

EVALUATION OF CANADIAN UNCONFINED AGGREGATE FREEZE-THAW TESTS
FOR IDENTIFYING NONDURABLE AGGREGATES

by

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Abstract

Concrete is most widely used material in construction industry, which is made up of cement, water and aggregates as its major ingredients. Aggregates contribute to 60 to 75 % of the total volume of concrete. The aggregates play a key role in the concrete durability. The U.S Midwest has many aggregates that can show distress in the field under freezing and thawing conditions. The objective of this research was to determine if the Test Method for the Resistance of Unconfined Coarse Aggregate to Freezing and Thawing, method CSA A23.2-24A, could be used to differentiate good from poor performing aggregates in concrete. In this study fifty one KDOT aggregates (including twelve ledge and thirty nine production samples) were tested for freeze thaw resistance using CSA A23.2-24A test method and were compared to the results of the standard KDOT aggregate qualification tests. In addition to performing the CSA test method using a 3% sodium chloride solution, a subset of the aggregates were tested using either a 3% magnesium chloride or calcium chloride solution to determine the effects of the salt type on the aggregate performance. No correlation was found between the CSA A23.2-24A test method results and the standard KDOT aggregate qualification tests. The results also indicated that the mass loss in the CSA A23.2-24A was similar for the aggregate sizes tested. The use of alternate salt solutions like $MgCl_2$ and $CaCl_2$ resulted in increased freeze thaw mass loss in limestone aggregates.

Table of Contents

List of Figures	vi
List of Tables	viii
Acknowledgements.....	x
Dedication.....	xi
Chapter 1 -Introduction.....	1
1.1 Research Background	1
1.2 Problem Statement.....	1
1.3 Objectives & Scope of Study.....	1
1.4 Thesis Outline	2
Chapter 2 -Literature Review.....	3
2.1 Freeze thaw damage mechanism for aggregates in concrete	3
2.2 Aggregate properties related to freeze thaw behavior	7
2.2.1 Porosity & Pore size distribution	7
2.2.2 Absorption & Bulk specific gravity.....	8
2.2.3 Effect of deicer salts on aggregate	9
2.2.4 Mineralogy – role of clays, impurities.....	10
2.2.5 Aggregate mineralogy.....	11
2.2.6 Aggregate size.....	12
2.3 Ways to test for aggregate freeze thaw	12
2.3.1 Test description - NTBUILD 485 Standards	13
2.3.2 EN 1367-1 European standards of freeze thaw testing.....	15
2.3.3 Test description - CEN/TC 154 & TC 227 standards in Iceland	15
2.3.4 Test description - The Washington Hydraulic Fracture Test (WHFT).....	16
2.3.5 Test description - Modified Hydraulic Fracture Testing Procedure	17
2.3.5.1 Hydraulic Fracture Testing – Smaller chamber	17
2.3.5.2 Modified Hydraulic Fracture Testing – Large Chamber	18
2.3.6 Iowa Pore Index test.....	18
2.3.7 Coarse aggregate freeze-thaw test TX DOT designation: TEX-432-A	19

2.3.8	NDR standard method T 103 soundness of aggregates by freezing and thawing	19
2.3.9	German standard DIN 4226 (1971) freeze thaw testing of aggregates.....	20
2.3.10	CSA A23.2-24A Test Method for unconfined coarse aggregate to freezing & thawing	20
Chapter 3 - Methods & Materials Used		23
3.1	KTMR-27 Modified Specific Gravity and Absorption of aggregate test method	24
3.2	Canadian freeze thaw testing CSA A23.2-24A (laboratory testing).....	25
3.2.1	CSA A23.2-24A test procedure	25
3.2.2	Materials tested	26
3.2.3	Locally available control aggregate	27
3.2.4	Modifications for particle size to accommodate production samples.....	31
3.2.5	Effects of salt type	31
3.3	BET Nitrogen Adsorption.....	31
3.3.1	Introduction.....	31
3.3.2	Procedure	34
3.3.3	Results & calculations.....	38
Chapter 4 -Results & Discussions.....		39
4.1	Specific Gravity and Absorption results using KTMR-27 test procedure	39
4.2	Comparison of original to the modified version of CSA A23.2-24A test method	41
4.3	Comparison of Average Freeze thaw loss of NaCl solution with MgCl ₂ and CaCl ₂ salt solutions	44
4.4	Nitrogen Adsorption Experiments	46
4.5	Comparison of average freeze thaw loss with different aggregate performance measures done by KDOT.....	49
Chapter 5 - Conclusions & Recommendations for Future Research		55
5.1	Conclusions.....	55
5.2	Recommendations for Future Research	56
References.....		57
Appendix A - CSA A23.2-24A tests on Ledge samples.....		61
Appendix B - CSA A23.2-24A tests on Production samples.		68
Appendix C - Modified CSA A23.2-24A tests results using MgCl ₂ and CaCl ₂ salt solutions		81

Appendix D - Results from KDOT tests on Companion aggregates 84
Appendix E - BJH Calculations on Selected KDOT Aggregates 87

List of Figures

Figure 2-1: Joint rot observed in concrete pavement near Claflin-Denison Intersection.	5
Figure 2-2: Joint rot in which poor quality aggregates, can be seen popping out of the concrete pavement at the joints.	5
Figure 2-3: D-cracks (low intensity) observed near joint on College Avenue.	6
Figure 3-1: Apparatus for performing the KTMR-27 test method	25
Figure 3-2: Cumulative Freeze Thaw loss vs. Sieving Time (min) for limestone aggregates and average of three Canadian aggregates sets on 0.75”-0.5” fraction.	29
Figure 3-3: Cumulative Freeze Thaw Loss vs. Sieving Time (min) for the local control limestone aggregate and the average of three Canadian aggregates sets on 0.5”-0.375” fraction.	29
Figure 3-4: Cumulative Freeze Thaw Loss (%) vs. Sieving Time (min) for limestone aggregates and the average of three Canadian aggregates sets on 0.375”-0.25” fraction.....	30
Figure 3-5: Comparison of Combined Freeze Thaw Loss (%) from all three sizes vs. Sieving Time (min) for average of six Canadian aggregate sets and twelve local control limestone aggregate sets.	30
Figure 3-6: Gas molecules adsorbed on to the surface	32
Figure 3-7: B.E.T Autosorb-1 test apparatus	33
Figure 3-8: Bulb into which the sample should go in.	35
Figure 3-9: Final arrangement of the bulb.	35
Figure 3-10: Bulb placed into the insulating heat bag.	36
Figure 3-11: Final arrangement of bulb before out gassing.....	36
Figure 3-12: Dewar being lifted up into its slot after filling up with Nitrogen.	37
Figure 3-13: BET plot for KDOT limestone aggregate with area below the graph.	38
Figure 4-1: Comparison of aggregate weight loss (%) for the 0.75-0.5 in. aggregates and the 0.375 - 0.25 in. aggregates tested.....	42
Figure 4-2: Comparison of aggregate weight loss (%) for the 1.5-1 in. aggregates and the 0.375 - 0.25 in. aggregates tested.	42
Figure 4-3: Comparison of aggregate weight loss for the 1-0.75 in. aggregates and the 0.375 - 0.25 in. aggregates tested.	43

Figure 4-4: Comparison of aggregate weight loss for the 0.75-0.5 in. aggregates and the 0.5 - 0.375 in. aggregates tested.	43
Figure 4-5: Comparison of aggregate freeze thaw weight loss for NaCl versus the MgCl ₂ and CaCl ₂ salt solutions.	44
Figure 4-6: Comparison of NaCl and CaCl ₂ salt solutions tested using CSA A23.3-24A (For Sample 1008 with DF=99).....	45
Figure 4-7: Comparison of NaCl and CaCl ₂ effects on the freeze thaw weight loss using CSA A23.3-24A. (For Sample 1918 with DF=98).....	45
Figure 4-8: Comparison of local aggregate freeze thaw weight loss (%) for NaCl, MgCl ₂ and CaCl ₂ salt solutions using CSA A23.2-24A test method.	46
Figure 4-9: Comparison of KTMR-22 Durability factors and Aggregate Surface Area.	46
Figure 4-10: Linear variation observed in the BET plot for sample 09-1468 B9 with DF=37. ...	47
Figure 4-11: Volume–Relative Pressure plot for sample 09-1248 B9 with DF=99.	47
Figure 4-12: Variation of c term with r _p for pores of various radii.....	48
Figure 4-13: Pore Volume Distribution curves for aggregates with different durability factors..	49
Figure 4-14: Aggregate weight loss (%) vs. Pavement Vulnerability Factor (PVF) for KDOT aggregates.	50
Figure 4-15: Aggregate weight loss (%) vs. KTMR-22 Durability Factor for KDOT aggregates.	50
Figure 4-16: Aggregate weight loss (%) vs. Aggregate Modified Soundness test for KDOT aggregates.	51
Figure 4-17: Aggregate weight loss (%) vs. Absorption values for KDOT aggregates.	51
Figure 4-18: Aggregate weight loss (%) vs. Wear (%) for KDOT aggregates.....	52
Figure 4-19: Aggregate Modified Soundness test vs. KTMR-22 Durability Factor for KDOT aggregates.	53
Figure 4-20: Wear (%) vs. KTMR-22 Durability Factor for KDOT aggregates.....	53
Figure 4-21: Pavement Vulnerability Factor vs. KTMR-22 Durability Factor for KDOT aggregates.	54

List of Tables

Table 2.1: Critical pore sizes range obtained from various researches.....	8
Table 2.2: Quantities of different size of aggregates in the sample.....	14
Table 2.3: Weights of individual aggregate size fractions (lb.).....	19
Table 2.4: Quantities of different sizes present in the sample	21
Table 3.1: Sample weights needed for different aggregate size fractions, (CSA, 2004).....	26
Table 3.2: Required mass of aggregates (gm) separated into different fractions (for production samples)	27
Table 3.3: Data for Canadian reference aggregate (unpublished data, MTO 2009).....	27
Table 3.4: Mean and standard deviation of freeze thaw mass loss for MTO aggregate local aggregate.	28
Table 4.1: Specific Gravity and Absorption values for ledge samples.....	39
Table 4.2: Specific Gravity and Absorption values for production samples	40
Table 4.3: Specific gravity and absorption values for Canadian aggregates along with one set of local control aggregates	41
Table A.1: CSA A23.2-24A test results for 1.5-1" ledge sample.....	62
Table A.2: CSA A23.2-24A test results for 1-0.75" ledge samples.	63
Table A.3: CSA A23.2-24A test results on 0.75-0.5" sieve fraction.....	64
Table A.4: CSA A23.2-24A test results on 0.5-0.375" sieve fraction.....	65
Table A.5: CSA A23.2-24A test results on 0.375-0.25" sieve fraction.....	66
Table A.6: Average Weighted Freeze Thaw Loss for ledge samples using CSA A23.2-24A method.....	67
Table B.1: CSA A23.2-24A test results on 0.75-0.5" sieve.....	69
Table B.2: CSA A23.2-24A test results obtained on 0.5-0.375" sieve.....	70
Table B.3: CSA A23.2-24A test results obtained on 0.375-0.25" sieve.....	71
Table B.4: Average Weighted Freeze Thaw Loss for production samples using CSA A23.2-24A method.....	72
Table B.5: CSA A23.2-24A test results on 0.75-0.5" size fractions.....	73
Table B.6: CSA A23.2-24A test results on 0.5-0.375" size fractions.....	74
Table B.7: CSA A23.2-24A test results on 0.375-0.25" size fractions.....	75

Table B.8: Average Weighted Freeze Thaw Loss for production samples using CSA A23.2-24A method.....	76
Table B.9: CSA A23.2-24A test results on 0.75-0.5" size fractions.....	77
Table B.10: CSA A23.2-24A test results on 0.5-0.375" size fractions.....	78
Table B.11: CSA A23.2-24A test results on 0.375-0.25" size fractions.....	79
Table B.12: Average Weighted Freeze Thaw Loss for production samples using CSA A23.2-24A method.....	80
Table C.1: Average Weighted Freeze Thaw Loss for production samples by using CaCl ₂ salt solution method.....	82
Table C.2: Average Weighted Freeze Thaw Loss for production samples by using MgCl ₂ salt solution method.....	83
Table D.1: KDOT results on ledge samples.	85
Table D.2: KDOT results on production samples.....	86
Table E.1: BJH calculations for Sample 09-1468 B9 (DF=37).....	88
Table E.2: BJH Calculations for sample 09-1010 (DF=99).	89
Table E.3: BJH Calculations for sample 09-1228 (DF=99).	90
Table E.4: BJH Calculations for 09-1231 sample (DF=98).	91
Table E.5: BJH Calculations for sample 09-1257 (DF=98).	92
Table E.6: BJH Calculations for sample 09-1430 (DF=99).	93
Table E.7: BJH Calculations for local control aggregates.....	94
Table E.8: BJH Calculations for Granite aggregate (09-1939).....	95

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Dedication

I dedicate this to my parents who helped throughout my life and truly are a guiding force behind my every success.

Chapter 1 -Introduction

1.1 Research Background

The Kansas Department Transportation (KDOT) wants to construct durable concrete pavements, with minimal maintenance needs. This goal can only be achieved by using durable aggregates that are resistant to freezing and thawing damage when used in concrete. KDOT uses the Soundness and Modified Soundness of Aggregates by Freezing and Thawing test method, KTMR-21, as a first screening test to consider aggregates to be used in concrete. However, the method that KDOT employs is time consuming, doesn't represent actual field conditions, and may pass non-durable aggregates. There is a critical need of a quick and field representative test method that classifies durable aggregates from the nondurable ones. The Canadian freeze thaw method CSA A23.2-24A test method, developed by the Ministry of Transportation Ontario (MTO) (CSA A23.2-24A, 2004) was developed to quickly screen aggregates for freezing and thawing durability. The method was developed to use salt solutions instead of water to saturate the aggregates before freezing to be more representative of the concrete field conditions.

1.2 Problem Statement

Freeze thaw deterioration of aggregates in concrete is the biggest durability faced by Kansas concrete pavements (Clowers, 1999). The main objective of this study is to determine any correlations between the CSA A23.2-24A method and the currently used KDOT aggregate qualification methods to allow for use of the simpler and more rapid CSA test method rather than the time consuming currently used set of tests.

1.3 Objectives & Scope of Study

The main objectives of this study are:

1. To determine the ability of Canadian aggregate freeze thaw test method CSA A23.2-24A to assess the freeze-thaw resistance of ledge sample unconfined coarse aggregates to freeze thaw damage by comparison to the currently used KDOT aggregate qualification tests.
2. To estimate the ability of Canadian aggregate freeze thaw test method CSA A23.2-24A to assess the freeze-thaw resistance of production coarse aggregates to freezing and thawing cycles by comparison to the currently used KDOT aggregate qualification tests.

3. To determine if the use of magnesium or calcium chloride salt solutions in the CSA A23.2-24A test method correlate better to the currently used KDOT aggregate qualification tests than sodium chloride salt solutions.

1.4 Thesis Outline

This thesis consists of following chapters.

Chapter 1- Introduction: Describes about the necessity of employing CSA A23.2-24A test method in screening durable aggregates from the nondurable ones. This chapter briefly explains the problem statement and draws objectives associated with the study.

Chapter 2- Literature Review: Describes briefly background work done by researchers on freeze thaw damage mechanism and describes different factors effecting freeze thaw durability of aggregates. This section also summarizes different freeze thaw methods employed around the world to screen durable aggregates.

Chapter 3- Methodology: Explains different study methods such as KTMR-27 test method to calculate specific gravity and absorption for KDOT aggregates, CSA A23.2-24A test method for resistance of unconfined coarse aggregate to freezing and thawing and BET Nitrogen Adsorption test methods to calculate specific surface area and to compute pore volume distributions.

Chapter 4- Results and Discussions: Reports results and discussions from the tests conducted as explained in Methodology section. This chapter links the results obtained from tests to the main objectives of the study and leads to conclusions.

Chapter 5- Conclusions and Recommendations: Highlights the major findings from the project and provides with recommendations for future research.

Chapter 2 -Literature Review

2.1 Freeze thaw damage mechanism for aggregates in concrete

Freezing and thawing damage is one of the major causes of distress in concrete pavements. The paste portion of the concrete can be especially susceptible to freezing and thawing damage in concrete, but can be protected by the use of air entraining admixtures (AEA) to stabilize microscopic bubbles in concrete. Concrete containing unsound coarse aggregates result in deterioration of concrete from repeated freezing and thawing cycles. There are several theories that explain frost behavior of aggregates. Initially a theory was proposed called the critical saturation theory, which stated that the freezing of water in pores result in expansion from the phase change, stressing the pore walls and cracking. Collins [1944] proposed the ice lens formation theory. According to this theory, in porous materials, ice lens are formed in a direction perpendicular to the heat flow (Sharon L. & Peter J., 1990). However, this theory couldn't explain the importance of air voids in paste. The role of air voids is thoroughly explained in hydraulic pressure theory (Powers T., 1949). According to this theory with the presence of dissolved chemicals in water, water freezes at a lower temperature than 32°F. In the case of a saturated paste, expansion due to freezing cannot be accommodated due to the lack of available space in pores. Aggregates are forced to expel water outside the particles since an increase in volume is encountered from the formation of ice. This expelled water has to move to an air void through cement paste which is a permeable medium (Michigan Tech Transportation Institute, 2002). The pressure required for water to travel a certain distance in a given time can be determined by Darcy's law as shown in Equation 2.1.

$$\Delta h = \frac{\eta}{k} * Q * \frac{l}{A} \qquad \text{Equation 2.1}$$

where,

Δh = Pressure Gradient, η is the fluid viscosity, k is the permeability, Q is the flow rate
 l is the length of the flow path, A is the flow area.

If the disruptive pressures generated are greater than the tensile strength of the material, then damage occurs. This theory is only applicable to air voids that are of the same size and equally spaced, which is not true in real concrete. This theory was later shown to have some

problems as some experiments have shown that the water travels towards capillary pores (Guthrie, 2002). The phenomenon of elastic accommodation can be better examined when the particle deforms elastically to accommodate an increase in volume due to pressure caused by ice formation. This parameter is a function of aggregate elastic properties and total amount of freezable water (Verbeck, G. & Landgren, R., 1960).

The osmotic pressure theory was developed to address some of the limitations of the hydraulic pressure theory. According to this theory, a thermodynamic equilibrium is maintained in capillary and gel pores when the temperature is above 32°F (Guthrie, 2002). The phenomenon of osmosis occurs when there is a difference in concentration levels. With the formation of ice in capillary pores, the concentration of dissolved species in the unfrozen water increases, which disturbs the pore solution equilibrium (Guthrie, 2002). Water from the gel pores moves towards the water in large pores through osmosis, reestablishing equilibrium (Mindess.S, 2003). Water rapidly freezes in capillary pores and internal stresses in the paste are increased from osmotic pressure occurred due to movement of water. A temperature decrease can then decrease the minimum pore size containing ice. The role of air voids are however not thoroughly explained in this theory (Powers T., 1975).

A theory applicable to all porous materials was developed by Litvan in 1975. According to this theory, water cannot freeze in situ and failure or mechanical damage can be attributed to the process of desorption which is essential to establish equilibrium with vapor pressure. This theory states that with the increase in freezing rate the risk of damage increases. However, the Litvan theory fails to establish a clear relationship between freezing rate, properties of paste and spacing of air voids. The critical particle size can be defined as the size below which aggregates are frost resistant. Critical size is a function of permeability and tensile strength of aggregate (Verbeck, G. & Landgren. R., 1960). Aggregates, which make up the majority of the concrete volume, can also be susceptible to freezing and thawing damage in concrete. D-cracking generally occurs in concrete pavements due to presence of poor freeze thaw resistant aggregates. Marginal quality aggregates have also been linked to joint rot in pavements, which is shown in Figures 2-1 and 2-2.



Figure 2-1: Joint rot observed in concrete pavement near Claflin-Denison Intersection.



Figure 2-2: Joint rot in which poor quality aggregates, can be seen popping out of the concrete pavement at the joints.

D-cracking is observed in aggregates with certain pore sizes and the damage is more predominant in the presence of deicer salts (Dubberke, 1983). However, freeze thaw resistance of concrete that contain aggregates which are prone to D-cracking can be improved by increasing the air content of the mixture (Schlorholtz, 2000). D-cracking can potentially be reduced by reducing particle size. D-cracking is commonly observed in limestone, dolomite and chert aggregates, which are all sedimentary rocks (Stark.D, 1976). It is believed that the aggregate pore size distribution plays a vital role in the freezing and thawing durability. Water present in fine pores doesn't freeze as readily as in larger pores because of freezing point depression (ACI, 1976). Aggregates prone to freeze thaw damage contribute to concrete deterioration as shown in Figures 2-3.



Figure 2-3: D-cracks (low intensity) observed near joint on College Avenue.

2.2 Aggregate properties related to freeze thaw behavior

2.2.1 Porosity & Pore size distribution

Concrete resistance to freezing and thawing can be affected by the porosity and absorption properties of the aggregate (Mindess.S, 2003). Freezing and thawing damage in aggregates occurs when the aggregate pores are filled with water and a freezing event occurs. A study done on the frost sensitivity of aggregates revealed that water present inside the aggregates is mostly due to adsorption (Hudec, 1987). During a freezing event, the water inside the pores can exert pressure on the pore walls which results in the formation of internal stresses and cracking (Hudec, 1987). The aggregate pore quantity and size distribution is a major factor in the aggregate frost durability (David N., 2009). Several studies (Hudec, 1978) (Kaneuji, 1978) (Kaneuji,M., D.N.Windslow, & W.L Dolch, 1980) showed that there is an interaction between pore size distribution and freeze thaw damage. From a study done by Kaneuji it was observed that for aggregates subject to freeze thaw tests, aggregates with larger pore sizes indicated lower durability (Kaneuji, 1978). Aggregates having large pores tend to accommodate more water into major part of pore space (Lewis et al., 1953). This is somewhat balanced by the fact that the larger pores have a lower saturation level because they empty first during drying. The aggregate permeability also tends to be higher, which makes it easier for the water in the aggregate to escape to an entrained air void during freezing, lowering the damage level. Freeze thaw damage is also encountered in aggregates with a large number of small pores (Lemish & Hiltrop, 1960; (Domaschuk,L. & Garychuk, G., 1988). There is a critical range of pore sizes as shown in Table 2.1, above which water frozen inside the pores can be easily expelled out from the pores. (Winslow, D.D., Lindgren,M.K., & Dolch,W.L, 1982).

