evaluation of Canadian Unconfined Aggregate F-T Tests for Identifying Nondurable Aggregates.* Department of Civil Engineering, Kansas State University Transportation Center: K-TRAN: KSU-10-9.
- 33. Dubberke, W. and Myers, J. D., 1980, *Iowa Pore Index Test.* Iowa Department of Transportation, Ames, Iowa, 4 pp.
- 34. Koubaa, A and Snyder, M. B. (2001). *Assessing Frost Resistance of Concrete Aggregates in Minnesota.* Journal of Cold Regions Engineering, V. 15, No. 4, pp. 187-210.
- 35. Scholer, C. F. and Shakoor, A. (1985). *Comparison of Aggregate Pore Characteristics as Measured by Mercury Intrusion Porosimeter and Iowa Pore Index Test*. American Concrete Institute Journal, July- Aug., V. 82, No. 4, pp. 453-458.
- 36. Olsen, M. P.J., Janssen, D. J., and Dempsey, B. J. (1983). *D-Cracking in Portland Cement Concrete Pavements.* Urbana, IL: University of Illinois and Illinois Department of Transportation, Final Report, FHWA/IL/UI-199.
- 37. Barbu, B. G. and Scullion, T. (2006), *Repeatability and Reproducibility Study for Tube Suction Test,* Technical Report: Report 5-4114-01-1, Texas Department of Transportation.
- 38. Kahraman, S. and Toraman, O. Y. (2008). *Predicting Los Angeles abrasion loss of rock aggregates from crushability index*. Bulletin of Material Science, Indian Academy of Sciences, Vol. 31, No. 2, p. 173–177.
- 39. Michael D.A. Thomas, Benoit Fournier, and Kevin J. Folliard. (April 2008). *Report on Determining the Reactivity of Concrete Aggregates and Selecting Appropriate Measures*

for Preventing Deleterious Expansion in New Concrete Construction. The Transtee Group, Inc. DTFH61-D-00035

$$
\lim_{\omega\rightarrow\infty}\mathbf{Z}[\mathbf{K}(\mathbf{L}(\omega),\omega)]
$$

