

**EFFECTS OF ADHESION FAILURE ON
MOISTURE DAMAGE OF WARM MIX
ASPHALT CONTAINING CECABASE ADDITIVE**

MUHAMMAD RAFIQ KHAN KAKAR

UNIVERSITI SAINS MALAYSIA

2015

**EFFECTS OF ADHESION FAILURE ON MOISTURE DAMAGE OF WARM
MIX ASPHALT CONTAINING CECABASE ADDITIVE**

By

MUHAMMAD RAFIQ KHAN KAKAR

**Thesis submitted in fulfillment of the
requirements for the degree of
Doctor of Philosophy**

August 2015

I wish to dedicate this thesis to my beloved parents, brothers, sisters and Nephew Azlan Khan who have been a constant source of support and encouragement during the challenges of graduate school and life. Who are always proud of me and whom I am proud of. Without their love and support, I would not be where I am today.

I am truly thankful for having you in my life.

ACKNOWLEDGEMENTS

In The Name Of Allah S.W.T, The Most Beneficent And The Most Merciful

I would like to express my deepest appreciation and utmost sincere gratitude to my advisor, Professor Dr. Meor Othman Bin Hamzah, for his continuous guidance, motivation and encouragement during the course of my study and research at the University Sains Malaysia (USM). I am extremely grateful for your assistance and suggestions. This dissertation would not have been possible without your guidance, persistent help and financial support. I would also like to thank my co-advisor, Senior Lecturer Dr. Tye Ching Thian for her valuable guidance, suggestions and sincere support.

Moreover, I am also thankful to Dr. Jan Valentin, Faculty of Civil Engineering, Czech Technical University in Prague, Czech Republic, Dr. David Woodward, Faculty of Art Design and Build Environment, University of Ulster, Northern Ireland, United Kingdom, Associate Professor Amit Bhasin, University of Texas at Austin, USA, Associate Prof. Dr. Choong Kok Keong, School of Civil Engineering, USM, Dr. Irman Abdul Rahman, School of Applied Physics, Faculty of Science and Technology, Universiti Kebangsaan Malaysia (UKM), and Dr. Minaketan Tripathy, Faculty of Pharmacy, Universiti Teknologi Malaysia for their utmost guidance, recommendation and sincere support.

I am also grateful to the technicians of Highway Engineering Laboratory, Mr. Fauzi Ali and Mr. Zulhairi Ariffin, the technicians of Heavy Structures Laboratory at

the School of Civil Engineering, USM and Mr. Tazidi, Laboratory Assistant, Faculty of Science and Technology, UKM.

In addition, many thanks to my friends and colleagues, Dr. Mohammed Zubair, Senior Lecturer, Universiti Putra Malaysia, Sayed Abulhasan Quadri, Babak Golchin, Mohammad Nishat Akhtar, Sayed Manawar Shah and Ashar Ahmed for always being kind, helpful and sharing all my happiness and struggle throughout my research journey.

A special tribute goes to my beloved father, Late Abdul Wadood Khan Kakar for his endless support and love. Heartfelt thanks to my mother Rehana Khan Kakar and family, and every individual, who helped me in completing my PhD in asphalt technology.

“Curiosity is one of the most permanent and certain characteristics of a vigorous intellect.”

(Samuel Johnson)

TABLE OF CONTENTS

Acknowledgements	ii
Table of Contents	iv
List of Tables	xii
List of Figures	xvi
List of Plates	xxii
List of Abbreviations	xxiii
List of Symbols	xxvii
Abstrak	xxix
Abstract	xxxii
CHAPTER 1 - INTRODUCTION	
1.1 Preface	1
1.2 Problem Statement	2
1.3 Research Objectives	6
1.4 Scope of Research	6
1.5 Significance of Research	7
1.6 Thesis Organization	10
CHAPTER 2 - LITERATURE REVIEW	
2.1 Introduction	12
2.2 Background	13
2.3 Moisture Damage Mechanism	16

2.3.1	Stripping	17
2.3.2	Mechanisms of the Stripping Process	18
2.3.3	Adhesion Failure as a Major Contributing Factor	19
2.3.4	Asphalt-aggregate Interface	21
2.4	Laboratory Testing Methods	22
2.4.1	Standard and Non-standard Laboratory Test Methods	22
2.4.2	Recently Developed Testing Techniques	27
2.4.3	Issues Related to Laboratory Moisture Damage Assessment	33
2.4.4	Technical Approaches and Future Research Developments	34
2.5	Research Investigations to Evaluate Moisture Damage	36
2.5.1	Binder-aggregate Constituent Studies	36
2.5.2	Chemical and Physical Behavior	44
2.5.3	Wettability and Viscosity	45
2.5.4	Binder-aggregate Interaction	46
2.5.4.1	Surface Chemistry	46
2.5.4.2	Molecular Composition and Chemical Composition	47
2.5.4.3	Mechanical Interlock	48
2.5.4.4	Heat Energy Transfer Rate and Balance	49
2.5.5	Mixture Based Performance Studies	49
2.5.6	Moisture Damage Micro Mechanical Modeling	66
2.6	Moisture Damage in Porous Asphalt Mixture	69
2.7	Discussion	72
2.8	Summary and Future Developments	73