Table 2.1: Critical pore sizes range obtained from various researches.

Study	Critical pore size	Comments on Study
Shakoor 1982	<0.01 μm	Unsound aggregates having high amount of clay content with 60% of pores less than 0.1 μm were considered for measurement of the pore size.
Shakoor 1982	0.01 μm -10 μm	Pore size was determined based on freeze thaw results on aggregates subjected to 5 % NaCl solution.
Salcedo 1984	0.045 μm -10 μm	Temperature and rate of temperature change was considered in determining the critical pore size for aggregates subjected to freeze thaw.
Dubberke and Marks 1985	0.04 μm -0.2 μm	Critical Pore size was determined for aggregates subjected to deicer salts

For pores in the 10 μm -0.1 μm range, the water in the pores has difficulty escaping the aggregate to reach an entrained air void before freezing damage occurs. On the contrary, large pore sizes allow water to easily escape, reducing pressure inside pores (David N., 2009). A study conducted to determine the relationship between pore size, durability & insoluble residue revealed that aggregates with more than 60 % of pores less than 0.1 μm were observed to be unsound (Shakoor, 1982). Aggregates have been shown to exhibit lower freeze-thaw durability with large pore volumes or small pore diameters (i.e., for pore sizes larger than 0.04 in. and not smaller than 45 Å) (Kaneuji, 1978). Critical pore size depends on temperature change and rate of temperature change, demonstrating that freezing and thawing damage is dependent on several parameters (Salcedo, 1984) Aggregates with certain types and distributions of clay and other minerals have also been shown to affect the performance of the aggregate. (Lemish,J. and Hiltrop. C.L, 1960).

2.2.2 Absorption & Bulk specific gravity

Several studies have attempted to establish a correlation between the concrete absorption, which is one measure of the total concrete porosity and the aggregate freeze-thaw durability in

concrete. It was observed during a study done on aggregates like shales, siltstones and argillaceous carbonate rocks that absorption of water had more destructive effects than freeze thaw damage (Hudec, P. & Dunn, J., 1972). Absorption of water into the concrete is mainly due to high porosity which results in lower compressive strength and increased freeze thaw damage. Aggregate expansion can occur from freezing of the aggregates in saturated condition (Powers T., 1949), although it was believed that a majority of the damage occurs from expansion of aggregates and not due to ice crystal formation (Hudec, 1987). Some studies have showed that aggregates with low absorption values ($< 0.3\%$) have good frost resistance (David N., 2009). A study conducted on some aggregates indicated a relationship between the absorption and durability factors. Aggregates with absorption percentage less than 1.5%, had durability factors higher than 80 and for absorption percentage greater than 2% the durability factors were observed to be less than 60 for Minnesota aggregates (Snyder, M. & Koubaa, A., 1996). Other studies have however shown that the use of absorption limits for aggregates have however shown to be poor general predictors of aggregate durability for a wide range of aggregates (Kaneuji, 1978). Bulk specific gravity can be related to porosity and mineralogy. It has been observed in various studies that absorption is generally assumed to be a better indicator of freeze thaw damage than specific gravity. Some studies have indicated that for some carbonate type aggregates there is a relationship that exists between durability and specific gravity. It was observed that aggregates with bulk specific gravities above 2.60 or 2.65 show good durability and are correlated with durability factors ((Koubaa & Snyder, 2001); (Harman et al., 1970)). This correlation was probably only applicable for aggregates from a small geographic area, and is not generally applicable. Aggregates having low specific gravities and possessing high absorption percentages such as lightweight aggregates may have very good freezing and thawing durability because of the lower aggregate elastic modulus and potentially larger pore sizes (Harman et al., 1970).

2.2.3 Effect of deicer salts on aggregate

Aggregate's performance under freeze thaw may be significantly different in the presence of deicer salts. Water can get absorbed into the aggregates through osmosis. This phenomenon can be observed when deicing salts like sodium chloride, magnesium chloride, calcium chloride etc. are added to aggregates that are already wet. The change in chemical concentration disrupts

the equilibrium between water in gel pores and capillary pores. In order to reestablish equilibrium, hydraulic forces develop within the. Osmotic phenomenon can be observed mostly in aggregates with high clay content and fine capillary pores (Shakoor, 1982), (Hudec, 1978). The difference in aggregate behavior for different salts such as NaCl, MgCl₂, and CaCl₂ needs determined.

2.2.4 Mineralogy – role of clays, impurities

Mineralogical composition and aggregate's structure are the two factors that help in assessing changes in source of aggregates and in determining harmful materials such as coal or other organic materials. Coarse aggregates that are made of crushed limestone indicate high affinity towards bonding with mortar, because of enhanced physical adhesion from texture of aggregate and might also be due to chemical bonding. Aggregate–mortar bond strength can be determined by assessing mineralogy of aggregates. Some researches indicated that pore water chemistry also has influence on the mineralogy of aggregates. Deleterious clay has been proved to be harmful for concrete (Buth et. al., 1964) (Buth et. al, 1967). The methylene blue test (AASHTO, 2007) is a simple method to determine clay content contained in aggregates (Yool et al, 1998). This test works based on the concept that clay materials have a large surface area and negative charge, which can be detected by an ion exchange phenomenon between methylene blue cations and clay ions. The results obtained from various studies have however created ambiguity amongst many researchers about the influence of clay content on aggregates. Some clays, such as smectite clays, exhibit swelling, whereas other types may be harmless. It is difficult however to measure the amount of the different clay types in aggregates. Previous studies also indicated that the presence of clay in any form likes dust, or surface coatings result in less durable aggregates. Other impurities such as magnesium, silica etc. also has a significant impact on aggregates.

There were contradictory results obtained from various studies regarding the role of magnesium content. One study indicated that damage is more significant in dolomites with calcium to magnesium ratio less than 9.0, although no clear connection has been made with other studies (Lemish,J. and Hiltrop. C.L, 1960). The presence of chlorides in the deicer salts has a negative impact on certain aggregates, such as carbonates (Dubberke, 1983). It was theorized that aggregates are more susceptible to attack by deicer salt if there is an increase in levels of

sulphur and manganese content (Dubberke, 1983). Various studies have proved that a smaller silica grain size results in enhanced surface area with which the salt reacts. Mineralogical composition and aggregate's structure are the two factors that help in assessing changes in source of aggregates and in determining harmful materials such as coal or other organic materials. Coarse aggregates that are made of crushed limestone indicate high affinity towards bonding with mortar, because of enhanced physical adhesion from texture of aggregate and might also be due to chemical bonding. Aggregate–mortar bond strength can be determined by assessing mineralogy of aggregates. Some researches indicated that pore water chemistry also has influence on the mineralogy of aggregates. Deleterious clay has been proved to be harmful for concrete (Buth et. al., 1964) (Buth et. al, 1967)). The methylene blue test (AASHTO, 2007) is a simple method to determine clay content contained in aggregates (Yool et al, 1998). This test works based on the concept that clay materials have a large surface area and negative charge, which can be detected by an ion exchange phenomenon between methylene blue cations and clay ions. The results obtained from various studies have however created ambiguity amongst many researchers about the influence of clay content on aggregates. Some clays, such as smectite clays, exhibit swelling, whereas other types may be harmless. It is difficult however to measure the amount of the different clay types in aggregates. Previous studies also indicated that the presence of clay in any form like dust, or surface coatings result in less durable aggregates. Other impurities such as magnesium, silica etc. also has a significant impact on aggregates.

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2.2.5 Aggregate mineralogy

The durability of aggregates is predominantly affected by the reactions that occur between aggregate and deicing salts under freeze thaw conditions (Dubberke, Wendell and

Vernon J. Marks, 1985). Deicer salts help the aggregates retain water for longer periods of time, keeping the aggregate pores in the concrete saturated longer which allows more water to freeze. Some aggregates have been shown to be more susceptible to salt than others. The d spacing inside the dolomite crystals subjected to deicer salt solution, indicated good correlation with aggregate durability (Wendell & Dubberke, 1987). The aggregate d-spacing can be determined by using x-ray diffraction and the maximum dolomite peak intensity indicated good relation with the aggregate service life in Iowa pavements (Wendell & Dubberke, 1987). Clays interspersed in the aggregates have also been shown to be more susceptible to freezing and thawing damage than those with clays in laminations (Shakoor, 1982).

2.2.6 Aggregate size

Aggregate gradation is one of the most important factors that influence the freeze thaw durability of concrete. When a freezing even occurs, water in the aggregates must escape the aggregate to an entrained air void in order to prevent damage from occurring. It is more difficult for water to escape to an air void in the paste from larger particles than smaller particles. The critical particle size may be defined as a size above which a particle which is fully saturated during freezing (Powers T., 1955). Critical size varies with the pore size distribution, connectivity, and tortuosity. In case of fine grained aggregates, lower permeability is generally observed which results in a critical particle size within the range of normal aggregates.

2.3 Ways to test for aggregate freeze thaw

There are many test methods used to determine the freeze thaw resistance of aggregates. Their testing procedures however changes from country to country depending on of the type and mineralogy of the aggregates. One early test developed, the sulfate soundness test ASTM C 88, was developed to simulate ice crystallization pressures in the aggregates from sulfate crystallization during five wetting and drying cycles. However, the results obtained from the test correlate poorly with the durability of aggregates in service or in concrete beam freezing and thawing tests (Garrity & Kriege, 1935). Many different aggregate freezing and thawing test methods have since been developed to determine the coarse aggregate suitability for use in concrete.

1. NTBUILD 485 Standards, Nordic.
2. EN 1367-1 European Standards of Freeze Thaw Testing

3. CEN/TC 154 & TC 227 Standards in Iceland or EN 1367-1 European Standards.
4. The Washington Hydraulic Fracture Test (WHFT).
5. Modified Hydraulic fracture method.
6. Iowa Pore Index test method.
7. Coarse Aggregate Freeze-Thaw Test TX DOT Designation: TEX-432-A
8. NDR Standard Method (Modified AASHTO T 103) Soundness of Aggregates by Freezing and Thawing.
9. German Standard DIN 4226 (1971) Freeze thaw testing of aggregates.
10. Canadian Freeze Thaw Testing (CSA A23.2-24A) Test Method for unconfined coarse aggregate to Freezing & Thawing.

2.3.1 Test description - NTBUILD 485 Standards

The NTBUILD 485 standard was adopted to assess the frost resistance of aggregates, when subjected to freezing and thawing. Field conditions are better represented in this method by using 1% NaCl in deionized water instead of pure deionized water. The method is performed on 0.16 to 2.48 in. diameter aggregates. In this test, aggregates of a narrow particle size range are soaked in either pure water or 1% NaCl solution at atmospheric pressure for 24 hrs. Aggregates exposed to deicer salts and regular freeze thaw cycles are subjected to 1% NaCl salt solution instead of pure water. The aggregates are then subjected to ten freeze thaw cycles. Table 2.2 shows the quantities of different aggregate sizes in the sample.

Table 2.2: Quantities of different size of aggregates in the sample

Aggregate size, (in.)	Mass or volume of aggregate required	
	Normal aggregate, lb.	Lightweight aggregate (bulk volume), gal
0.16–0.31	2.2	0.13
0.31–0.63	4.4	0.26
0.63–1.26	8.8	0.4
1.26–2.48	13.2	-

The aggregates are thoroughly washed and dried to a constant mass in an oven at 230 ± 9 °F. After cooling, the aggregates are weighed again. In the case of light weight aggregates, the specimens are soaked for 24 ± 1 hr at 68 ± 5.4 °F in water or 1% NaCl solution. The salt solution must be maintained at least 0.39 inches above the aggregates throughout the soaking period. The sample containers should be placed in the freezer so as to not touch each other, with a minimum spacing of 1.97 inches. The samples present in the cabinet are subjected to ten freezing and thawing cycles, with the temperature at the center of the cabinet used as the reference and control temperature.

The aggregate freezing should be from 68 ± 5.4 °F to 32 ± 1.8 °F over a period of 150 ± 30 min. (NTBUILD 485, 2004). The specimens in the cabinet are then maintained at $(32 \text{ to } 30.8)$ °F for 210 ± 30 min. and then further be reduced to 0.5 ± 4.5 °F over a 180 ± 30 minute period. This low temperature should be maintained for at least 240 min. After each cycle of freezing the specimens are subjected to thawing at 68 ± 5.4 °F. The maximum thawing period allowed for this test is 10 hrs. Each freeze thaw cycle spans for 24 hrs and percentage mass loss is calculated by Equation 2.2.

$$F = \left(\frac{w_1 - w_2}{w_1} \right) * 100$$

Equation 2.2

where,

F is the percentage mass loss due to freeze thaw, W_1 is initial dry mass of the three test specimens before cycling, in grams, W_2 is the final dry mass of the three test specimens after cycling that is retained on the specified sieve, in grams (NTBUILD 485, 2004).

2.3.2 EN 1367-1 European standards of freeze thaw testing

The EN 1367-2 test method was adopted to assess the durability of coarse aggregates to freezing and thawing cycles. This method is similar to NTBUILD 485, except that fresh water is used instead of 1 % NaCl. Single sized test aggregates are soaked initially in water at atmospheric pressure. These test aggregate samples are then subjected to 10 freeze thaw cycles which includes cooling to 0.5°F under water and thawing at 68 °F in a water bath (EN 1367-1, 2007). After the end of freeze thaw cycles, the specimens are washed and sieved on and the residue is cooled to constant mass. The mass loss is calculated based on weights obtained by combining the residues from the three test specimens, with the mass of residue obtained expressed as a percentage of the mass of the combined test specimens (EN 1367-1, 2007). The freeze thaw loss (F) is given by Equation 2.3

$$F = ((M_1 - M_2) / M_1) * 100 \qquad \text{Equation 2.3}$$

where,

M_1 is the initial dry total mass of the three test specimens, in grams, M_2 is the final dry total mass of the three test specimens, that is retained on the specified sieve, in grams, F is the percentage loss in mass of the three test specimens after freeze-thaw cycling

2.3.3 Test description - CEN/TC 154 & TC 227 standards in Iceland

Icelandic pavements are subjected to around 100 freezing and thawing cycles every year. De-icing salts are commonly used in urban areas in Iceland. This method was introduced as CEN 154/TG 9 to attempt to improve on EN 1367-1 and be more representative of actual field conditions. The aggregates in this test are subjected to 70 freeze/ thaw cycles, ten cycles per day. The temperature inside the freezing equipment is maintained between 24.8°F to 39.2°F. This method is not commonly used worldwide because it failed to adequately mimic field conditions.

2.3.4 Test description - The Washington Hydraulic Fracture Test (WHFT)

The Washington Hydraulic Fracture test method is a rapid method used to detect D-cracking aggregates. In this method, water is forced into the pores of the oven dried aggregate particles by using a pressurized nitrogen source (Rebecca A., 2003). The compressed air inside the aggregate pores expands and thereby expels water due to a sudden pressure release, creating internal stress. Aggregates whose pore structure is resistant to high pore pressure release are not susceptible to fractures. Freeze thaw durability of the aggregates can be determined by observing the amount of fracturing that occurred on aggregates. This test is inexpensive and consumes lesser time than the most conventional methods. The Washington Hydraulic Fracture Test (WHFT) is used on coarse aggregate particles varying from 0.75” to 1.25”. 2 in. deep by 10 in. diameter containers which can typically accommodate 5.7 to 6.6 lb. of aggregate are used. The aggregate samples are submerged in a water based silane solution for one min (Rebecca A. & Snyder, 2003). Aggregates are then oven dried and those retained on 0.375 in. sieve are used in the test. The aggregates are pressurized using nitrogen at 1150.1 psi, which forces water into the aggregate pores (Rebecca A. & Snyder, 2003). The pressure application is released which increases the internal stresses that are developed due to the compressed air present in the pores. These internal stresses result in particle fractures which may occur during freezing and thawing events (Rebecca A. & Snyder, 2003). The above method is repeated for a total of ten cycles. Aggregate particles are oven dried and sieved over 0.375 in. and 0.18 in. screens. The number of particles and aggregate mass retained over each sieve is determined. The aggregate samples retained on standard sieve 0.375 in. are again subjected to an additional ten cycle of pressurization. The process is repeated for 50 cycles of pressurization. The percentage of fractured particles during each ten cycles of pressurization (Rebecca A. & Snyder, 2003) is given by Equation 2.4.

Estimated percent fractures after i pressurization cycles

$$FP_i = \left(\frac{N_{4i} + N_i - N_0}{N_0} \right) * 100 \quad \text{Equation 2.4}$$

where,

N_{4i} = number of particles passing the 0.375 in sieve and retained on the 0.18 in. [#4] sieve after “ i ” pressurization cycles, N_i = number of particles retained on the 0.375 in sieve, and N_0 = number of particles initially tested.

Hydraulic Fracture Index (HFI) can be defined as number of cycles required producing ten percent fractured aggregates and is given by Equation 2.5.

$$HFI = A + 10 * ((10 - FP_A) / (FP_B - FP_A)) \quad \text{Equation 2.5}$$

Where,

A = number of cycles just prior to achieving 10 percent fracturing, FP_A = percentage of fracturing just prior to achieving 10 percent fracturing (particle mass loss) and FP_B = percentage of fracturing just after achieving 10 percent fracturing.

However, if ten percent fracturing doesn't occur by the end of 50 pressurization cycles then HFI is given by Equation 2.6.

$$(HFI) = 50 * (10 / FP_{50}) \quad \text{Equation 2.6}$$

where,

FP_{50} = percent fracturing after 50 pressurization cycles.

2.3.5 Test description - Modified Hydraulic Fracture Testing Procedure

2.3.5.1 Hydraulic Fracture Testing – Smaller chamber

The Washington Hydraulic Fracture Test (WHFT) Method was modified to better characterize the nature of fracturing of aggregates during the test. The major changes include the use of additional sieves that helps with the additional sieve analysis to predict freeze thaw resistance as a function of mass retained on the various sieves. This modified test method is used to determine the effect of freezing and thawing on aggregate size fractions less than 0.75 in. The aggregate size fractions used in the test method are 0.75 to 1.5 in., 0.5 to 0.75 in. and 0.19 to 0.5 in.

The test procedure using the small chamber method is similar to that used in the WHFT method and explained earlier (Rebecca A. & Snyder, 2003). The original chamber is about 2 in. deep by 10 in. test chamber sufficient to accommodate about 5.7-6.6 lb. while the smaller chamber is used to handle smaller quantities of limited aggregate size fractions. The number of particles and mass of aggregates retained over each screen is measured and the material retained on the 0.18 in. sieve is subjected to 10 cycles of pressurization. 50 cycles of pressurization in total are used. The data for the modified hydraulic fracture test has to be normalized to verify if any variations are present in particle counts and mass retained between individual samples.

Normalization is done by establishing a comparison between mass of particles retained on each sieve after 50 cycles to mass of aggregate sample on each sieve at 0 pressurization cycles (Rebecca A. & Snyder, 2003).

2.3.5.2 Modified Hydraulic Fracture Testing – Large Chamber

In this method, the test chamber capable of accommodating larger quantities of coarse aggregates is employed. Coarse Aggregates are prepared using the 0.75 to 1.5 in. size fraction. The only other difference with the standard WHFT is that replicate tests are not required since because of the larger sample size (Rebecca A. & Snyder, 2003). The normalization procedure for large chamber is similar to that described for small chamber data.

2.3.6 Iowa Pore Index test

The Iowa Pore Index test is used to evaluate the durability of coarse aggregates. In this method, a pressure of 35 psi is applied by opening the air pressure valve to inject water into oven dried aggregates during a period of 1 to 15 min. The amount of water injected into the aggregates is measured. The volume of water absorbed during the first minute is the primary load and the volume intruded during the next fourteen minutes is the secondary load. The Iowa pore index quality number is given by Equation 2.7

$$IQ = \left(\frac{SL}{PL - 10} \right) * V * 0.55 \quad \text{Equation 2.7}$$

where,

IQ is Iowa Pore Index Quality number, SL is Secondary load, PL is Primary load, V is Total volume

This test can effectively identify aggregates with pore system having 0.04-0.2 micron diameter size and relates well to the aggregate service records. This test might give misleading results with nonhomogeneous aggregate samples. Tests conducted using the Iowa Pore Index Test method indicate that D cracking is generally found in aggregates which are fine grained and durable aggregates are either coarse grained or extremely fine grained (Dubberke, W., 1998).

2.3.7 Coarse aggregate freeze-thaw test TX DOT designation: TEX-432-A

TEX 432-A method is used to measure the resistance of coarse aggregates to degradation by freezing and thawing, thereby determining the soundness of aggregates subjected to weathering. In this method aggregates are sieved using the weights of the individual size fractions shown in the Table 2.3.

Table 2.3: Weights of individual aggregate size fractions (lb.)

Weight of individual sizes (lb.)					
Size of aggregate		Test grade			
Passing (in.)	Retained(in.)	A	B	C	D
0.75	0.63	0.88 ± .02	0.55 ± .02		
0.63	0.5	0.55 ± .02	0.55 ± .02		
0.5	0.375	0.44 ± .02	0.44 ± .02	0.44 ± .02	
0.375	0.187	0.22 ± 0.01	0.22 ± 0.01	0.22 ± 0.01	0.22 ± 0.01
0.187 (No.4)	0.09 (No.8)	0.066 ± 0.01		0.066 ± 0.01	0.066 ± 0.01

Aggregates are initially soaked in trays for 24 hrs and then subjected to 2hrs of freezing at 15°F. The thawing cycle is done at room temperature until there is no evidence of ice in the water. The aggregates are then subjected to 50 freezing and thawing cycles after which the aggregates are then dried and weighed. The percentage loss for each size fraction is calculated as showed in Equation 2.3.