APPENDIX A. SUPPLEMENTARY FIGURES

Actual Water Absorption Backup Figures

(a) Water Absorbed within 30minutes-24 hrs. vs. SPI

Figure 34: Relationship between Water Absorbed and SPI

Absorption/Desorption Behavior Curve of Each Source of Aggregate

Source ID: 03081

(i) Carbonate Absorption Curve

(iii) Non-Carbonate Absorption Curve

(a) Source ID 03081 Absorption/Desorption Behavior Curve

(i) Carbonate Absorption Curve

(iii) Non-Carbonate Absorption Curve

(b) Source ID 03090 Absorption/Desorption Behavior Curve

Source ID: 14074

(ii) Carbonate Desorption Curve

(iii) Non-Carbonate Absorption Curve

(c) Source ID 14074 Absorption/Desorption Behavior Curve

Source ID: 19001

(i) Carbonate Absorption Curve

(iii) Non-Carbonate Absorption Curve

(d) Source ID 19001 Absorption/Desorption Behavior Curve

(i) Carbonate Absorption Curve

(iii) Non-Carbonate Absorption Curve

(e) Source ID 34002 Absorption/Desorption Behavior Curve

Source ID: 86001

(ii) Carbonate Desorption Curve

(iii) Non-Carbonate Absorption Curve

(f) Source ID 86001 Absorption/Desorption Behavior Curve

Source ID: 19109

94

(i) Carbonate Absorption Curve

(iii) Non-Carbonate Absorption Curve

(g) Source ID 19109 Absorption/Desorption Behavior Curve

(i) Carbonate Absorption Curve

(iii) Non-Carbonate Absorption Curve

(h) Source ID 67001 Absorption/Desorption Behavior Curve

Source ID: 56192

(i) Carbonate Absorption Curve

(ii) Carbonate Desorption Curve

(iii) Non-Carbonate Absorption Curve

(iv) Non-Carbonate Desorption Curve

(i) Source ID 56192 Absorption/Desorption Behavior Curve

Source ID: 82001

101

(i) Carbonate Absorption Curve

(ii) Carbonate Desorption Curve

(iii) Non-Carbonate Absorption Curve

(iv) Non-Carbonate Desorption Curve

(j) Source ID 82001 Absorption/Desorption Behavior Curve

(i) Carbonate Absorption Curve

(ii) Carbonate Desorption Curve

(iii) Non-Carbonate Absorption Curve

(iv) Non-Carbonate Desorption Curve

(k) Source ID 56003 Absorption/Desorption Behavior Curve

Source ID: 70008

(i) Carbonate Absorption Curve

(ii) Carbonate Desorption Curve

(iii) Non-Carbonate Absorption Curve

(iv) Non-Carbonate Desorption Curve

(l) Source ID 70008 Absorption/Desorption Behavior Curve

Source ID: 79091

108

(i) Carbonate Absorption Curve

(ii) Carbonate Desorption Curve

(a) Source ID 79091 Absorption/Desorption Behavior Curve

Source ID: 70006

(i) Carbonate Absorption Curve

(ii) Carbonate Desorption Curve

(m) Source ID 70006 Absorption/Desorption Behavior Curve

Source ID: 82002

(i) Carbonate Absorption Curve

(ii) Carbonate Desorption Curve

(n) Source ID 82002 Absorption/Desorption Behavior Curve

Figure 35: Absorption/Desorption Behavior Curve of Each Source of Aggregate

APPENDIX B. ORIGINAL DATA

IPI Test Results of Each Source of Aggregate

Table 21: IPI Test Results of Each Source of Aggregate

Aggregate ID: 56003 Carbonate aggregate content (% by wt of bulk $a \sigma \sigma \sigma$

aggregate).		22.0370					
Testing Date	Sample	Weight, g		Reading ΔH , ml		Primary pore index (1 min reading)	Secondry pore index (14 min reading)
May 27th, 2013	carbonate-sample A	4499	56	14	3	84.02	28.01
May 27th, 2013	carbonate-sample B	4499.4	60	15	3	92.01	30.00
May 27th, 2013	carbonate-sample C	4500.2	58	15	3	88.00	30.00
May 27th, 2013	non-carbonate-sample A	4499	38	6	3	48.01	12.00
May 27th, 2013	non-carbonate-sample B	4500.2	40	5	3	52.00	10.00
May 27th, 2013	non-carbonate-sample C	4500.7	38	6	3	47.99	12.00
May 27th, 2013	non-carbonate-sample D	4500.5	38	5	3	47.99	10.00
May 27th, 2013	non-carbonate-sample E	4499.7	40	6	3	52.00	12.00
May 27th, 2013	Bulk Aggregate	4500.9	48	9	3	67.99	18.00
AVE of samples		Carbonate				88.01	29.34
		Non-carbonate				49.60	11.20
		Bulk Aggregate				67.99 4.00 2.19 4.54% 4.42% 88.01 49.33	18.00
	Carbonate						1.15
STD of samples		Non-carbonate				1.10	
%STD of samples	Carbonate						3.93%
		Non-carbonate					9.79%
AVE of mid 3 samples		Carbonate					29.34
		Non-carbonate					11.33

Aggregate ID: 56192 Carbonate aggregate content (% by wt of bulk α goregate): 44.23%

aggiegaic).		——…∠					
Testing Date	Sample	Weight, $\mathbf{g}% _{0}$		Reading ΔH , ml		Primary pore index (1 min reading)	Secondry pore index (14 min reading)
Jan 29th, 2013	carbonate-sample A	4499.1	62	15	3	96.02	30.01
Jan 30th, 2013	carbonate-sample B	4500.5	64	14	3	99.99	28.00
Jan 30th, 2013	carbonate-sample C	4500.7	60	15	3	91.99	30.00
Jan 31th, 2013	carbonate-sample D	4500.3	64	17	3	99.99	34.00
Jan 31th, 2013	carbonate-sample E	4500.4	62	17	3	95.99	34.00
Jan 29th, 2013	non-carbonate-sample A	4500.2	39	3		50.00	6.00
Jan 30th, 2013	non-carbonate-sample B	4499.8	36	$\overline{2}$	$\overline{1}$	44.00	4.00
Jan 30th, 2013	non-carbonate-sample C	4499.5	38	3	1	48.01	6.00
Jan 31th, 2013	non-carbonate-sample D	4499.1	36	$\mathfrak{2}$	-1	44.01	4.00
Jan 31th, 2013	non-carbonate-sample E	4500.7	38	3		47.99	6.00
Jan 31th, 2013	Bulk Aggregate	4499.6	54	9	$\overline{2}$	80.01	18.00
AVE of samples		Carbonate				96.80	31.20
		Non-carbonate				46.80	5.20
		Bulk Aggregate				80.01	18.00
STD of samples		Carbonate				3.35	2.68
		Non-carbonate				2.68	1.10
%STD of samples		Carbonate				3.46%	8.60%
		Non-carbonate				5.73%	21.06%
AVE of mid 3 samples		Carbonate				97.33	31.33
		Non-carbonate				46.67	5.33

Aggregate ID: 67001

Carbonate aggregate content (% by wt of bulk aggregate): 25.30 %

Aggregate ID: 82001 Carbonate aggregate content (% by wt of bulk

 Λ ggregate ID: 86001

Specific Gravity and Absorption of Each Source of Aggregates

Table 22: Specific Gravity and Absorption of Each Source of Aggregates

128

Aggregate ID: 19109	
---------------------	--

Carbonate aggregate content (% by wt of bulk aggregate): 13.53%

Aggregate ID:

67001

Aggregate ID: 70008

 C arbonate aggregate content $\left(\%$ by wt of bulk aggregate): 21.30%

Table 22 continued

Aggregate ID: 79091

Carbonate aggregate content (% by wt of bulk aggregate): 100%

Table 22 continued