2.3.8 NDR standard method T 103 soundness of aggregates by freezing and thawing

The NDR standard method is used to determine the resistance of aggregates to degradation by subjecting aggregate to freeze thaw cycles. This method is helpful in measuring the soundness of aggregates. In this method, the aggregates are frozen up to -15°F for 90 minutes and thawed for 30 minutes in a room temperature (21-27°F) tank of 0.5% methyl alcohol. After sixteen cycles of freezing and thawing, the samples are oven dried at 230 ± 9°F to constant

weight and the samples are sieved through a number 8 sieve. The percent passing through number 8 sieve is calculated as percent loss which is an indicator of freeze thaw durability.

2.3.9 German standard DIN 4226 (1971) freeze thaw testing of aggregates

German standard DIN 4226 specifies two alternative procedures for testing aggregate freeze-thaw durability, Freezing and thawing under water and Air freezing followed by thawing in water.

Total immersion, freezing & thawing under water:

In this method the immersed aggregate samples are subjected to a reduction in temperature over a period of 7 to 10 hrs. The samples are held at a temperature of -4°F to 5°F for at least 4 hrs. The samples are then thawed for 5 hrs in water at 68°F. After 10 freezing and thawing cycles, each aggregate size is oven dried and sieved on the next smaller sieve size. The percentage mass loss is then calculated.

Air freezing followed by thawing in water:

Aggregates that are to be tested using the alternative procedure using air freezing are initially soaked for two hrs. After two hrs of soaking the aggregates are drained thoroughly and are placed in a freezing cabinet at a temperature of -4°F to 5°F for six hrs. The aggregates are then removed from the freezing cabinet and thawed in water at 68°F for one hour. The aggregates are oven dried and sieved after 20 freezing and thawing cycles.

2.3.10 CSA A23.2-24A Test Method for unconfined coarse aggregate to freezing & thawing

The CSA A23.2-24A test method was developed by University Of Windsor in association with Ministry of Transportation Ontario. In this method, samples are sieved, with the mass of each aggregate sieve size shown in Table 2.4.

Table 2.4: Quantities of different sizes present in the sample

Weights of test sample		
Passing, in.	Retained, in.	Mass, lb.
1.5	1	11
1	0.75	5.5
0.75	0.5	2.8
0.5	0.375	2.2
0.375	0.2	1.1

Each individual aggregate size is separately soaked in a 3% NaCl solution for 24 hours. The aggregates are then drained and frozen at -0.4 ± 3.6 °F for 16 ± 2 hours, followed by 8 ± 1 hours of thawing at room temperature. After 5 freezing and thawing cycles, the aggregates are dried, sieved and weighed. Studies indicated that results from Canadian freeze thaw test are better than magnesium sulfate soundness test to evaluate aggregates for concrete.

Aggregates are placed in containers such that aggregates coarser than 0.75 in. are placed in two containers so that the entire amount is tested. 3 % NaCl solution is filled in the container in such a way that all aggregates are completely immersed in the salt solution. The containers are sealed to prevent evaporation and are kept at room temperature for 24 ± 2 hrs. After one day of soaking the containers are drained off by using a 0.197 in. mesh. The containers are sealed to maintain 100 percent humidity condition. The containers are arranged in baskets in such a way that spacers are installed in between containers to prevent contact. The baskets are then placed in a freezer at -0.4 ± 3.6 °F for $16 \text{ hrs} \pm 2$ hrs. After removing the aggregate containers from the freezer, they are allowed to thaw for 8 ± 1 hr at room temperature. After each thawing period, all aggregate containers are turned one quarter turn and are subjected to freezing. After five cycles of freezing and thawing, the aggregates are washed with tap water five times. The water present in the container is drained, after which the aggregates are oven dried to constant mass at 230 ± 9 °F. Each aggregate is placed on the same sieve used for the sample preparation and is sieved for 3 min. The weight of aggregate retained on each sieve is recorded. The percentage mass loss on each sieve due to freeze thaw cycles is calculated according to Equation 2.8.

$$F = (\Sigma(M_0 - M_f)) * 100 / (M_0)$$

Equation 2.8

where, F is the aggregate percentage loss for that aggregate size fraction, M_0 is the aggregate size fraction weight before freezing, and M_f is the mass at the original aggregate size fraction after the freezing and thawing cycles.

A set of control aggregates should be tested with each group of aggregate sets tested. Any problems with the freezing and thawing process will be apparent in the mass loss values found with the control aggregate. The Ministry of Transportation Ontario maintains a stockpile of control aggregates.

Chapter 3 - Methods & Materials Used

The main objective of this project is to determine if the CSA A23.2-24A test method for the resistance of unconfined coarse aggregate to freezing and thawing can be used as a rapid and more accurate method than currently used procedures in determining the freezing and thawing durability of aggregates in concrete. This study aims to correlate the results of the CSA A23.2-24A test method to the current aggregate test methods. To accomplish this, aggregates previously tested by KDOT using the current KDOT aggregate qualification methods were tested using the CSA A23.2-24A method.

The current KDOT aggregate qualification tests include the following standards:

- The KTMR-21 Soundness and Modified Soundness of Aggregates by Freezing and Thawing Test Method (KTMR-21, 2007) is used to test the freezing and thawing resistance of bare aggregates. 25 freezing and thawing cycles are conducted on aggregates and durable aggregates are selected based on the assumption that the cumulative mass of coarse aggregates after freezing and thawing cycles must be above 85% of the initial cumulative mass of aggregates greater than the No.8 sieve before freezing.
- The KTMR-22 test method is a modified version of the ASTM C 666 method B rapid concrete freezing and thawing test. The ASTM C 666 test is modified to include a 90 day curing period and is used as the final performance test for use of limestone aggregates in Kansas concrete pavements. The KTMR-22 test method can take up to six months to complete. The current tests are used for preliminary aggregate qualification pending the results of the KTMR-22 test.
- The KTMR-25 test method is used for testing the abrasion and impact resistance of coarse aggregates using the Los Angeles testing machine. In this test, sizes of coarse aggregate smaller than 1.5 in. are tested for resistance to degradation by impact and abrasion using the Los Angeles testing machine. The test method is similar to AASHTO T 96, except that the fines and impurities such as clay and woody material are removed before testing.

- The KTMR-28 method is used to determine the total amount of acid insoluble residue of limestone or dolomite aggregates. In this method, the carbonate fraction of the aggregate is dissolved in hydrochloric acid, after which the sample is filtered to collect and weigh the residue.

A rapid test that better correlates with the results of the KTMR-22 test method would help prevent some poor performing aggregates from being used in concrete pavements pending the outcome of the longer duration KTMR-22 test. The CSA A23.2-24A test method for the resistance of unconfined coarse aggregate to freezing and thawing, was developed as such a rapid test method to screen aggregates for freeze-thaw durability and was chosen for this study for comparison to the currently used test methods. Aggregates were tested for specific gravity and absorption using the KTMR-27 test method for comparison with KDOT results.

3.1 KTMR-27 Modified Specific Gravity and Absorption of aggregate test method

Aggregates were tested for specific gravity and absorption (%) for comparison with the KDOT values obtained. The KTMR-27 test method is similar to the AASHTO T85 procedure, used to determine the specific gravity and absorption of aggregates. The main difference between these two methods is that the aggregates are soaked for 24±4 hrs in the KTMR-27 method, whereas the aggregates are soaked for 17 ±1 hr before measuring the saturated surface dry weight in the AASHTO T85 method.

The aggregates tested using the KTMR-27 test method were initially sieved, washed and dried. Each sample was then recombined and weighed to give 5 lb. of sample passing the ¾ in. sieve and retained on the ½ in. sieve and 5 lb. of sample passing the ½ in. sieve and retained on the 3/8 in. sieve. The aggregates were soaked in water for 24±4 hrs and then brought to a saturated surface dry (SSD) condition by drying the aggregates with a towel by hand, until the free water was removed from the aggregate surface. Aggregates were then re-immersed in a water bath as shown in Figure 3-1 at 77 ± 1.8°F and were stirred to eliminate entrapped air and weighed. The sample was then dried to a constant mass at 230 ± 9°F. The weight was recorded after the sample cooled to room temperature.

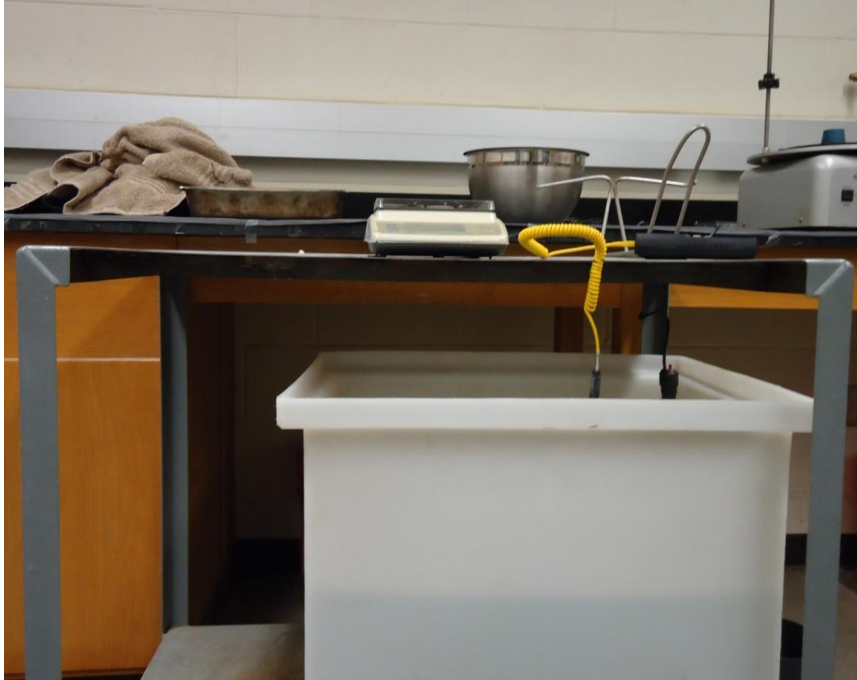


Figure 3-1: Apparatus for performing the KTMR-27 test method

Specific Gravity and Absorption (%) were calculated using Equation 3.1 and 3.2, for different KDOT aggregates and were compared to KDOT values.

$$\text{Bulk Specific Gravity (BSG)} = \frac{A}{B - C} \quad \text{Equation 3.1}$$

$$\text{Absorption (\%)} = \frac{(B - A) * 100}{A} \quad \text{Equation 3.2}$$

where, A is the mass of oven dried Sample in air (lb.), B is the mass of saturated surface dry sample in air (lb.) and C is the mass of saturated sample in water (lb.).

3.2 Canadian freeze thaw testing CSA A23.2-24A (laboratory testing)

3.2.1 CSA A23.2-24A test procedure

The CSA A23.2–24A test method was developed by the University of Windsor, in association with the Ministry of Transportation Ontario (MTO). Aggregates were exposed to a salt solution and then subjected to five unconfined freezing and thawing cycles. After the freezing and thawing cycles, the aggregates were re-sieved, with the mass loss of each aggregate

size determined. In the CSA A23.2-24A test method, aggregates were sieved, with each size aggregate tested in separate containers. Aggregates retained on the 0.2 in. sieve were tested and pre-sieved using a sieve shaker. Ledge aggregate samples were separated into fractions and weighed according to Table 3.1.

Table 3.1: Sample weights needed for different aggregate size fractions, (CSA, 2004)

Weights of test samples		
Aggregate size		Weight, lb.
Passing, in.	Retained, in.	
1.5	1	11
1	0.75	5.5
0.75	0.5	2.8
0.5	0.375	2.2
0.375	0.25	1.1

Each aggregates size was placed in plastic autoclavable a container that was then filled with 3% by mass of NaCl solution for 24 ± 2 hours at room temperature. The solution inside each container was rapidly drained off by inverting the container while covered with a mesh with openings smaller than 0.2 inches for about 5 sec. All the containers were then sealed thoroughly to ensure 100 % humidity and were arranged in trays with wooden spacers in between each container. This was done to ensure that no two containers touched each other. These trays were placed in a chest freezer at -0.4 ± 3.6 °F for 16 ± 2 hrs. followed by thawing for 8 ± 1 hr at room temperature. All containers were turned one quarter turn between each cycle of freezing and thawing. After five cycles of freezing and thawing, the aggregates were washed with tap water five times, drained and oven dried to a constant mass at 230 ± 9 °F. Each aggregate set was placed on the same sieve used for the sieve analysis. The percentage mass loss on each sieve due to freeze thaw cycles was calculated using Equation 2.8:

3.2.2 Materials tested

Fifty one KDOT aggregates (including 12 ledge and 39 production samples) were tested using the CSA A23.2-24A method. Production samples were sieved and weighed according to

Table 3.2. Kansas limestone production samples only contain aggregates less than $\frac{3}{4}$ in. the CSA A23.2-24A test method, which necessitated a modification in the methodology to accommodate the smaller sizes.

Table 3.2: Required mass of aggregates (gm) separated into different fractions (for production samples)

Weights of test sample		
Passing, in.	Retained, in.	Weight, lb.
0.75	0.5	5.5
0.5	0.375	4.4
0.375	0.25	2.2

3.2.3 Locally available control aggregate

CSA A23.2-24A specifies to test a control aggregate alongside the aggregates of interest during testing. This requirement was included in the specification to detect any biases or abnormality in freezing during the testing from power loss or mechanical problems that might have otherwise been undetected. The control aggregate was obtained from the Ministry of Transportation Ontario (MTO), however only enough of the Canadian reference aggregate from Brenchin quarry no. 2 was obtained to develop a new locally available limestone control aggregate, supplied by Midwest Concrete Materials (M.C.M). Table 3.3 shows the material properties for the Canadian reference aggregate obtained from the MTO.

Table 3.3: Data for Canadian reference aggregate (unpublished data, MTO 2009)

Brenchin quarry No. 2 (Canadian reference aggregate)		
Test	Mean loss (%)	Range (%)
Micro Deval Abrasion	19.1	17.5-20.7
Unconfined freeze thaw test CSA (A23.2-24A)	15.6	10.2-20.9
Sulfate soundness test	13.2	8-18.4
Specific Gravity	2.67	2.658-2.682

The average and standard deviation of freeze thaw mass loss for MTO aggregate and local aggregates are shown in Table 3.4.

Table 3.4: Mean and standard deviation of freeze thaw mass loss for MTO aggregate local aggregate.

Aggregate type	No. of sets	Mean	Standard Deviation
MTO reference aggregate	6	15.51	0.584
Local limestone aggregate	12	15.1	2.09

The mean loss for MTO reference aggregate (15.51%) compared very favorably with the mean loss (15.6 %) result by the MTO (15.6%).

Local aggregates were sieved to the same size as described in Table 3.4 and were tested alongside the KDOT aggregates. Three sets of the Canadian standard aggregate were run and then sieved at one minute intervals to determine what sieve time in the KSU sieve shaker would correspond with the three minutes of sieve time used at MTO on the standard aggregate. The cumulative percent freeze thaw loss for each fraction of local standard aggregate was compared to the average of 3 sets of Canadian standard aggregate as shown in Figure 3-2, Figure 3-3 and Figure 3-4. Figure 3-5 shows a comparison of the combined freeze thaw loss (%) from all three sizes vs. sieving time (min) for average of six Canadian aggregate sets and twelve limestone aggregate sets. It was found that 3 minutes of sieving with the KSU sieving equipment for the Canadian control aggregate yielded very similar results to that obtained by the MTO.

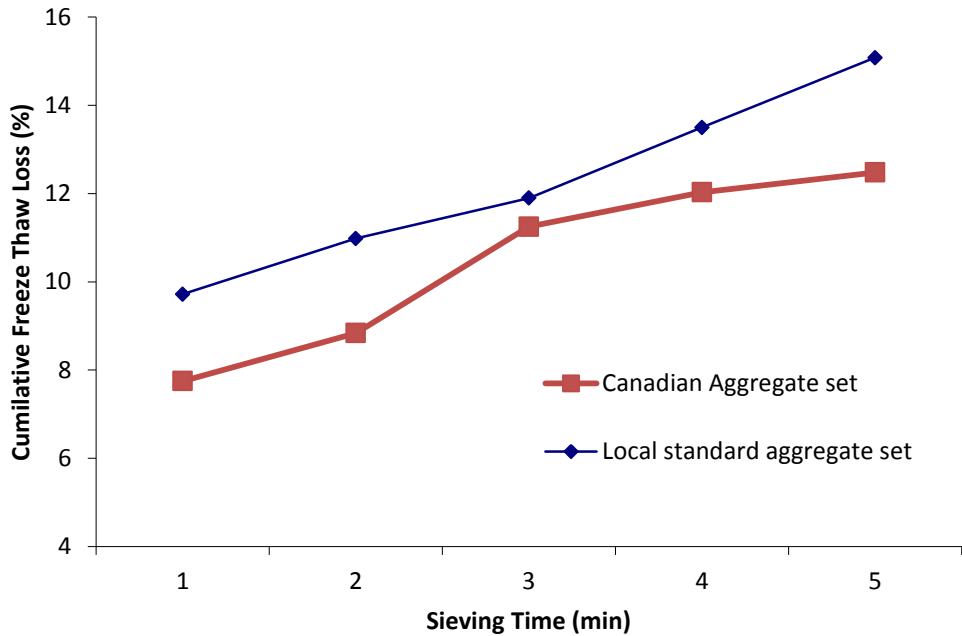


Figure 3-2: Cumulative Freeze Thaw loss vs. Sieving Time (min) for limestone aggregates and average of three Canadian aggregates sets on 0.75”-0.5” fraction.

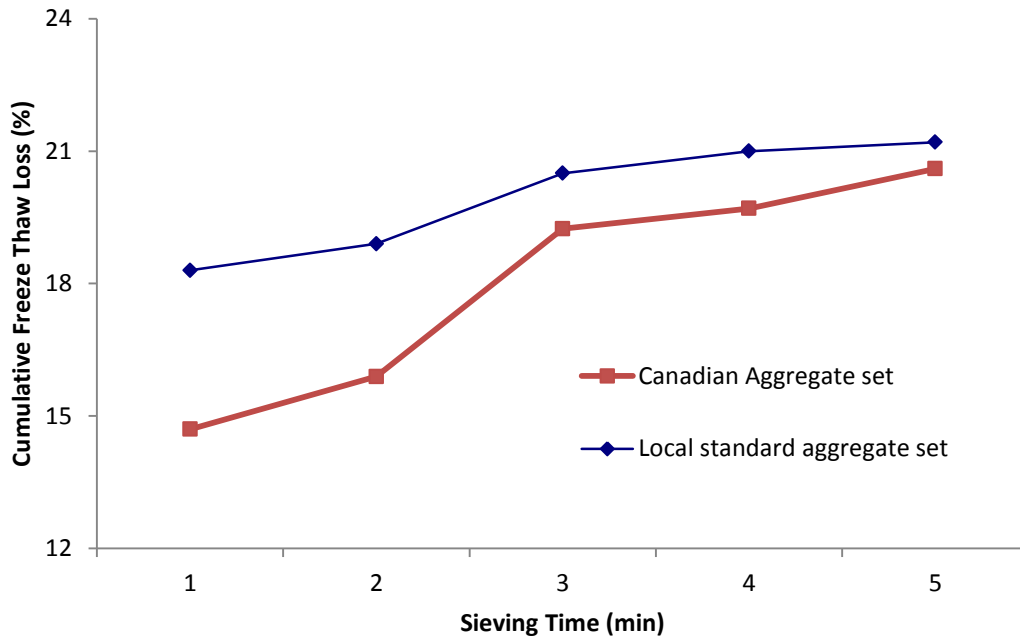


Figure 3-3: Cumulative Freeze Thaw Loss vs. Sieving Time (min) for the local control limestone aggregate and the average of three Canadian aggregates sets on 0.5”-0.375” fraction.

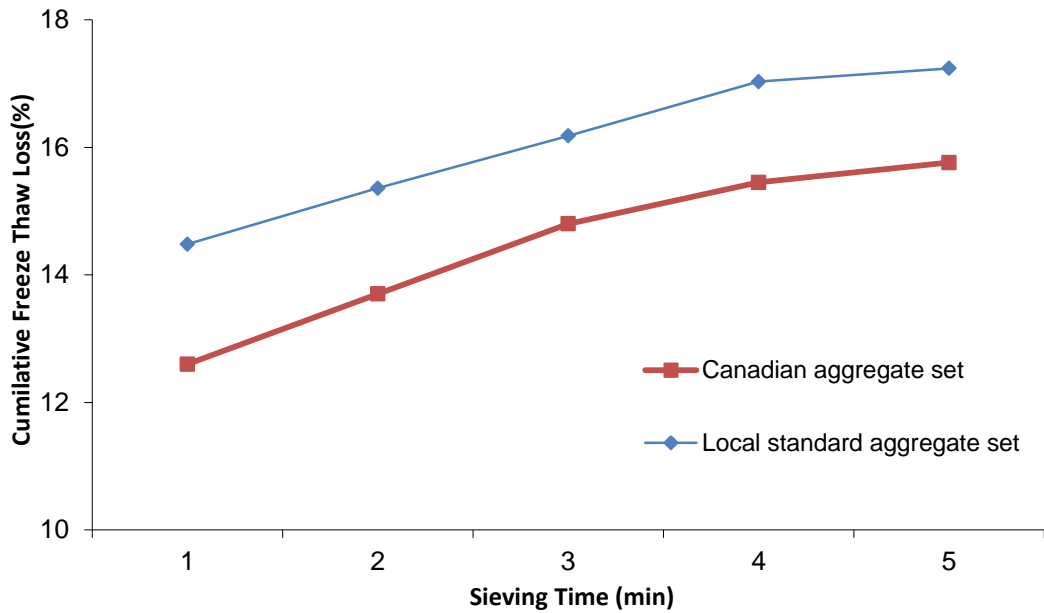


Figure 3-4: Cumulative Freeze Thaw Loss (%) vs. Sieving Time (min) for limestone aggregates and the average of three Canadian aggregate sets on 0.375”-0.25” fraction.

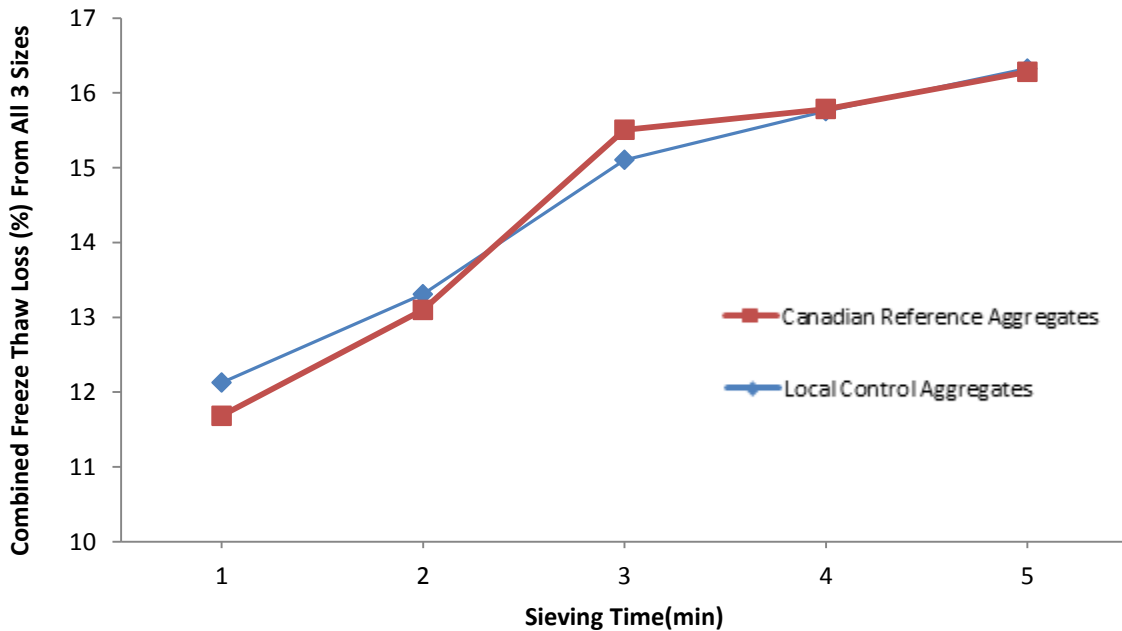


Figure 3-5: Comparison of Combined Freeze Thaw Loss (%) from all three sizes vs. Sieving Time (min) for average of six Canadian aggregate sets and twelve local control limestone aggregate sets.

3.2.4 Modifications for particle size to accommodate production samples

The CSA A23.2-24A test method requires the use of aggregates as large as 1.5 in. Because Kansas production limestone samples only contain aggregates below $\frac{3}{4}$ in., the test method was modified to use smaller aggregate sizes. Ledge samples were still tested using aggregates up to 1.5 in. in size, however in order to test if the larger aggregates that show more susceptibility to D-cracking in concrete also show more susceptibility to freezing and thawing damage in the CSA A23.2-24A test method. For the production version of the same aggregate source as the ledge samples, larger aggregates from the ledge samples were crushed and sieved to give enough of the smaller size particles as shown in Table 3.2. Aggregates between $\frac{1}{2}$ in. and $\frac{3}{4}$ in. for the production samples were placed in two separate one-liter autoclavable containers. The production sample aggregates between $\frac{3}{8}$ in. and $\frac{1}{2}$ in. for the production samples were placed in one one-liter autoclavable container, and the production sample aggregates between $\frac{1}{4}$ in. and $\frac{3}{8}$ in. were placed in one 500 mL autoclavable bottle for testing.

3.2.5 Effects of salt type

The CSA A23.3-24A test method was modified to use 3% by weight MgCl_2 and CaCl_2 salt solutions. This method was used to determine if aggregate loss during freeze and thaw cycles in the presence of MgCl_2 or CaCl_2 salts would better correlate with the KTMR-22 test method. While the amount of aggregates obtained for each quarry was not sufficient to allow for testing both MgCl_2 and CaCl_2 on all aggregates, MgCl_2 was used on nine of the ledge samples, while CaCl_2 was also used on seven production aggregate samples to assess the effect of different salts on aggregates.

3.3 BET Nitrogen Adsorption

3.3.1 Introduction

The Brunauer Emmett Teller (BET) method was employed to calculate specific surface areas from measurements of physical adsorption of gas molecules (Brunauer, Emmett, E, & Teller, 1938). The adsorption process creates a thin film of molecules or atoms on the surface of the adsorbent. The adsorbate (nitrogen in this case) gets attracted to the adsorbent pore surface. Gas molecules adsorb onto the solid surfaces of different size pores at different pressures as shown in Figure 3-6, which can be used in determining the specific surface area of a solid.

Nitrogen molecules condensing on the pore wall

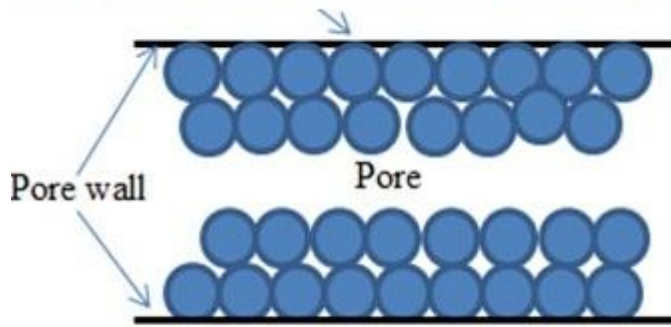


Figure 3-6: Gas molecules adsorbed on to the surface

The BET calculations assume that the top layer atoms adsorbed to the pore surface are in equilibrium with the nitrogen vapor. The BET equations are the most widely used methods for calculating the surface area of solid materials from the volume adsorbed at different vapor pressures as shown in Equation 3.4:

$$\frac{1}{W * \left(\left(\frac{P_0}{P} \right) - 1 \right)} = \left(\frac{1}{W_m * C} \right) + \left(\frac{C - 1}{W_m * C} \right) * \left(\frac{P}{P_0} \right) \quad \text{Equation 3.4}$$

where, W is the weight of gas adsorbed at a relative pressure, P/P_0 , W_m is the weight of adsorbate constituting a monolayer of surface coverage.

C is the BET constant, which is related to the energy of adsorption in the first adsorbed layer. Consequently the C value is an indication of the magnitude of the interactions between adsorbent and adsorbate. The C value greatly affects the adsorbate cross sectional area. Nitrogen is the most widely used gas for surface area determination due to its C values, which varies between 50 and 300 (Lowell, Shields, Charalambous, & Manzione, 1982). Very high C values produce significant errors in calculating cross sectional area, making nitrogen an excellent adsorbate for cement pastes and aggregates (Lowell, Shields, Charalambous, & Manzione, 1982). Multi-point BET method is preferred to single point BET method for calculating surface area because of the reduced error. Multi-point BET measurements were used in this study. Figure 3-7 shows B.E.T Autosorb 1 test apparatus used in this study.



Figure 3-7: B.E.T Autosorb-1 test apparatus

Adsorption isotherms are obtained when gas pressures in the sample are increased whereas the desorption process occurs when quantities of gas are removed from the sample, with the fall in relative pressures. The Barrett Joyner and Halenda (BJH) method was used to compute the aggregates pore size distributions (Barrett, Joyner, & Halenda, 1951). BJH works on a principle that when relative pressure is lowered, the volume will desorb from the surface, reducing the thickness of the physically adsorbed gas layer. The theory assumes that all pores are filled with liquid initially and the initial relative pressure (P/P_0) is close to unity. The relationship between pore volume V_{p1} and volume V_1 , which is caused due to change in relative pressure, is given by Equation 3.5.

$$V_{p1} = V_1 \left(\frac{(r_{p1})}{\left(r_{k1} + \frac{\Delta t_1}{2}\right)} \right)^2 \quad \text{Equation 3.5}$$

where, V_{p1} is the pore volume, r_{p1} is the largest pore radius, Δt_1 is the reduction in thickness, and r_{k1} is the inner capillary radius.

The pore volume V_{pn} is given by Equation 3.6:

$$V_{pn} = \left(\frac{(r_{pn})}{\left(r_{kn} + \frac{\Delta t_n}{2} \right)} \right)^2 * (\Delta V_n - \left(\Delta t_n * \sum_{j=1}^{n-1} A_{cj} \right)) \quad \text{Equation 3.6}$$

where A_{cj} is the area exposed by the previously emptied pores from which the gas is desorbed. The area of each pore (A_p) is calculated individually as shown in Equation 3.7 and is further summed up for any step in the desorption process.

$$A_p = 2V_p - r_{pn} \quad \text{Equation 3.7}$$

It is assumed that all pores that are emptied during the pressure reduction have an average radius r_p , the C value is used for computation of pore size distribution and is calculated using Equation 3.8:

$$C = (r_p - t_r)/r_p \quad \text{Equation 3.8}$$

where, t_r is thickness of the adsorbed layer.

3.3.2 Procedure

Aggregates were crushed to a diameter below 0.31 in. (which is the diameter of the bulb), so that the sample was small enough to fit in the bulb as shown in Figure 3-8. The initial sample weight was obtained by subtracting the weight of the empty bulb with plug (W_1 gm) from the weight of the sample with the bulb and plug (W_2 gm).

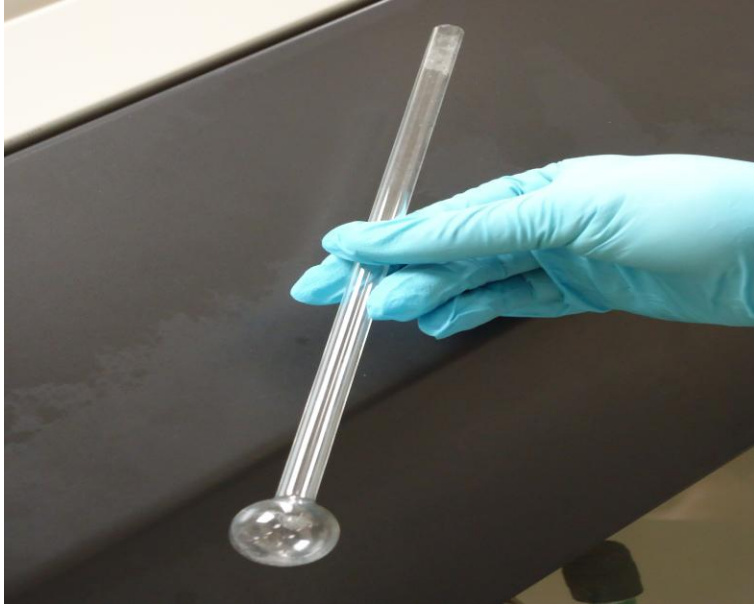


Figure 3-8: Bulb into which the sample should go in.

After the bulb is attached to the degasser using the attachments shown in Figure 3-9, the bulb is placed into the insulating bag and secured to the Autosorb 1 apparatus.



Figure 3-9: Final arrangement of the bulb.

Figure 3-10 shows the arrangement of the glass bulb in the insulating heat bag. Heating was done under vacuum or continuously flowing inert gas, to precondition the sample in order to ensure that all of the physically bonded impurities such as moisture were removed before testing. Figure 3-11 shows the sample in the heating bag during outgassing.



Figure 3-10: Bulb placed into the insulating heat bag.



Figure 3-11: Final arrangement of bulb before out gassing.

An outgassing temperature of 176°F (preferably as low as possible) was used. After outgassing the sample for 4 hrs, the heater in the instrument panel was turned off and the sample was allowed to cool at room temperature. The bulb was detached from the apparatus, after which the weight of the sample after out gassing was measured to make sure that there were no detectable physical and chemical changes in the test samples. The true weight of the sample (original weight of sample without any existing vapors and gases adsorbed on to the surface) was calculated by subtracting the recorded weight (after out gassing) 'W₃' gm. from the initial weight 'W₁' gm. After degassing, the sample was attached to the apparatus for the nitrogen to be introduced. A dewar flask containing liquid nitrogen was placed around the sample before starting the nitrogen gas inflow, as shown in Figure 3-12.

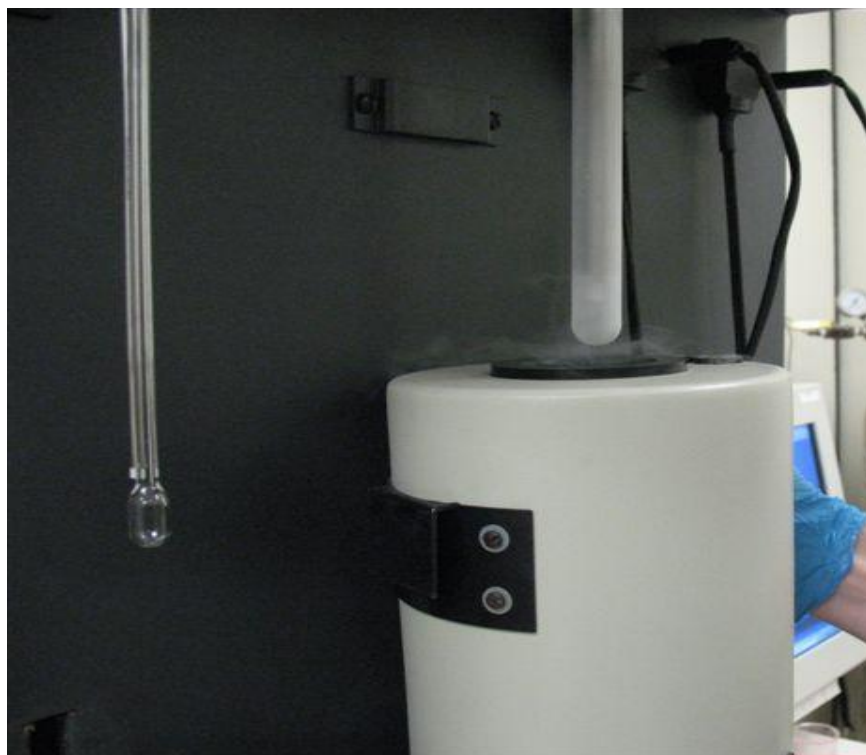


Figure 3-12: Dewar being lifted up into its slot after filling up with Nitrogen.

After fixing the dewar in its slot the metallic parts are arranged as described in Figure 3.9, after which the bulb is threaded into the slot before the thermistor and calibration bulb.

3.3.3 Results & calculations

The specific area can be seen in the BET plot. Example of a sample BET plot for one aggregate sample tested is shown in Figure 3-13. The area of each pore (A_p) is calculated Equation 3.9:

$$A_p = 2 * \frac{V_p}{r_p} * 10000 \quad \text{Equation 3.9}$$

The cumulative pore area is obtained by summing up the A_p values. The total sum of areas shouldn't exceed BET area shown in the Figure 3-13.

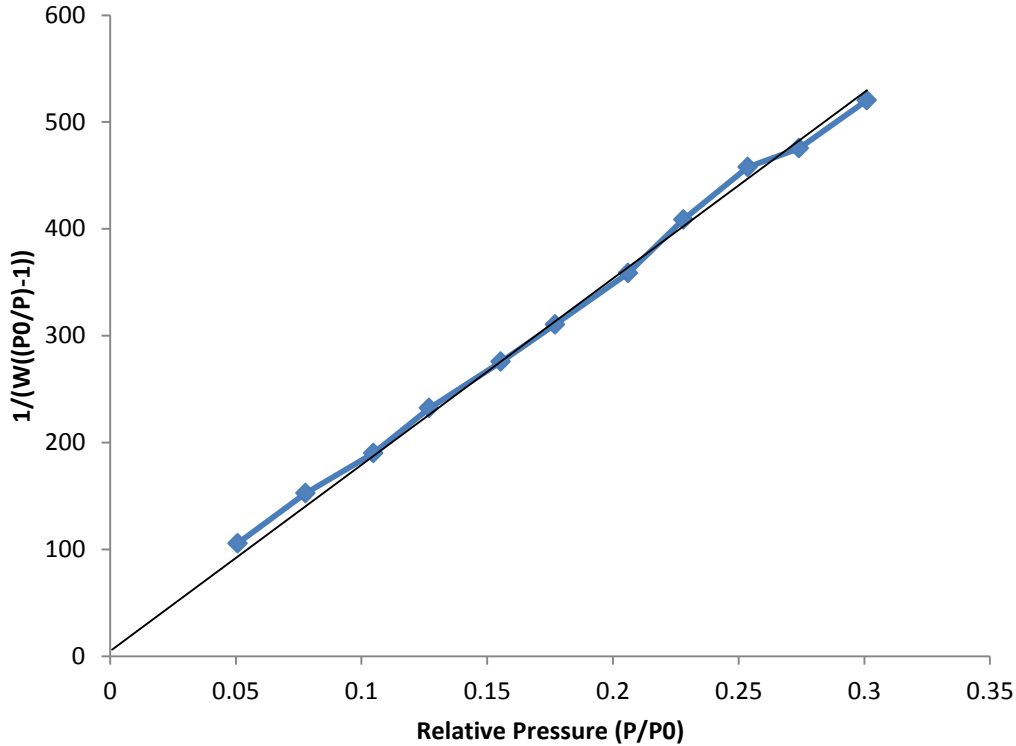


Figure 3-13: BET plot for KDOT limestone aggregate with area below the graph.

Chapter 4 -Results & Discussions

This chapter reports result from Canadian freeze thaw testing using CSA A23.2-24A test method on ledge and production samples supplied by KDOT and nitrogen adsorption testing on a subset of aggregates. The results of these experiments are compared to the standard aggregate qualification tests run by KDOT to determine if the CSA A23.2-24A test method could be used as a more rapid substitute for the currently run tests. For the CSA A23.2-24A experiments, a modified version of the CSA A23.2-24A test method was used on production samples to allow for the testing of the smaller size aggregates. The CSA A23.2-24A method was also modified by using CaCl₂ or MgCl₂ solutions to investigate the impact of the salt solution on the aggregate durability.

4.1 Specific Gravity and Absorption results using KTMR-27 test procedure

Specific gravity and absorption were determined using the KTMR-27 procedure. Each coarse aggregate composite sample was tested for specific gravity and absorption for quality control purposes. Table 4.1 shows specific gravity and absorption values for ledge samples. Table 4.2 shows specific gravity and absorption values for production samples. The measured absorption and specific gravity results compared well to the values measured by KDOT.

Table 4.1: Specific Gravity and Absorption values for ledge samples

KDOT lab id	BED	Bulk Specific Gravity	Bulk Specific Gravity (in SSD)	Apparent Specific Gravity	Absorption (%)	KDOT test results	
						BSG	Absorption
09-1468	8	2.59	2.63	2.7	1.53	2.63	3.00
09-1468	9	2.44	2.51	2.62	2.81	2.52	2.80
09-1469	1	2.52	2.6	2.75	3.36	2.6	1.40
09-1469	2	2.48	2.55	2.67	2.87	2.48	2.80
09-1884	1	2.42	2.54	2.75	4.88	2.56	1.89
09-1884	3	2.43	2.55	2.78	5.07	2.51	2.84
09-1885	1	2.55	2.59	2.68	1.96	2.58	1.80
09-1939	.	2.6	2.62	2.65	0.62	.	.
09-1940	.	2.61	2.64	2.66	0.56	.	.
09-1474	1	2.57	2.62	2.65	1.90	2.57	1.95
09-3051	2	2.47	2.55	2.59	3.10	2.47	3.00
09-3051	3	2.5	2.57	2.61	3.00	2.50	3.10

Table 4.2: Specific Gravity and Absorption values for production samples

KDOT lab id	Bed	Bulk Specific Gravity	Bulk Specific Gravity (in SSD)	Apparent Specific Gravity	Absorption (%)	KDOT test results	
						BSG	Absorption(%)
09-1008	1	2.45	2.52	2.65	3.21	2.44	3.60
09-1010	1	2.50	2.55	2.64	2.17	2.50	2.60
09-1227	1	2.54	2.60	2.71	2.50	2.57	2.00
09-1228	1	2.58	2.62	2.68	1.45	2.58	1.80
09-1231	1	2.47	2.55	2.68	3.14	2.51	2.76
09-1248	4	2.48	2.57	2.72	3.60	2.48	3.60
09-1257	1	2.59	2.63	2.69	1.50	2.59	1.50
09-1430	1	2.54	2.58	2.64	1.52	2.55	1.80
09-1454	1	2.57	2.63	2.72	2.10	2.6	1.60
09-1706	2	2.48	2.55	2.66	2.73	2.48	3.20
09-1917	5	2.53	2.58	2.66	1.92	2.52	2.60
09-1918	4	2.48	2.56	2.68	2.98	2.5	2.50
09-2257	1	2.58	2.63	2.72	2.06	2.59	1.60
09-2102	4	2.50	2.77	2.69	2.90	2.5	2.90
09-2943	5	2.52	2.58	2.68	2.40	2.52	2.94
09-2788	4	2.49	2.57	2.7	3.00	2.5	3.00
09-3497		2.45	2.52	2.58	2.50	2.53	2.2
09-3645		2.58	2.62	2.67	3.20	.	.
09-3453	3	2.62	2.63	2.68	1.20	2.54	2.30
10-0354	C	2.49	2.58	2.63	4.00	1.6	2.60
08-2058	1	2.55	2.60	2.64	1.80	2.57	3.10
09-2642	2	2.68	2.72	2.78	3.40	2.61	1.50
09-3453	4	2.54	2.59	2.65	2.70	2.55	2.60
08-355		2.65	2.71	2.74	1.80	2.57	2.20
08-2323		2.58	2.63	2.66	4.20	2.51	3.60
09-2642	1	2.50	2.61	2.67	2.80	2.46	3.0
10-0211	2	2.55	2.58	2.63	2.71	2.63	1.60
10-0424	1	2.63	2.67	2.685	2.00	2.49	2.80

The KTMR-27 test method was also used to test specific gravity and absorption values for the Canadian standard aggregate and the local control aggregate used in the project. Table 4.3 shows the specific gravity and absorption values for Canadian aggregates along with one set of local aggregates.

Table 4.3: Specific gravity and absorption values for Canadian aggregates along with one set of local control aggregates

Specific Gravity and Absorption values for Canadian reference aggregates				
Aggregate Type	Bulk Specific Gravity	Bulk Specific Gravity (in SSD)	Apparent Specific Gravity	Absorption %
Canadian aggregates	2.53	2.58	2.67	2.17
Local control aggregates	2.54	2.59	2.67	1.97

4.2 Comparison of original to the modified version of CSA A23.2-24A test method

Ledge and production samples were tested using the CSA A23.3-24A test method. Kansas production limestone aggregates do not contain sufficient quantity of large coarse aggregates to run the original CSA A23.2-24A standard test method. The Canadian standard aggregate likewise does not contain the large aggregate sizes. The standard allows for testing the Canadian standard aggregates using aggregates smaller than $\frac{3}{4}$ ". A modified version of the CSA A23.2-24A test method was used on 39 production samples supplied by KDOT, with ten of the aggregate sets tested using the same smaller size fractions from the ledge samples that were used with the rest of the production samples. One of the most important things that can be observed from the freeze thaw testing was that the aggregates performed similar in the testing, independent of the size fraction tested. Figure 4-1 shows a comparison of the 0.75-0.5 in. and 0.375-0.25 in. aggregate weight loss for all aggregates sets tested after three minutes of sieving, while Figure 4-2 shows a comparison of the 1.5-1 in. and 0.375-0.25 in. aggregate weight loss for the ledge samples tested, Figure 4-3 shows a comparison of the 1-0.75 in. and the 0.375 - 0.25 in. aggregate size fraction weight loss for all ledge samples aggregate sets tested after three minutes of sieving, Figure 4-4 shows a comparison of the 0.75-0.5 in. and 0.375-0.25 in. aggregate weight loss for all aggregates sets tested after three minutes of sieving.

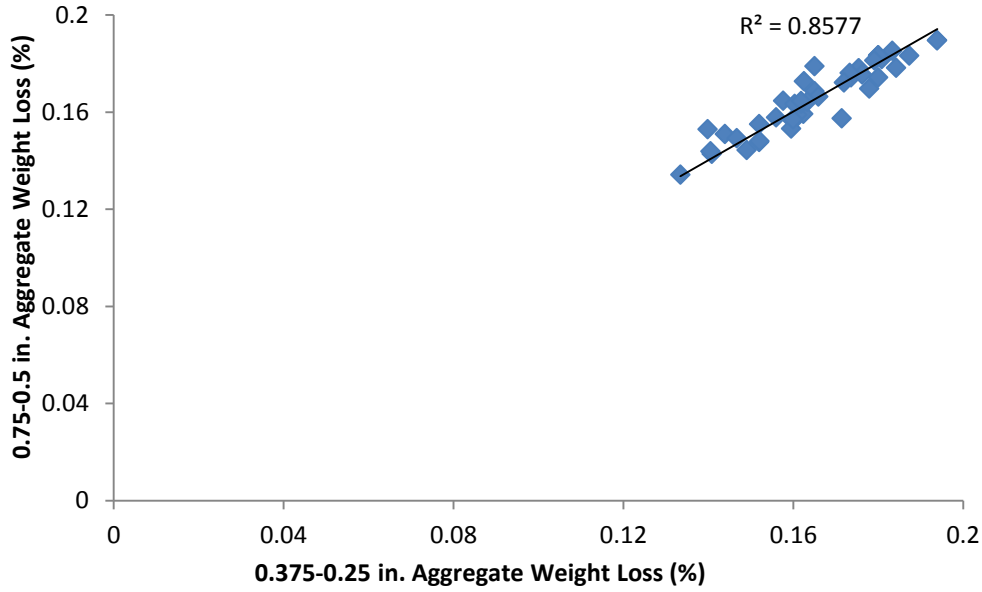


Figure 4-1: Comparison of aggregate weight loss (%) for the 0.75-0.5 in. aggregates and the 0.375 - 0.25 in. aggregates tested.

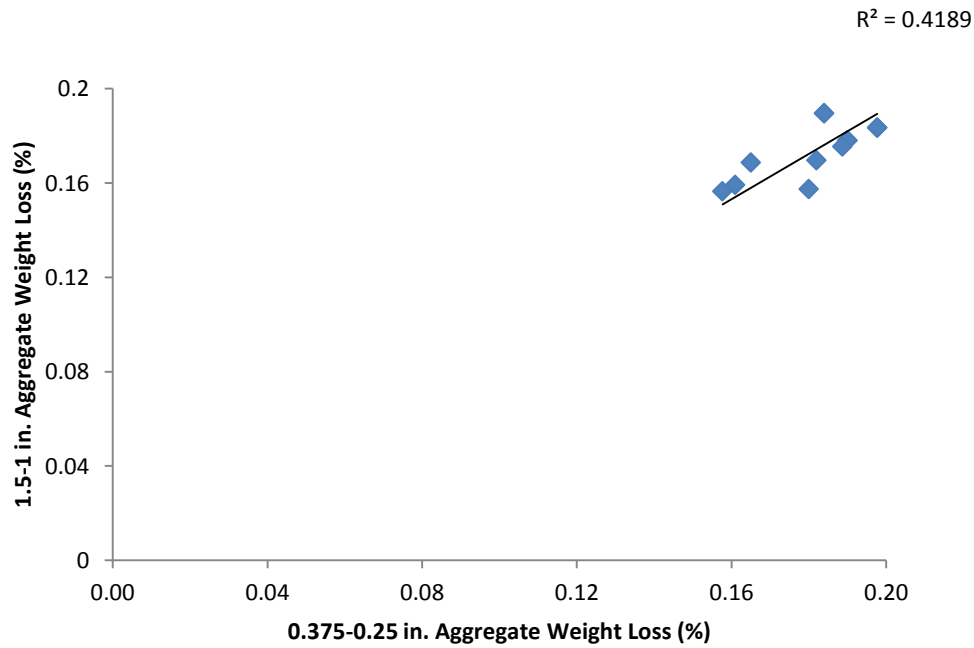


Figure 4-2: Comparison of aggregate weight loss (%) for the 1.5-1 in. aggregates and the 0.375 - 0.25 in. aggregates tested.

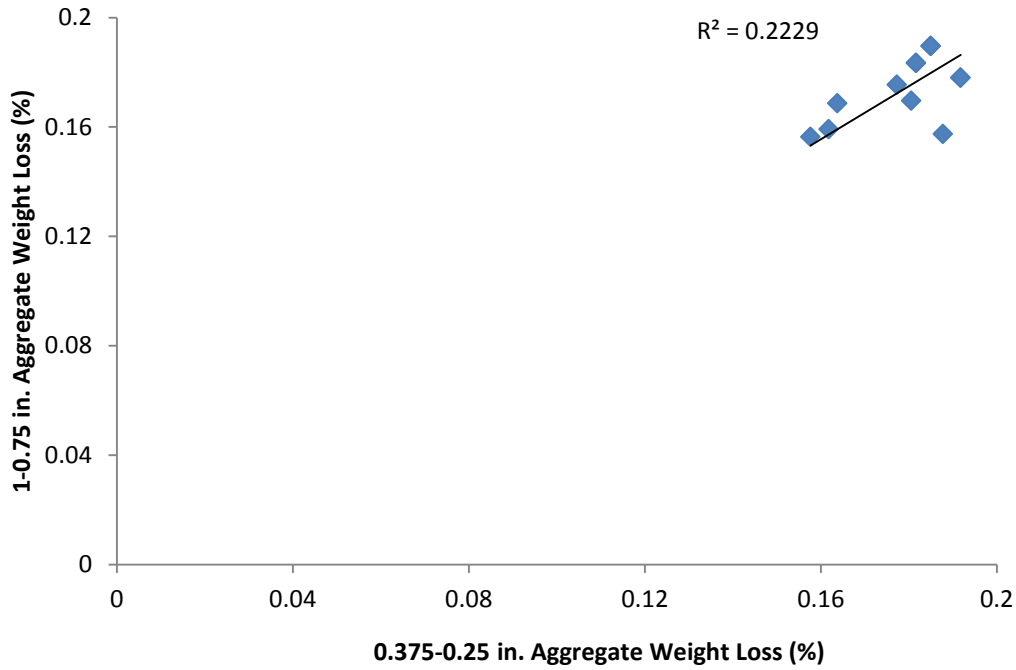


Figure 4-3: Comparison of aggregate weight loss for the 1-0.75 in. aggregates and the 0.375 - 0.25 in. aggregates tested.

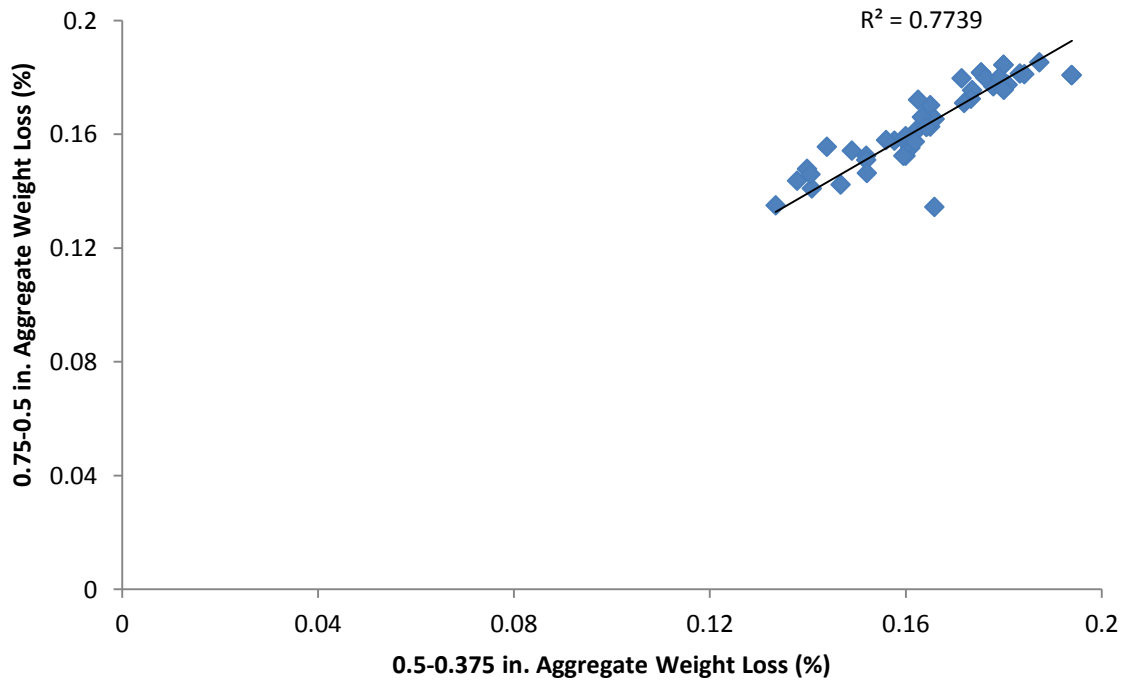


Figure 4-4: Comparison of aggregate weight loss for the 0.75-0.5 in. aggregates and the 0.5 - 0.375 in. aggregates tested.

4.3 Comparison of Average Freeze thaw loss of NaCl solution with MgCl₂ and CaCl₂ salt solutions

CSA A23.2-24A test method was modified to check if the limestone aggregates in Kansas was more sensitive to other salts. The MgCl₂ and CaCl₂ solutions were used on the local control aggregate. MgCl₂ salt solution was used on nine of the ledge samples, while CaCl₂ salt solution was also used on seven production samples. The MgCl₂ behaved similarly to the NaCl solution; however the CaCl₂ solution showed consistently lower weight loss, which may be due to different ionic concentrations. Figure 4.5 shows a comparison of the weight loss with the NaCl solution vs. the MgCl₂ and CaCl₂ salt solution, Figure 4-6 & Figure 4-7 shows comparisons of the weight loss with sieving time for the NaCl and CaCl₂ solutions for KDOT aggregate 09-1008 and 09-1918. Figure 4-8 shows the effects of NaCl, CaCl₂ and MgCl₂ salts on local aggregates.

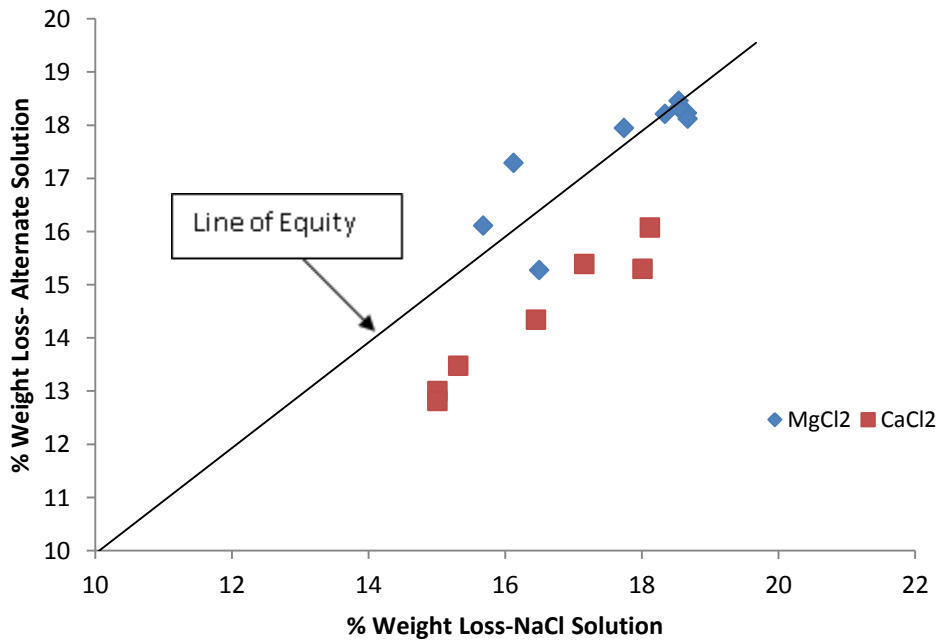


Figure 4-5: Comparison of aggregate freeze thaw weight loss for NaCl versus the MgCl₂ and CaCl₂ salt solutions.

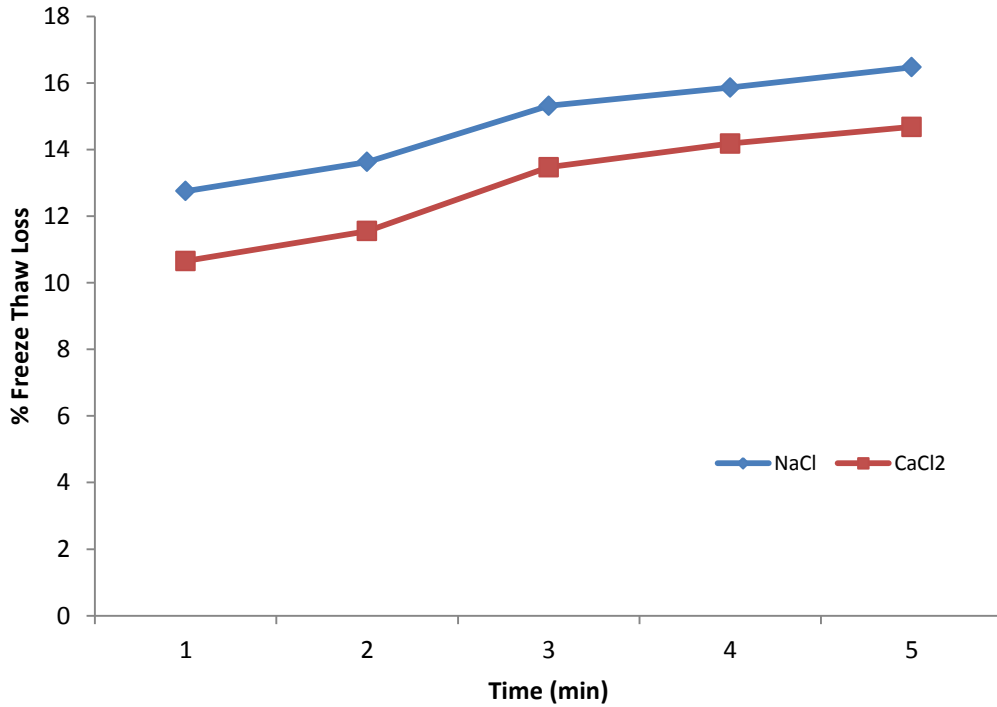


Figure 4-6: Comparison of NaCl and CaCl₂ salt solutions tested using CSA A23.3-24A (For Sample 1008 with DF=99).

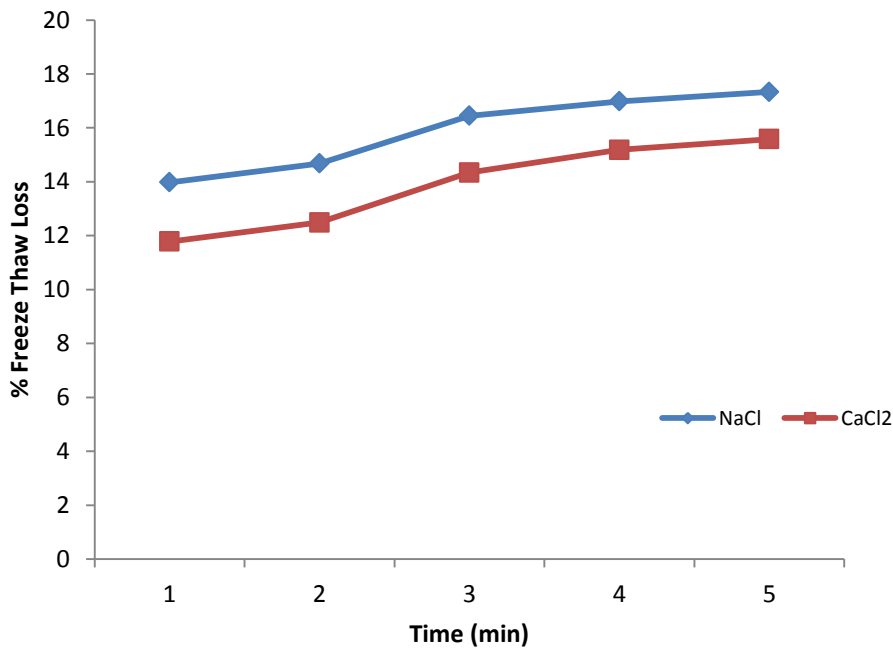


Figure 4-7: Comparison of NaCl and CaCl₂ effects on the freeze thaw weight loss using CSA A23.3-24A. (For Sample 1918 with DF=98).

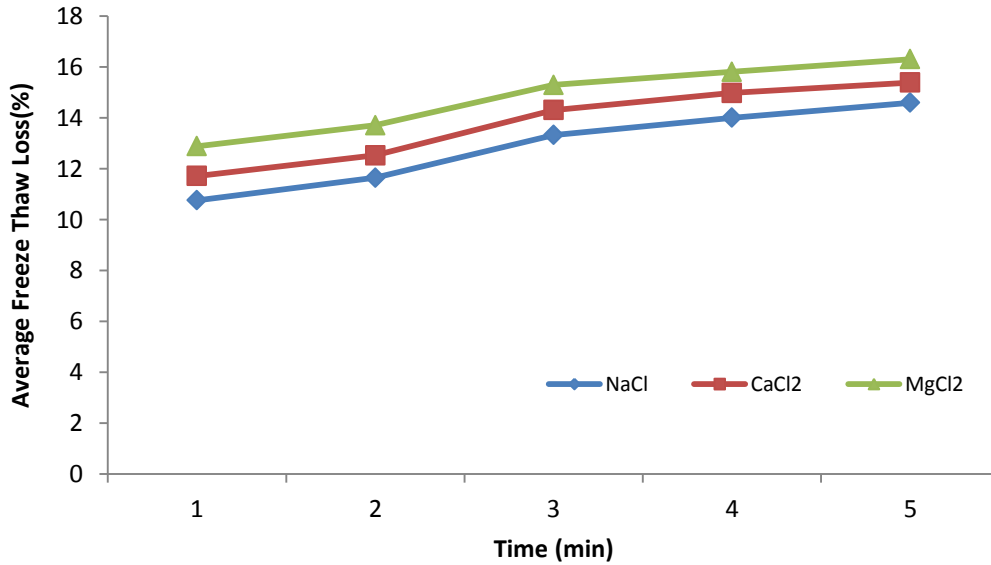


Figure 4-8: Comparison of local aggregate freeze thaw weight loss (%) for NaCl, MgCl₂ and CaCl₂ salt solutions using CSA A23.2-24A test method.

4.4 Nitrogen Adsorption Experiments

BET Nitrogen Testing was performed on a subset of the aggregates used in this study to determine if any correlation between the aggregate surface area and freezing and thawing performance existed. Figure 4-9 shows the surface area of pores of aggregates with different durability factors.

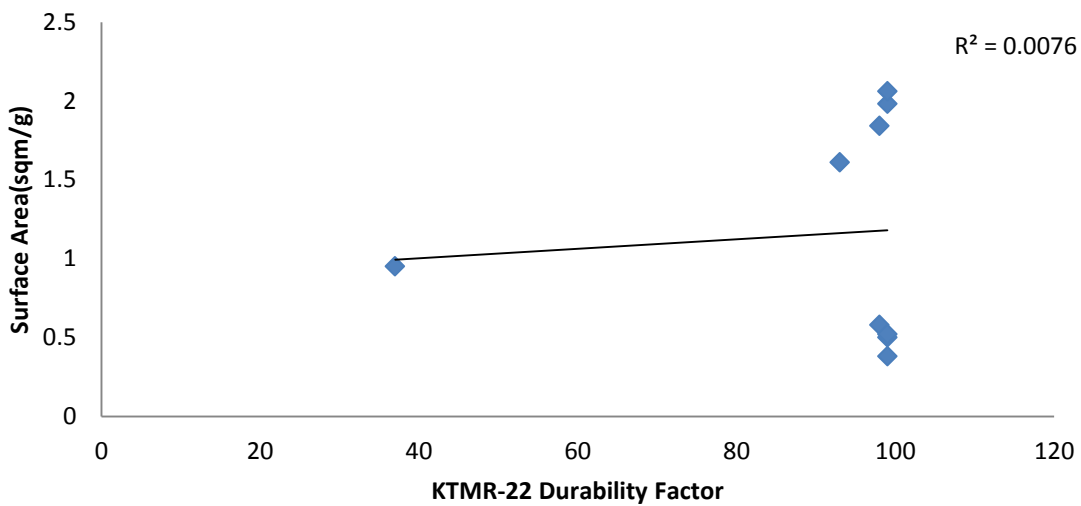


Figure 4-9: Comparison of KTMR-22 Durability factors and Aggregate Surface Area.

BET plots were used to check for variations in volume–pressure isotherms. Figure 4-10 shows volume–relative pressure plot for Sample 09-1468 B9 which has DF=37, Figure 4-11 shows BET plot for Sample 09-1248 with DF=99.

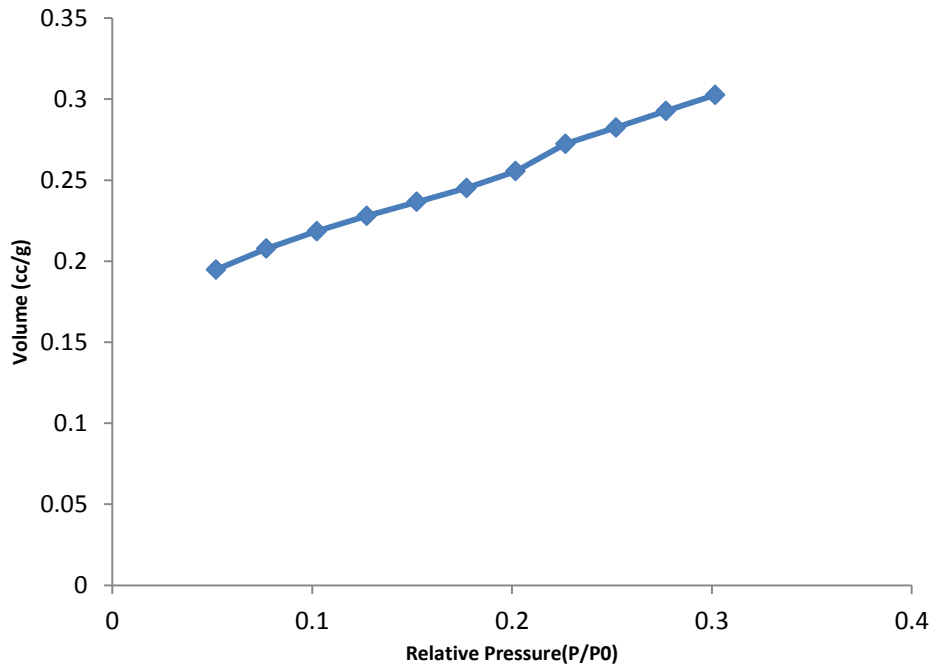


Figure 4-10: Linear variation observed in the BET plot for sample 09-1468 B9 with DF=37.

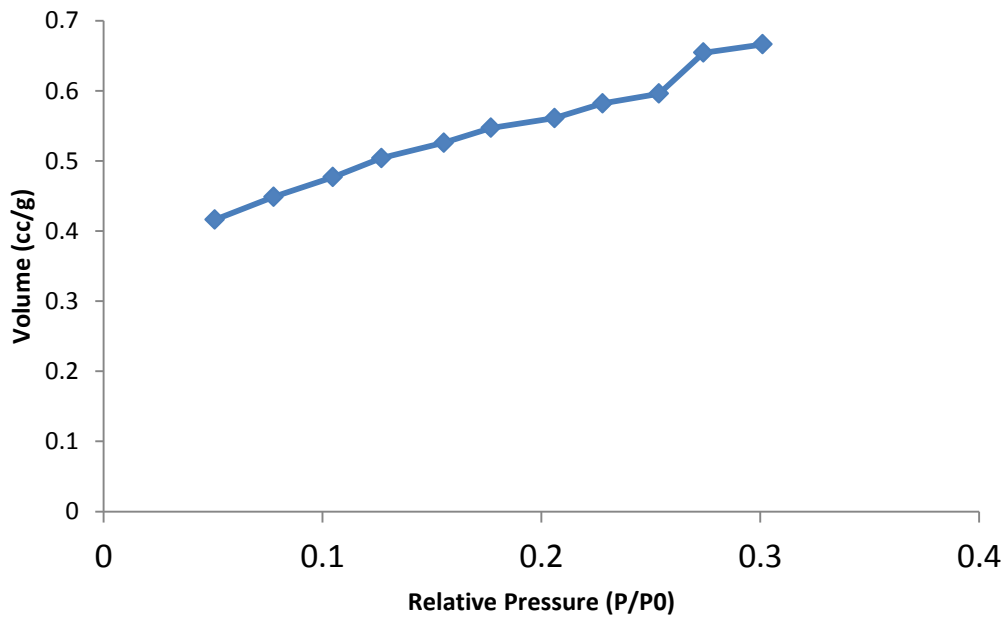


Figure 4-11: Volume–Relative Pressure plot for sample 09-1248 B9 with DF=99.

The BJH technique was used to calculate the pore size distribution of the aggregates. This method helped with the determination of distribution of areas and volumes among pores of varying radii. c value is basically an indicator to get correct pore size distribution. c value is given in Equation 4.1.

$$c = (r_p - t_r) / r_p \tag{Equation 4.1}$$

where, r_p is the average capillary radius, t_r is the thickness of physically adsorbed layer

The c values vary with the change in pore radii. It was observed that for all the aggregates tested, c values potentially vary in between 0.9 and 1 for different pore radii as shown in Figure 4-12. Pore volume distribution curves were used to compare with the durability factors. Figure 4-13 shows all the possible values of c for different pore radii. Figure 4.13 shows the pore volume distributions of aggregates with different durability factors.

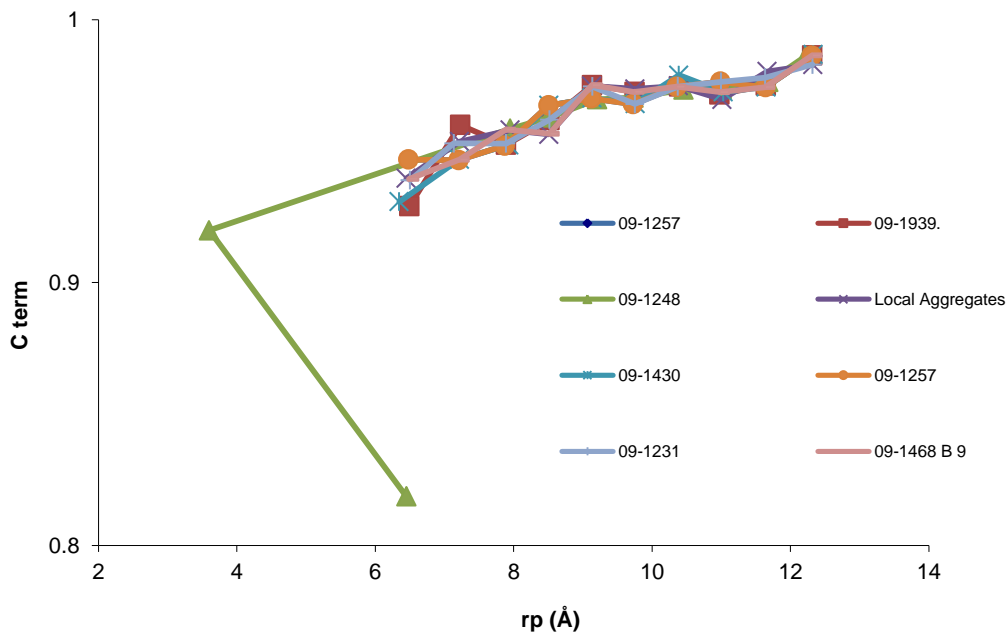


Figure 4-12: Variation of c term with r_p for pores of various radii.

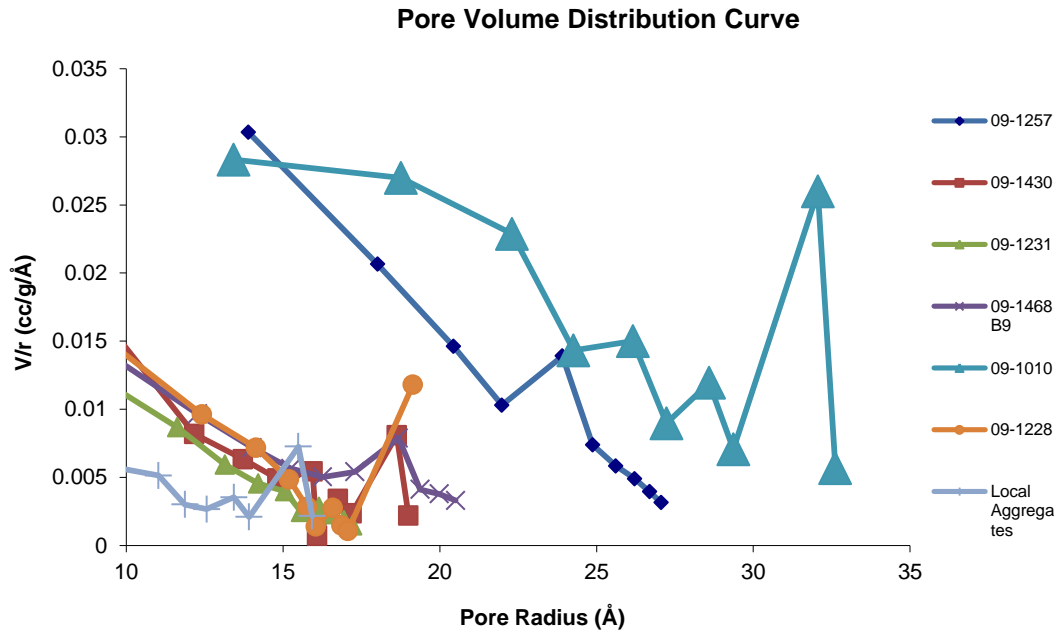


Figure 4-13: Pore Volume Distribution curves for aggregates with different durability factors.

4.5 Comparison of average freeze thaw loss with different aggregate performance measures done by KDOT

Comparisons were made between the production sample composite weight loss for all of the aggregate sieve fractions after sieving for three minutes and the aggregate performance measures used by KDOT. Figure 4-14 shows the aggregate weight loss vs. the pavement vulnerability factor (PVF). Figure 4-15 shows the aggregate weight loss vs. the KTMR-22 durability factor. Figure 4-16 shows the aggregate weight loss vs. the aggregate modified soundness test. Figure 4-17 shows the aggregate weight loss (%) vs. absorption values. Figure 4-18 shows aggregate weight loss (%) vs. wear (%). The figures show no correlation between the 3 min weight loss (%) of the aggregates tested and aggregate performance measures.

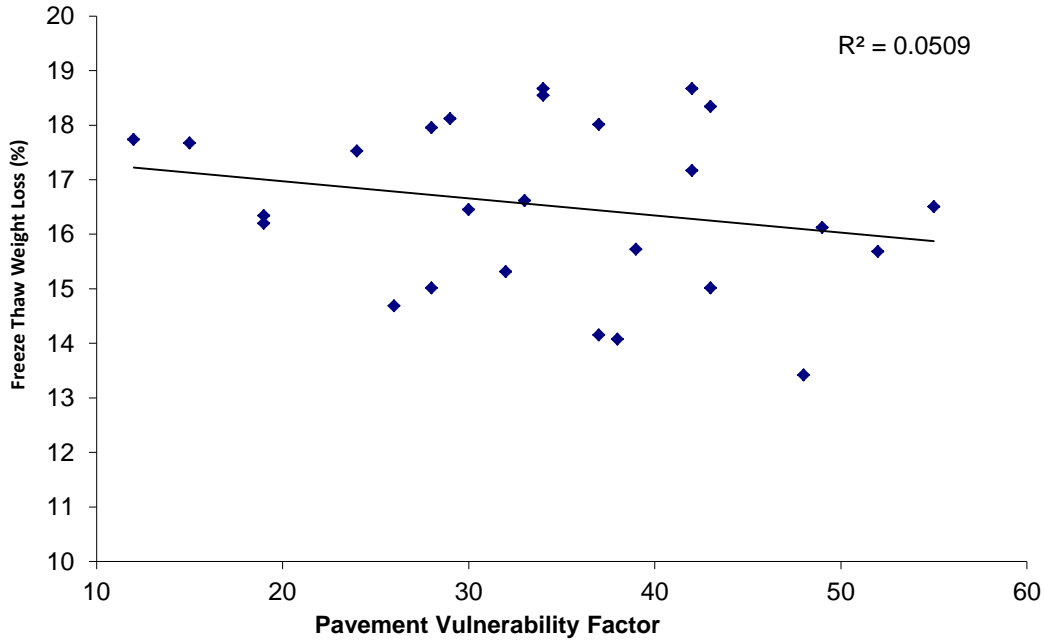


Figure 4-14: Aggregate weight loss (%) vs. Pavement Vulnerability Factor (PVF) for KDOT aggregates.

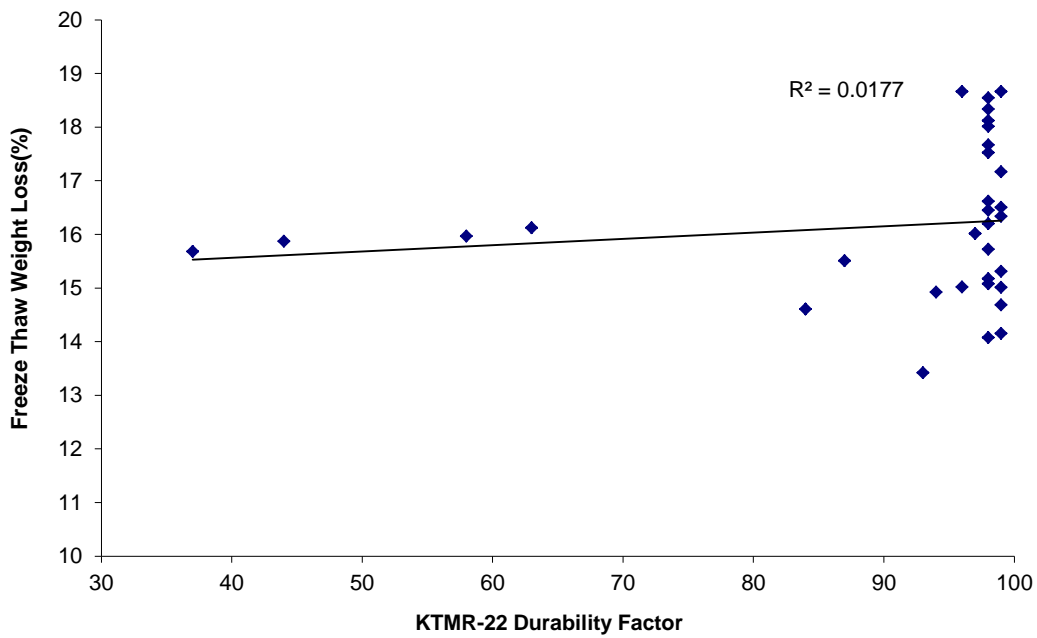


Figure 4-15: Aggregate weight loss (%) vs. KTMR-22 Durability Factor for KDOT aggregates.

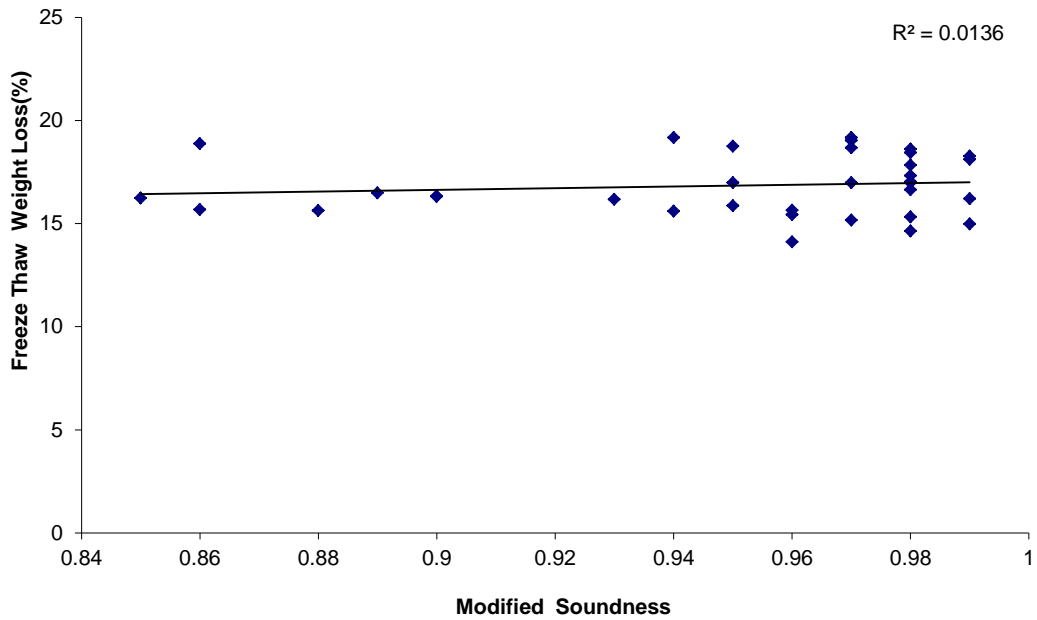


Figure 4-16: Aggregate weight loss (%) vs. Aggregate Modified Soundness test for KDOT aggregates.

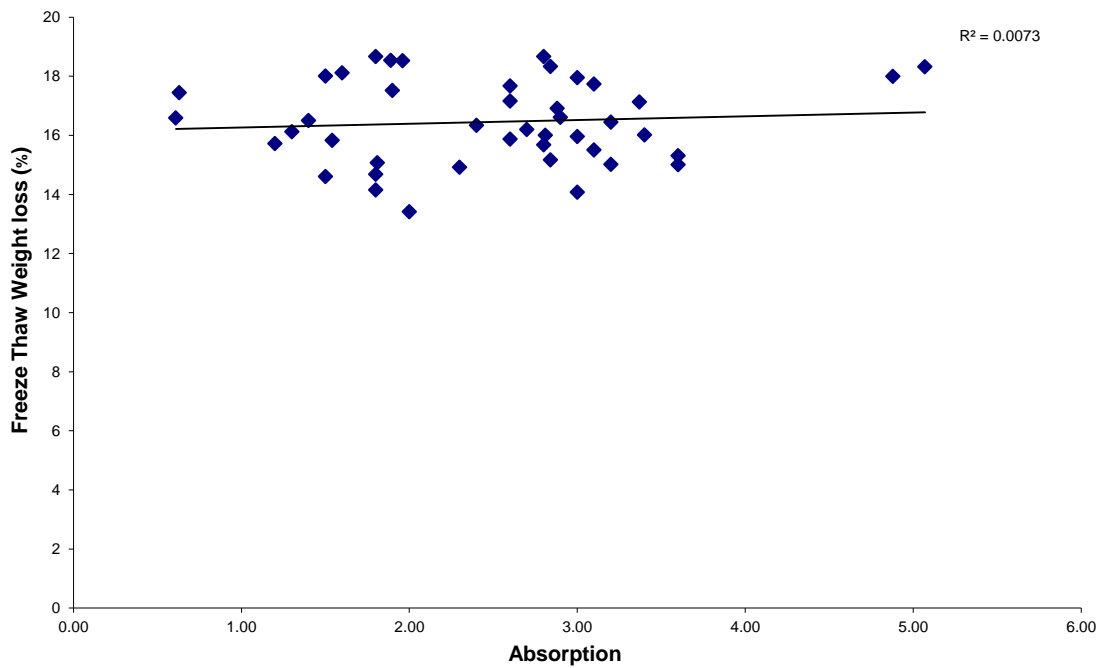


Figure 4-17: Aggregate weight loss (%) vs. Absorption values for KDOT aggregates.

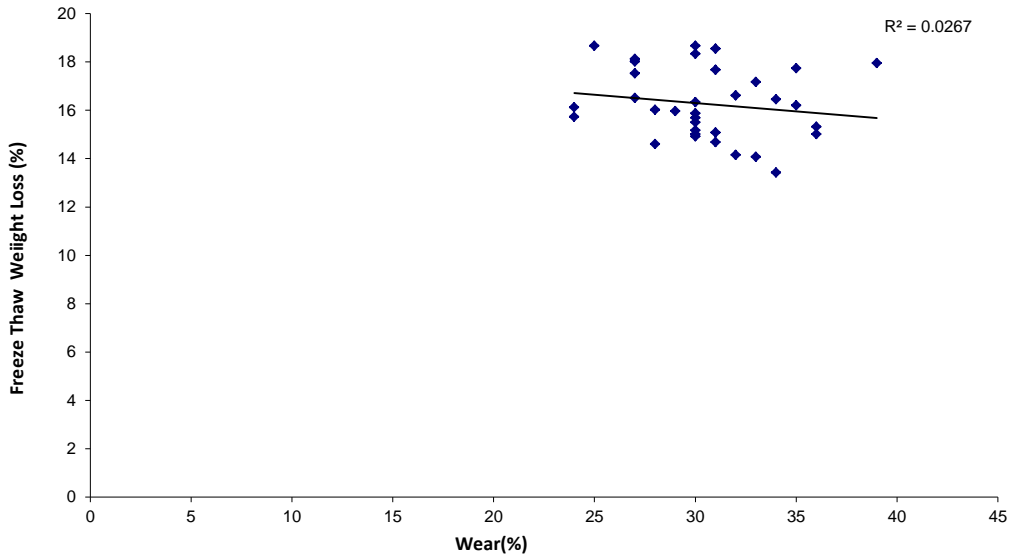


Figure 4-18: Aggregate weight loss (%) vs. Wear (%) for KDOT aggregates.

KDOT aggregate performance measures were compared to each other to check for any trend. Figure 4-19 shows results from aggregate modified soundness test vs. KTMR-22 durability factor. Figure 4-20 shows Wear (%) vs. KTMR-22 durability factor. Figure 4-21 shows pavement vulnerability factor vs. KTMR-22 durability factor. The figures show no correlation between the aggregate performance measures.

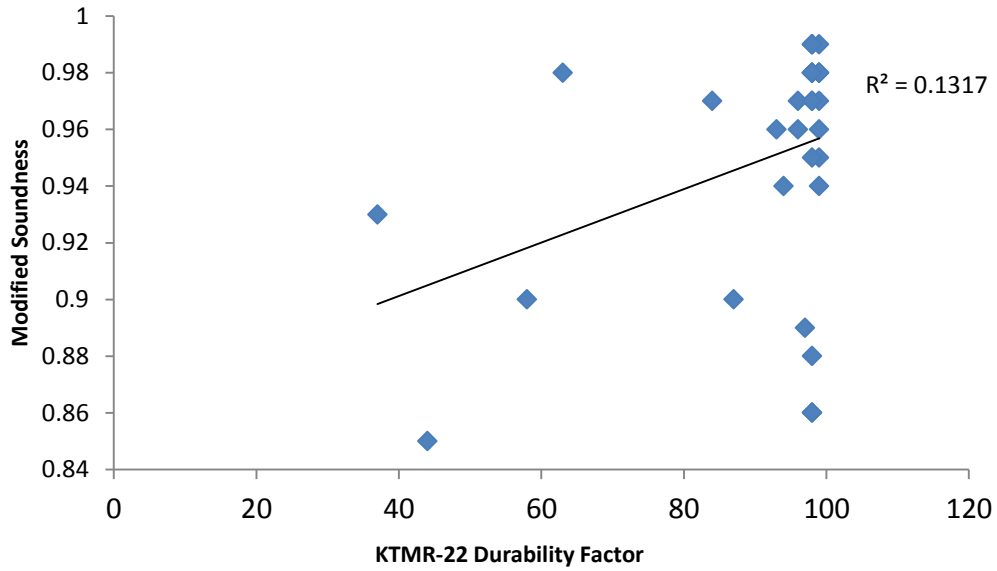


Figure 4-19: Aggregate Modified Soundness test vs. KTMR-22 Durability Factor for KDOT aggregates.

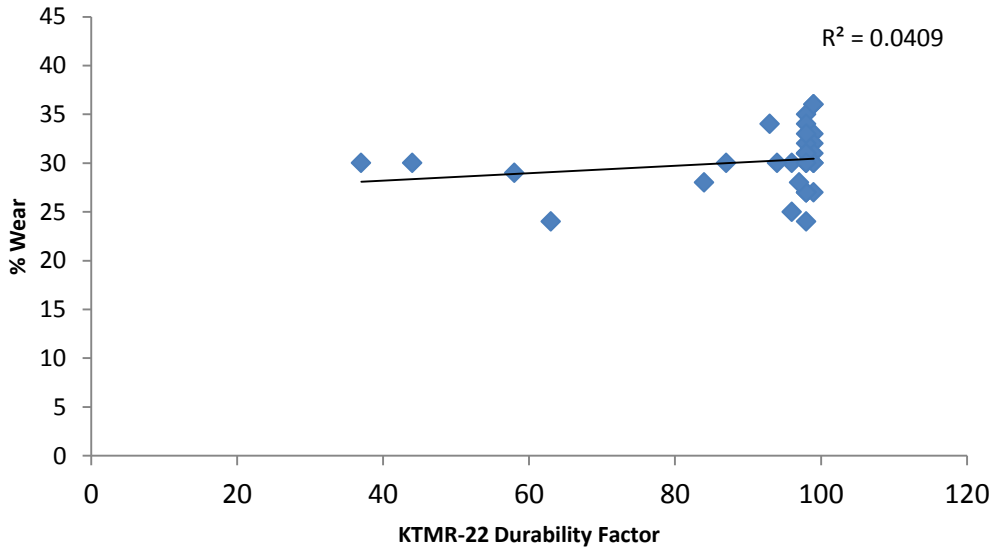


Figure 4-20: Wear (%) vs. KTMR-22 Durability Factor for KDOT aggregates.

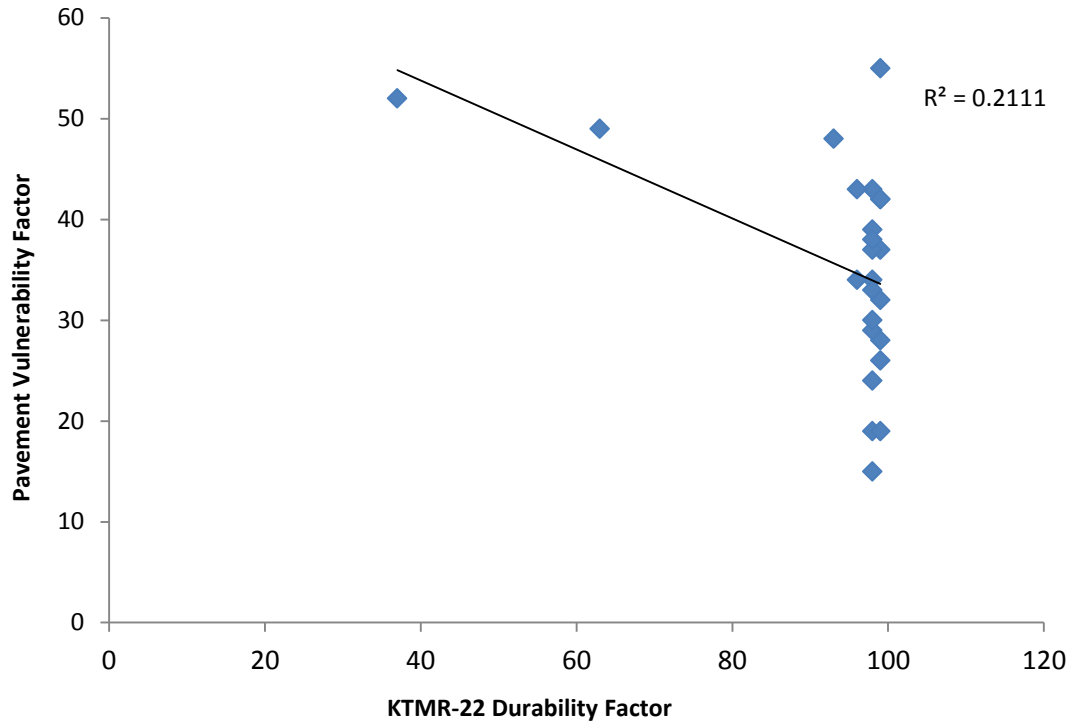


Figure 4-21: Pavement Vulnerability Factor vs. KTMR-22 Durability Factor for KDOT aggregates.

The results suggest that the CSA A23.2-24A test method does not clearly differentiate well and poor performing aggregate in concrete under freezing and thawing conditions.

Chapter 5 - Conclusions & Recommendations for Future Research

5.1 Conclusions

The objective of this project was to identify the non-durable aggregates, using CSA A23.2-24A test. KDOT aggregates were tested using KTMR-27 method for specific gravity and absorption. The specific gravity and absorption values obtained from the test were similar to the KDOT results.

The University of Windsor in collaboration with Ontario Ministry of Transportation developed CSA A23.2-24A method. In this method, aggregates were exposed to 3% NaCl solution and subjected to unconfined freezing and thawing cycles. After this process, the aggregates were re-sieved and the mass loss on each aggregate size was measured. A total of 51 aggregate samples including 12 ledge and 39 production samples were tested using the CSA A23.2-24A method. With the aid of Canadian reference aggregates, a new locally available limestone control aggregates was developed to comply with the CSA A23.2-24A specification. For Canadian control aggregates, three minutes of sieving with KSU sieving equipment yielded similar results as that obtained by MTO.

A modified version of CSA A23.2-24A was adopted because Kansas production limestone samples only contain aggregates below $\frac{3}{4}$ in. This modified version of CSA A23.2-24A test method concludes that aggregates perform similar in testing independent of size fraction. To confirm if the Kansas limestone aggregates are sensitive to alternative salt solutions, CSA A23.2-24A method was modified using 3% of CaCl_2 and 3% of MgCl_2 salt solutions. It was concluded that MgCl_2 gave similar results as when NaCl is used and the use of CaCl_2 resulted in a consistently decreased freeze thaw loss (%). Nitrogen adsorption testing done on selected aggregates concluded that there was no correlation between aggregate surface area and freeze thaw performance.

Experimental results found in this study lead to the following conclusions:

1. A comparison of original to modified version of CSA A23.2–24A test method concludes that aggregates perform similar in testing independent of size fraction.

2. Considering the consistent decrease in freeze thaw mass loss (%) for CaCl_2 , we can conclude that Kansas limestone aggregates are less sensitive to CaCl_2 than when compared to NaCl . Alternate salt solution like MgCl_2 behaves similarly to the NaCl solution.

3. When CSA A23.2–24A test is performed on companion KDOT aggregates and compared with KDOT test results, there is no trend observed between both the test results. Therefore, CSA A23.2-24A test method should not be performed for assessing Kansas coarse aggregate's resistance to freeze thaw.

5.2 Recommendations for Future Research

CSA A23.2-24A test method was designed to determine freeze thaw resistance of aggregates that are more sensitive to deicer salts. These aggregates can be generally found in areas where excessive freezing and thawing occurs. Generally Kansas aggregates do not experience severe freeze thaw damage when compared to the Canadian aggregates and this may be one of the reasons for severe freeze thaw mass loss in KDOT aggregates using CSA A23.2-24A test method. Only limited number of KDOT aggregates was subjected to different deicer salts like MgCl_2 and CaCl_2 and it was observed that Kansas aggregates were less sensitive to CaCl_2 than compared to MgCl_2 . The conclusion obtained from this project has led to various future recommendations:

1. Future study on using CSA A23.2-24A method on Kansas aggregates should focus on reducing the length of freeze thaw period, so that reduced freeze thaw cycles may yield good correlation with KDOT test results.
2. Further study is recommended to determine, if a consistent correlation can be found between CSA A23.2-24A test results and aggregate performance measures like durability factor, modified freeze thaw, pavement vulnerability factor by testing wide variety of aggregates.
3. CSA A23.2-24A test using MgCl_2 and CaCl_2 was done on limited aggregates and additional research should be done in the future to determine the effect of these salts on Kansas limestone aggregates.

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Appendix A - CSA A23.2-24A tests on Ledge samples

Table A.1: CSA A23.2-24A test results for 1.5-1" ledge sample.

Sno.	KDOT lab id	BED	Weight Loss (%) on 1.5-1" sieve				
			1 min	2 min	3 min	4 min	5 min
1	09-1468-P	8	13.79	14.78	16.10	16.34	16.50
2	09-1468-P	9	13.52	14.17	15.76	16.05	16.83
3	09-1469-P	1	13.30	14.44	16.50	16.71	17.14
4	09-1469-P	2	15.39	16.88	18.40	18.78	19.00
5	09-1884	1	16.32	16.97	19.76	20.05	20.23
6	09-1884	3	16.70	17.44	19.00	19.31	19.59
7	09-1885	1	15.39	16.88	18.40	18.78	19.00
8	09-1939.	.	16.32	16.97	19.76	20.05	20.23
9	09-1940.	.	16.70	17.44	19.00	19.31	19.59
10	09-3051.	2	16.58	17.28	18.20	18.58	18.82
11	09-3051.	3	16.00	16.31	18.87	19.40	19.78
12	09-1474.	1	15.60	16.04	18.00	18.45	18.75

Table A.2: CSA A23.2-24A test results for 1-0.75" ledge samples.

Sno.	KDOT lab id	BED	Weight Loss (%) on 1-0.75" sieve				
			1min	2min	3 min	4 min	5 min
1	09-1468-P	8	13.67	14.66	16.18	16.34	16.50
2	09-1468-P	9	13.52	14.17	15.76	16.05	16.83
3	09-1469-P	1	13.30	14.36	16.3	16.71	17.02
4	09-1469-P	2	15.19	14.86	18.	19.14	19.70
5	09-1884	1	15.72	16.37	18.18	18.85	19.23
6	09-1884	3	15.18	16.14	19.18	19.91	20.14
7	09-1885	1	15.19	14.88	18.50	19.14	19.70
8	09-1939.	.	15.72	16.37	18.16	18.85	19.23
9	09-1940.	.	15.18	16.16	19.18	19.91	20.14
10	09-3051.	2	14.99	15.30	18.06	18.45	18.92
11	09-3051.	3	15.32	15.73	17.72	18.40	18.83
12	09-1474.	1	14.37	15.01	18.78	19.02	19.37

Table A.3: CSA A23.2-24A test results on 0.75-0.5" sieve fraction.

Sno.	KDOT lab id	BED	Weight Loss (%) on 0.75-0.5" sieve				
			1min	2min	3 min	4 min	5 min
1	09-1468-P	8	13.95	14.88	16.24	16.55	16.76
2	09-1468-P	9	13.52	14.48	16.00	16.4	16.88
3	09-1469-P	1	13.30	14.68	16.50	16.71	17.14
4	09-1469-P	2	14.78	16.90	19.39	20.15	20.31
5	09-1884	1	15.52	16.48	18.00	19.20	20
6	09-1884	3	14.74	15.48	17.54	18.31	19.94
7	09-1885	1	14.78	16.90	19.39	20.15	20.31
8	09-1939.	.	15.52	16.48	18.00	19.2	20
9	09-1940.	.	14.74	15.48	17.54	18.31	19.94
10	09-3051.	2	14.94	16.69	17.79	18.6	19.28
11	09-3051.	3	15.83	16.21	17.62	18.4	18.68
12	09-1474.	1	14.57	15.60	17.14	17.8	18.2

Table A.4: CSA A23.2-24A test results on 0.5-0.375" sieve fraction.

Sno.	KDOT lab id	BED	Weight Loss (%) on 0.5-0.375" sieve				
			1min	2min	3 min	4 min	5 min
1	09-1468-P	8	13.78	14.66	16.18	16.97	17.11
2	09-1468-P	9	12.48	13.47	15.24	16.18	16.72
3	09-1469-P	1	12.46	13.72	16.27	17.25	18.11
4	09-1469-P	2	15.68	16.68	18.08	18.37	18.71
5	09-1884	1	16.68	17.47	18.44	18.18	18.52
6	09-1884	3	16.06	16.92	18.17	18.55	18.98
7	09-1885	1	15.68	16.68	18.08	18.37	18.71
8	09-1939.	.	16.68	17.47	18.44	18.18	18.52
9	09-1940.	.	16.06	16.92	18.17	18.55	18.98
10	09-3051.	2	16.68	17.18	17.68	18.17	18.41
11	09-3051.	3	17.18	17.53	17.99	18.26	18.5
12	09-1474.	1	15.87	16.46	17.96	18.36	18.7

Table A.5: CSA A23.2-24A test results on 0.375-0.25" sieve fraction.

Sno.	KDOT lab id	BED	Weight Loss (%) on 0.5-0.375" sieve				
			1min	2min	3 min	4 min	5 min
1	09-1468-P	8	14.32	14.66	15.92	16.96	17.3
2	09-1468-P	9	13.34	13.96	15.64	16.2	16.74
3	09-1469-P	1	13.44	14.46	16.86	17.52	17.92
4	09-1469-P	2	13.58	15.26	18.96	19.36	19.82
5	09-1884	1	15.06	15.98	18.34	18.84	19.74
6	09-1884	3	15.84	16.44	17.8	18.32	18.82
7	09-1885	1	13.58	15.26	18.96	19.36	19.82
8	09-1939.	.	15.06	15.98	18.34	18.84	19.74
9	09-1940.	.	15.84	16.44	17.8	18.32	18.82
10	09-3051.	2	14.44	15.46	16.96	18.36	19.02
11	09-3051.	3	14.74	15.58	17.54	19.3	19.82
12	09-1474.	1	13.98	14.24	15.74	16.92	17.82

Table A.6: Average Weighted Freeze Thaw Loss for ledge samples using CSA A23.2-24A method.

Sno.	KDOT lab id	BED	County	Average weighted Freeze thaw loss				
				1 min	2min	3 min	4 min	5 min
1	09-1468-P	8	Franklin	13.90	14.73	16.12	16.64	16.84
2	09-1468-P	9	Franklin	13.28	14.05	15.68	16.18	16.80
3	09-1469-P	1	Osage	13.16	14.33	16.50	16.98	17.47
4	09-1469-P	2	Osage	14.93	16.12	18.67	19.16	19.51
5	09-1884	1	Elk	15.86	16.66	18.54	19.02	19.55
6	09-1884	3	Elk	15.71	16.49	18.34	18.88	19.50
7	09-1885	1	Coffey	14.93	16.12	18.67	19.16	19.51
8	09-1939.	.	Sweet Home	15.86	16.66	18.54	19.02	19.55
9	09-1940.	.	Dell Rapids	15.71	16.49	18.34	18.88	19.50
10	09-3051.	2	Miami	15.53	16.39	17.74	18.44	18.89
11	09-3051.	3	Miami	15.82	16.27	17.95	18.75	19.12
12	09-1474.	1	Miami	14.88	15.47	17.52	18.11	18.57

Appendix B - CSA A23.2-24A tests on Production samples.

Table B.1: CSA A23.2-24A test results on 0.75-0.5" sieve.

Sno.	KDOT lab id	BED	MF sample id	Quarry no.	Weight Loss (%) on 0.75-0.5" sieve				
					1 min.	2 min.	3 min.	4 min.	5 min.
1	09-1008.	1-3.	746348	4-063-02	12.36	13.20	15.20	15.64	16.01
2	09-1010	1-3.	746354	4-025-02	14.18	15.16	17.20	17.71	18.03
3	09-1227	1-5.	747555	4-063-05	10.45	11.70	13.34	14.17	14.50
4	09-1228	1-2.	747556	4-050-06	11.71	12.50	14.08	14.75	15.18
5	09-1231	1-4.	748251	4-011-01	13.93	14.66	16.19	16.75	17.00
6	09-1248	4-5.	749369	4-030-02	12.10	12.84	14.39	14.84	15.03
7	09-1257	1	749653	4-061-10	15.60	16.35	17.92	18.45	18.84
8	09-1430	1-2.	750458	4-006-14	11.61	12.40	13.98	14.59	15.09
9	09-1454	1	751122	4-061-10	15.92	16.52	18.42	19.12	19.58
10	09-1706	2-6.	753423	4-002-01	12.36	13.20	15.20	15.64	16.01

Table B.2: CSA A23.2-24A test results obtained on 0.5-0.375" sieve.

Sno.	KDOT lab id	BED	MF sample id	Quarry no.	Weight Loss (%) on 0.5-0.375" sieve				
					1 min.	2 min.	3 min.	4 min.	5 min.
1	09-1008.	1-3.	746348	4-063-02	13.11	13.75	15.24	15.89	16.50
2	09-1010	1-3.	746354	4-025-02	14.53	15.07	17.10	17.64	18.01
3	09-1227	1-5.	747555	4-063-05	11.69	12.29	13.50	13.97	14.54
4	09-1228	1-2.	747556	4-050-06	12.00	12.44	14.10	14.50	15.01
5	09-1231	1-4.	748251	4-011-01	13.61	14.31	16.07	16.79	17.44
6	09-1248	4-5.	749369	4-030-02	12.57	13.55	15.56	16.24	16.58
7	09-1257	1	749653	4-061-10	14.50	15.58	18.00	18.60	19.29
8	09-1430	1-2.	750458	4-006-14	12.39	12.95	14.79	15.24	15.70
9	09-1454	1	751122	4-061-10	15.29	16.14	18.12	18.51	18.94
10	09-1706	2-6.	753423	4-002-01	12.97	13.60	15.10	15.56	15.94

Table B.3: CSA A23.2-24A test results obtained on 0.375-0.25" sieve.

Sno.	KDOT lab id	BED	MF sample id	Quarry no.	Weight Loss (%) on 0.375-0.25" sieve				
					1 min.	2 min.	3 min.	4 min.	5 min.
1	09-1008.	1-3.	746348	4-063-02	12.78	13.93	15.5	16.06	16.89
2	09-1010	1-3.	746354	4-025-02	14.14	15.43	17.21	18.17	19.29
3	09-1227	1-5.	747555	4-063-05	11.25	12.07	13.42	14.21	15.03
4	09-1228	1-2.	747556	4-050-06	11.47	12.26	14.27	15.71	16.13
5	09-1231	1-4.	748251	4-011-01	13.75	14.62	16.34	17.57	18.19
6	09-1248	4-5.	749369	4-030-02	12.07	13.03	15.09	15.87	16.21
7	09-1257	1	749653	4-061-10	15.96	16.79	18.12	18.81	19.52
8	09-1430	1-2.	750458	4-006-14	13.24	13.85	15.29	16.12	16.83
9	09-1454	1	751122	4-061-10	15.63	16.11	17.82	18.41	18.93
10	09-1706	2-6.	753423	4-002-01	12.23	12.79	14.76	15.12	15.68

Table B.4: Average Weighted Freeze Thaw Loss for production samples using CSA A23.2-24A method.

Sno.	KDOT lab id	BED	MF sample id	Quarry no.	Average weighted Freeze thaw loss				
					1 min	2min	3 min	4 min	5 min
1	09-1008.	1-3.	746348	4-063-02	12.75	13.63	15.31	15.86	16.47
2	09-1010	1-3.	746354	4-025-02	14.28	15.22	17.17	17.84	18.44
3	09-1227	1-5.	747555	4-063-05	11.13	12.02	13.42	14.12	14.69
4	09-1228	1-2.	747556	4-050-06	11.72	12.40	14.15	14.99	15.44
5	09-1231	1-4.	748251	4-011-01	13.76	14.53	16.20	17.03	17.54
6	09-1248	4-5.	749369	4-030-02	12.25	13.14	15.01	15.65	15.94
7	09-1257	1	749653	4-061-10	15.35	16.24	18.01	18.62	19.21
8	09-1430	1-2.	750458	4-006-14	12.41	13.06	14.69	15.32	15.87
9	09-1454	1	751122	4-061-10	15.61	16.26	18.12	18.68	19.15
10	09-1706	2-6.	753423	4-002-01	12.52	13.20	15.02	15.44	15.88

Table B.5: CSA A23.2-24A test results on 0.75-0.5" size fractions.

Sno.	KDOT lab id	BED	MF sample id	Quarry no.	Weight Loss (%) on 0.75-0.5" sieve				
					1 min.	2 min.	3 min.	4 min.	5 min.
11	09-1917.	5-7.	755203	1-045-11	15.59	16.04	18.00	18.51	18.84
12	09-1918.	4-5.	755317	4-030-02	14.04	14.55	16.33	16.99	17.33
13	09-1475.	1			13.12	13.82	15.60	16.17	16.51
14	09-2257.	1	757664						
15	09-2102.	4	756719	4-030-02					
16	09-3497.				15.17	16.14	18.01	18.70	19.17
17	09-3645.				13.96	14.94	16.59	17.07	17.70
18	09-2943.	5-7.	765280	1-046-11	13.28	14.69	16.42	17.01	17.19
19	09-2788.	4-5.	763711	4-030-02	11.57	12.42	13.78	14.42	14.81
20	09-3453.	3		4-061-09	12.86	13.76	14.90	15.63	16.10
21	10-0354	C,D		5-018-01	14.51	15.34	16.04	16.42	16.86

Table B.6: CSA A23.2-24A test results on 0.5-0.375" size fractions.

Sno.	KDOT lab id	BED	MF sample id	Quarry no.	Weight Loss (%) on 0.5-0.375" sieve				
					1 min.	2 min.	3 min.	4 min.	5 min.
11	09-1917.	5-7.	755203	1-045-11	14.50	15.08	17.59	18.38	18.71
12	09-1918.	4-5.	755317	4-030-02	13.97	14.94	16.60	17.11	17.44
13	09-1475.	1			13.30	13.84	15.80	16.25	16.60
14	09-2257.	1	757664		14.36	14.90	15.90	16.25	16.47
15	09-2102.	4	756719	4-030-02	14.79	15.30	16.34	16.72	16.95
16	09-3497.				15.00	15.81	17.56	17.93	18.13
17	09-3645.				13.44	13.44	13.44	13.44	13.44
18	09-2943.	5-7.	765280	1-046-11	13.78	14.39	16.25	16.94	17.20
19	09-2788.	4-5.	763711	4-030-02	12.24	13.00	14.36	14.87	15.22
20	09-3453.	3		4-061-09	13.32	14.48	15.43	15.97	16.49
21	10-0354	C,D		5-018-01	14.25	14.80	15.67	16.29	16.53

Table B.7: CSA A23.2-24A test results on 0.375-0.25" size fractions.

Sno.	KDOT lab id	BED	MF sample id	Quarry no.	Weight Loss (%) on 0.375-0.25" sieve				
					1 min.	2 min.	3 min.	4 min.	5 min.
11	09-1917.	5-7.	755203	1-045-11	14.93	15.78	17.42	17.91	18.26
12	09-1918.	4-5.	755317	4-030-02	13.95	14.57	16.42	16.86	17.24
13	09-1475.	1			13.02	13.79	15.78	16.19	16.53
14	09-2257.	1	757664		15.21	15.86	17.44	18.09	18.47
15	09-2102.	4	756719	4-030-02	14.11	14.78	16.90	17.93	18.36
16	09-3497.								
17	09-3645.								
18	09-2943.	5-7.	765280	1-046-11					
19	09-2788.	4-5.	763711	4-030-02					
20	09-3453.	3		4-061-09	12.36	13.44	14.44	15.22	15.86
21	10-0354	C,D		5-018-01	14.77	15.61	16.34	16.76	17.10

Table B.8: Average Weighted Freeze Thaw Loss for production samples using CSA A23.2-24A method.

Sno.	KDOT lab id	BED	Company	MF sample id	Quarry no.	Average weighted Freeze thaw loss				
						1 min	2min	3 min	4 min	5 min
11	09-1917.	5-7.	Hunt Martin	755203	1-045-11	15.01	15.63	17.67	18.27	18.60
12	09-1918.	4-5.	Hunt Martin	755317	4-030-02	13.99	14.69	16.45	16.98	17.34
13	09-1475.	1	APAC KS			13.15	13.82	15.73	16.20	16.54
14	09-2257.	1	APAC KS	757664		14.78	15.38	16.67	17.17	17.47
15	09-2102.	4	Hunt Martin	756719	4-030-02	14.45	15.04	16.62	17.33	17.66
16	09-3497.					15.09	15.97	17.78	18.32	18.65
17	09-3645.					13.70	14.19	15.01	15.25	15.57
18	09-2943.	5-7.	Hunt Martin	765280	1-046-11	13.53	14.54	16.34	16.98	17.19
19	09-2788.	4-5.	Hunt Martin	763711	4-030-02	11.91	12.71	14.07	14.64	15.02
20	09-3453.	3	APAC-KS		4-061-09	12.84	13.89	14.92	15.60	16.15
21	10-0354	C,D	Whitaker		5-018-01	14.51	15.25	16.01	16.49	16.83

Table B.9: CSA A23.2-24A test results on 0.75-0.5" size fractions.

Sno.	KDOT lab id	BED	Producer id	Quarry no.	Weight Loss (%) on 0.75-0.5" sieve				
					1 min.	2 min.	3 min.	4 min.	5 min.
22	08-2058.	1		MO-042	14.05	14.54	15.95	16.58	16.90
23	09-2642.	2	803903	1-046-16	12.58	13.22	14.67	15.21	15.70
24	09-3453.	4	804008	4-061-09	14.53	15.25	15.99	16.45	16.75
25	08-355.				13.38	14.05	15.20	15.69	16.06
26	08-2323.				12.90	13.50	14.05	14.50	14.84
27	09-2642.	1	803903	1-046-16	14.82	15.43	16.18	16.57	16.87
28	10-0211	2,3	802439	MO-023					
29	10-0424	1,2,3	802244	4-025-02					

Table B.10: CSA A23.2-24A test results on 0.5-0.375" size fractions.

Sno.	KDOT lab id	BED	Producer id	Quarry no.	Weight Loss (%) on 0.5-03725" sieve				
					1 min.	2 min.	3 min.	4 min.	5 min.
22	08-2058.	1		MO-042	13.78	14.38	15.24	16.30	16.59
23	09-2642.	2	803903	1-046-16	13.04	13.51	14.23	14.82	15.32
24	09-3453.	4	804008	4-061-09	14.31	14.87	15.92	16.25	16.44
25	08-355.				12.88	13.76	14.63	15.12	15.52
26	08-2323.				13.24	13.93	14.59	15.07	15.41
27	09-2642.	1	803903	1-046-16	14.47	15.07	15.74	16.08	16.28
28	10-0211	2,3	802439	MO-023	13.50	14.23	15.075	15.62	16.06
29	10-0424	1,2,3	802244	4-025-02	14.70	15.38	15.88	16.23	16.47

Table B.11: CSA A23.2-24A test results on 0.375-0.25" size fractions.

Sno.	KDOT lab id	BED	Producer id	Quarry no.	Weight Loss (%) on 0.375-0.25" sieve				
					1 min.	2 min.	3 min.	4 min.	5 min.
22	08-2058.	1		MO-042	13.64	14.27	15.32	16.08	16.51
23	09-2642.	2	803903	1-046-16	13.08	13.72	14.92	15.45	16.03
24	09-3453.	4	804008	4-061-09	14.87	15.18	15.70	15.99	16.14
25	08-355.				13.21	13.93	14.81	15.85	16.30
26	08-2323.				13.51	13.91	14.38	15.00	15.45
27	09-2642.	1	803903	1-046-16	14.99	15.66	16.44	16.75	17.05
28	10-0211	2,3	802439	MO-023					
29	10-0424	1,2,3	802244	4-025-02	12.58	13.79	14.46	15.12	15.53

Table B.12: Average Weighted Freeze Thaw Loss for production samples using CSA A23.2-24A method.

Sno.	KDOT lab id	BED	Producer id	Quarry no.	Average weighted Freeze thaw loss (%)				
					1 min	2min	3 min	4 min	5 min
22	08-2058.	1		MO-042	13.82	14.39	15.50	16.32	16.66
23	09-2642.	2	803903	1-046-16	12.90	13.48	14.60	15.16	15.68
24	09-3453.	4	804008	4-061-09	14.57	15.10	15.87	16.23	16.44
25	08-355.				13.16	13.91	14.88	15.55	15.96
26	08-2323.				13.21	13.78	14.34	14.85	15.23
27	09-2642.	1	803903	1-046-16	14.64	15.253	15.96	16.33	16.57
28	10-0211	2,3	802439	MO-023	13.50	14.23	15.07	15.62	16.06
29	10-0424	1,2,3	802244	4-025-02	13.64	14.58	15.17	15.67	16.00

**Appendix C - Modified CSA A23.2-24A tests results using MgCl₂
and CaCl₂ salt solutions**

Table C.1: Average Weighted Freeze Thaw Loss for production samples by using CaCl₂ salt solution method.

Sno.	KDOT lab id	BED	MF sample id	Quarry no.	Average weighted Freeze thaw loss (%)				
					1 min	2min	3 min	4 min	5 min
1	09-1008.	1-3.	746348	4-063-02	10.65	11.55	13.47	14.18	14.68
2	09-1010	1-3.	746354	4-025-02	12.877	13.81	15.39	16.24	16.56
3	09-1248	4-5.	749369	4-030-02	10.76	11.50	13.00	13.73	14.04
4	09-1454	1	751122	4-061-10	13.92	14.74	16.07	16.70	16.93
5	09-1918.	4-5.	755317	4-030-02	11.78	12.49	14.341	15.18	15.58
6	09-1706	2-6.	753423	4-002-01	10.31	11.149	12.81	13.46	13.94
7	09-1257	1	749653	4-061-10	12.59	13.36	15.29	16.05	16.50

Table C.2: Average Weighted Freeze Thaw Loss for production samples by using MgCl₂ salt solution method.

Sno.	KDOT lab id	BED	Company	Average weighted Freeze thaw loss (%)				
				1 min	2min	3 min	4 min	5 min
1	09-1468-P	8	Hunt Martin	14.130	15.23	17.28	17.79	18.14
2	09-1468-P	9	Hunt Martin	12.58	14.08	16.11	16.90	17.39
3	09-1469-P	1	Mid states	12.00	13.23	15.27	16.17	16.72
4	09-1469-P	2		16.12	16.82	18.23	18.72	19.03
5	09-1884	1	Martin Marietta	16.68	17.35	18.46	18.79	19.02
6	09-1885	1		16.30	16.98	18.12	18.68	18.84
7	09-1939.	.	Granite Mountain	16.55	17.21	18.32	18.85	19.07
8	09-1940.	.	L.G.Everest	16.62	17.13	18.21	18.55	18.85
9	09-3051.	2	APAC	16.127	16.58	17.94	18.30	18.66

Appendix D - Results from KDOT tests on Companion aggregates

Table D.1: KDOT results on ledge samples.

KDOT Results on Ledge samples									
Sno.	KDOT lab id	BED	DF	EXP	MOD FT	%A.I	%WEAR	CY	PVF
1	09-1468-P	8	63	0.116	0.98	3.23	24		49
2	09-1468-P	9	37	0.181	0.93	7.92	30		52
3	09-1469-P	1	99	0.012	0.97	4.51	27		55
4	09-1469-P	2	99	0.009	0.94	5.42	30		42
5	09-1884	1	98	0.01	0.97	2.52	31		34
6	09-1884	3	98	0.009	0.86	5.59	30		43
7	09-1885	1	96	0.017	0.97	2.38	25		34
8	09-1939.	.							
9	09-1940.	.							
10	09-3051.	2			0.98	1.06	35	300	12
11	09-3051.	3			0.95	3.03	39	300	28
12	09-1474.	1	98	0.002	0.99	1.52	27	300	24

Table D.2: KDOT results on production samples.

KDOT Results on Production samples									
Sno.	KDOT lab id	BED	DF	EXP	MOD FT	%A.I	%WEAR	CY	PVF
1	09-1008.	1-3.	99	0.008	0.95	4.50	36	300	32
2	09-1010	1-3.	99	0.008	0.98	4.80	33	300	42
3	09-1227	1-5.	93	0.028	0.96	4.7	34	300	48
4	09-1228	1-2.	99	0.015	0.99	2.8	32	300	37
5	09-1231	1-4.	98	0.012	0.98	1.7	35	300	19
6	09-1248	4-5.	99	0	0.96	3.7	36	300	28
7	09-1257	1	98	0.01	0.98	2.3	27	300	37
8	09-1430	1-2.	99	0.008	0.98	1.7	31	300	26
9	09-1454	1	98	0.008	0.97	1.8	27	300	29
10	09-1706	2-6.	96	0.004	0.96	6.3	30	300	43
11	09-1917.	5-7.	98	0.012	0.99	1.2	31	300	15
12	09-1918.	4-5.	98	0.014	0.95	3.8	34	300	30
13	09-1475.	1	98	0.018	0.99	2	24	300	39
14	09-2257.	1	97	0.018	0.98	1.95	27	300	32
15	09-2102.	4	98	0.007	0.98	3.8	32	300	33
16	09-3497.		98	0.07	0.99	1.38	34	300	19
17	09-3645.								
18	09-2943.	5-7.	99	0.001	0.98	1.4	30	300	19
19	09-2788.	4-5.	98	0.004	0.98	4.9	33	300	38
20	09-3453.	3	94	0.014	0.94	6.23	30	300	16
21	10-0354	C,D	97	0.09	0.89	24.85	28	300	74
22	08-2058.	1	87	0.032	0.9	10.32	30	300	56
23	09-2642.	2	84	0.045	0.97	4.14	28	237	52
24	09-3453.	4	44	0.127	0.85	8.67	30	300	56
25	08-355.		63	0.12	0.98	3.23	24	300	49
26	08-2323.		99	0.01	0.78	5.30	30	300	41
27	09-2642.	1	58	0.082	0.9	6.81	29	237	52
28	10-0211	2,3	86	0.047	0.93	5.90	26	300	59
29	10-0424	1,2,3	98	0.008	0.95	3.39	32	300	32

Appendix E - BJH Calculations on Selected KDOT Aggregates

Table E.1: BJH calculations for Sample 09-1468 B9 (DF=37).

P/P_0	V	Average Radius	C term	$V_{pn}(\text{Eqn25})$	V/r	r_p	A_p
0.0770	0.2077	3.4783	0.9393	0.0488	0.0140	9.4502	280.5480
0.1023	0.2185	3.9588	0.9467	0.0382	0.0097	12.2808	473.7818
0.1273	0.2279	4.4139	0.9582	0.0317	0.0072	14.0190	617.3891
0.1524	0.2365	4.8576	0.9566	0.0275	0.0057	15.2493	730.5077
0.1772	0.2451	5.3007	0.9751	0.0266	0.0050	16.2648	831.0431
0.2018	0.2555	5.7466	0.9725	0.0311	0.0054	17.2908	939.1973
0.2267	0.2724	6.2046	0.9745	0.0489	0.0079	18.6852	1096.7790
0.2520	0.2824	6.6857	0.9724	0.0275	0.0041	19.3741	1179.1423
0.2771	0.2927	7.1893	0.9743	0.0272	0.0038	19.9868	1254.9091
0.3018	0.3026	7.7111	0.9863	0.0255	0.0033	20.5062	1320.9822

Table E.2: BJH Calculations for sample 09-1010 (DF=99).

P/P_0	V	Average Radius	C term	V_{pn} (Eqn25)	V/r	r_p	A_p
0.0776	0.3715	3.47167494	0.939222	0.098307	0.028317	13.42686	566.3356
0.10461	0.40168	3.98556174	0.953658	0.107523	0.026978	18.76267	1105.896
0.1268	0.4318	4.42999925	0.95833	0.101329	0.022873	22.30835	1563.364
0.15216	0.4534	4.85118106	0.96203	0.069499	0.014326	24.26668	1849.889
0.1759	0.479	5.28691945	0.969964	0.079318	0.015003	26.16079	2149.943
0.2013	0.4964	5.72996522	0.967773	0.051349	0.008961	27.22941	2329.173
0.2282	0.5222	6.21432987	0.978736	0.074206	0.011941	28.59134	2567.995
0.2523	0.5395	6.70309665	0.97249	0.047324	0.00706	29.36687	2709.197
0.2773	0.6083	7.19439851	0.977997	0.186911	0.02598	32.05958	3228.798
0.3011	0.6255	7.70549459	0.982882	0.04367	0.005667	32.61746	3342.146

Table E.3: BJH Calculations for sample 09-1228 (DF=99).

P/P_0	V	Average Radius	C term	V_{pn} (Eqn25)	V/r	r_p	A_p
0.0774	0.0868	3.4616	0.9314	0.0506	0.0146	9.6443	292.1935
0.1025	0.0975	3.9647	0.9535	0.0381	0.0096	12.4181	484.4348
0.1275	0.1069	4.4172	0.9583	0.0317	0.0072	14.1384	627.9526
0.1526	0.1143	4.8611	0.9566	0.0236	0.0049	15.1917	725.0020
0.1776	0.1192	5.3059	0.9751	0.0150	0.0028	15.7718	781.4294
0.2028	0.1220	5.7591	0.9680	0.0080	0.0014	16.0513	809.3630
0.2270	0.1281	6.2167	0.9745	0.0172	0.0028	16.5900	864.6028
0.2525	0.1318	6.6936	0.9764	0.0098	0.0015	16.8689	893.9240
0.2775	0.1349	7.1985	0.9744	0.0077	0.0011	17.0699	915.3482
0.3016	0.1691	7.7131	0.9863	0.0911	0.0118	19.1455	1151.4878

Table E.4: BJH Calculations for 09-1231 sample (DF=98).

P/P_0	V	Average Radius	C term	V_{pn} (Eqn25)	V/r	r_p	A_p
0.07396	0.1267	3.45563569	0.93894	0.043425	0.012566	8.944501	251.326
0.10243	0.1362	3.93151894	0.952919	0.03411	0.008676	11.62928	424.8456
0.1275	0.144	4.41662247	0.95286	0.026155	0.005922	13.15077	543.2844
0.15252	0.1509	4.8605541	0.961959	0.022107	0.004548	14.20913	634.2492
0.17758	0.1577	5.30522918	0.974553	0.021059	0.00397	15.07221	713.6397
0.20275	0.1625	5.7584921	0.967908	0.014103	0.002449	15.58089	762.6222
0.2275	0.1687	6.22102622	0.974538	0.017539	0.002819	16.14661	819.0075
0.252	0.1743	6.69339209	0.976335	0.015264	0.00228	16.59011	864.6168
0.2774	0.1794	7.1924511	0.977949	0.013325	0.001853	16.94187	901.67
0.3027	0.1839	7.72419522	0.982911	0.011254	0.001457	17.21344	930.8092

Table E.5: BJH Calculations for sample 09-1257 (DF=98).

P/P₀	V	Average Radius	C term	V_{pn} (Eqn25)	V/r	r_p	Ap
0.0765	0.4496	3.47164647	0.946798	0.105284	0.030327	13.89525	606.538
0.1017	0.4726	3.94905811	0.946519	0.081556	0.020652	18.01555	1019.577
0.1268	0.4918	4.40387704	0.95206	0.064387	0.01462	20.43629	1311.987
0.152	0.5073	4.84976389	0.967363	0.049947	0.010299	21.98204	1517.963
0.1766	0.531	5.29179992	0.970067	0.073622	0.013913	23.91203	1796.213
0.202	0.5454	5.74273176	0.967838	0.042417	0.007386	24.87588	1943.937
0.227	0.5583	6.20930077	0.97449	0.036305	0.005847	25.61316	2060.874
0.25216	0.5704	6.69020087	0.976339	0.032813	0.004905	26.21564	2158.968
0.27719	0.5814	7.19184885	0.974304	0.028403	0.003949	26.69089	2237.954
0.30226	0.5913	7.71713506	0.986316	0.024286	0.003147	27.06361	2300.894

Table E.6: BJH Calculations for sample 09-1430 (DF=99).

P/P_0	V	Average Radius	C term	V_{pn} (Eqn25)	V/r	r_p	A_p
0.077	0.0806	3.43151997	0.93073	0.051705	0.015068	9.794337	301.3528
0.104	0.0898	3.97443461	0.94686	0.032553	0.008191	12.16862	465.1658
0.127	0.0982	4.42629765	0.952308	0.028033	0.006333	13.72574	591.8298
0.1524	0.1056	4.85507396	0.967354	0.02384	0.00491	14.82087	690.0374
0.1778	0.1149	5.3061479	0.970148	0.028817	0.005431	15.94472	798.655
0.2033	0.1164	5.76556526	0.967948	0.004061	0.000704	16.08472	812.7412
0.2286	0.1239	6.2366648	0.978835	0.021348	0.003423	16.74847	881.2018
0.2525	0.1297	6.70893148	0.972455	0.01579	0.002354	17.18999	928.2748
0.2779	0.1513	7.20268344	0.974343	0.058295	0.008094	18.62857	1090.145
0.3017	0.158	7.71839472	0.98672	0.016962	0.002198	19.00039	1134.096

Table E.7: BJH Calculations for local control aggregates.

P/P_0	V	Average Radius	C term	V_{pn} (Eqn25)	V/r	r_p	A_p
0.08	0.17	3.5	0.94	0.044	0.013	0.1	0.0
0.10	0.18	4.0	0.95	0.032	0.008	7.2	163.0
0.13	0.19	4.4	0.96	0.026	0.006	9.4	279.6
0.15	0.20	4.9	0.96	0.025	0.005	11.0	382.3
0.18	0.20	5.3	0.97	0.016	0.003	11.9	442.6
0.20	0.21	5.8	0.97	0.015	0.003	12.6	496.0
0.23	0.21	6.2	0.97	0.022	0.004	13.4	566.8
0.25	0.22	6.7	0.97	0.014	0.002	13.9	608.6
0.28	0.24	7.2	0.98	0.052	0.007	15.5	753.7
0.30	0.25	7.7	0.98	0.017	0.002	15.9	797.0

Table E.8: BJH Calculations for Granite aggregate (09-1939).

P/P_0	V	Average radius	C term	V_{pn} (Eqn 25)	V/r	r_p	A_p
0.08	0.0687	3.483578	0.92909589	0.031039	0.00891	7.5316815	178.2006
0.10	0.0758	3.963149	0.95995609	0.025467	0.006426	9.8811254	306.7171
0.13	0.0823	4.416003	0.95237775	0.021815	0.00494	11.361634	405.5149
0.15	0.088	4.859227	0.96180462	0.018284	0.003763	12.371008	480.7678
0.18	0.0932	5.302991	0.97510839	0.016122	0.00304	13.130013	541.5711
0.20	0.0882	5.763254	0.97251553	0.01537	0.00267	12.466578	488.2246
0.23	0.1011	6.216987	0.97458576	0.037321	0.006003	13.915276	608.2874
0.25	0.108	6.686145	0.97158303	0.019048	0.002849	14.552399	665.2645
0.28	0.1134	7.194503	0.9749948	0.014187	0.001972	14.977553	704.7043
0.30	0.1183	7.72038	0.98632192	0.012504	0.00162	15.317904	737.0956