CHAPTER 3 - MATERIALS AND METHODS

3.1	Introduction	76
3.2	Experimental Plan	76
3.2.1	Stage 1	77
3.2.2	Stage 2	79
3.2.3	Stage 3	79
3.3	Materials	79
3.3.1	Asphalt Binder	79
3.3.2	Aggregate	80
3.3.3	Filler	82
3.3.4	Warm Mix Asphalt	82
3.4	Preparation of Cecabase-modified binder	83
3.5	Experimental Investigations	84
3.5.1	Conventional Test on Asphalt Binder and Aggregate	84
3.5.2	Asphalt Binder Aging	84
3.5.3	Rotational Viscometer	86
3.5.4	Dynamic Shear Rheometer	87
3.5.5	Mixture Testing	89
3.5.6	Gyratory Compaction	89
3.5.7	Mixture Design	90
3.5.8	Moisture Conditioning Process	91
3.5.9	Indirect Tensile Strength	92
3.5.10	Direct Tensile Strength	93
3.5.11	Universal Testing Machine	93

3.5.12	Accelerated Laboratory Vacuum Saturation	94
3.5.13	Development of Asphalt Aggregate Substrates	95
3.5.14	Pneumatic Adhesion Tensile Testing Instrument	96
3.6	Application of Image Analysis	97
3.7	Surface Free Energy Evaluation	99
3.7.1	Sessile Drop Method Using Goniometer	101
3.7.2	Wilhelmy Plate Device Method	102
3.7.3	Universal Sorption Device	103
3.8	Summary	104
CHAPTER 4 - RHEOLOGY OF BINDERS AND VOLUMETRIC PROPERTIES OF MIXTURES INCORPORATING CECABASE		
4.1	Introduction	105
4.2	Blending Temperature of Cecabase Warm Mix Additive	105
4.3	High Temperature Rheological Properties	107
4.3.1	Effects of Cecabase Content on Binder Viscosity	107
4.3.2	Effects of Cecabase Content on Construction Temperature	111
4.4	Intermediate Temperature Rheological Performance	112
4.4.1	Effects of Cecabase Content on Visco-elastic Properties	112
4.4.2	Effects of Cecabase Content on $G^*/\sin \delta$	115
4.5	Mixture Design and Volumetric Properties	117
4.6	Effects of Number of Gyration on Ease of Compaction	126
4.7	Effects of Number of Gyration on Accumulated Compaction Energy	129
4.7.1	Compaction Energy Index	134
4.8	Summary	136

CHAPTER 5 - QUANTIFICATION OF ADHESION FAILURE IN ASPHALT MIXTURES USING IMAGE ANALYSIS TECHNIQUE

5.1	Introduction	138
5.2	Specimen Preparation	142
5.2.1	Laboratory Moisture Conditioning	144
5.3	Direct Tensile Strength Test	145
5.4	Indirect Tensile Strength	147
5.5	Image Analysis	147
5.5.1	Optical Image Classification	149
5.6	Effects of Moisture Conditioning on Adhesion Failure Using Image Analysis	154
5.7	Failures Due to Broken Aggregates	157
5.8	Effects of Moisture Conditioning on Direct Tensile Strength	159
5.9	Statistical Analysis on Direct Tensile Test Results	161
5.10	Indirect Tensile Strength and Image Analysis	163
5.11	Effects of Moisture Conditioning on Adhesion Failure using Indirect Tensile Strength	164
5.12	Failures due to Broken Aggregate using Indirect Tensile Strength Test	167
5.13	Effects of Moisture Conditioning on Indirect Tensile Strength	167
5.14	Statistical Analysis on Indirect Tensile Test Results	170
5.15	Correlation Between Percent Adhesion Failure of DTT and ITS	172
5.16	Summary	175

CHAPTER 6 - EFFECTS OF ADHESION FAILURE ON ASPHALT
AGGREGATE SUBSTRATE DIRECT TENSILE AND PULL OFF STRENGTH

6.1	Introduction	178
6.2	Preparation of Aggregate Substrates and Asphalt	181
6.2.1	Coring and Cutting Process of Aggregate Boulders	181
6.2.2	Fabrication of Aggregate Substrate Molds	183
6.2.3	Accelerated Laboratory Moisture Damage Conditioning Process	187
6.3	Image Analysis on Binder-aggregate Substrates using Direct Tensile Test	188
6.3.1	Effects of Moisture Conditioning and Binder Aging on Adhesion Failure Using Image Analysis Technique	190
6.3.2	Effects of Moisture Conditioning and Binder Aging on Direct Tensile Strength	194
6.4	Statistical Analysis of Direct Tensile Aggregate Substrate Test Results	197
6.5	Pull-Off Tension Test	201
6.5.1	Effects of Moisture Conditioning and Binder Aging on Adhesion Failure Using Image Analysis Technique	204
6.5.2	Effects of Moisture Conditioning and Binder Aging on Bitumen Bond Strength	208
6.6	Statistical Analysis of Pull-off Tension Test Results	210
6.7	Correlation Between Percent Adhesion Failure in Binder Aggregate Substrate and Pull-off Tension Test Results	214
6.8	Discussion	219
6.9	Summary	220

CHAPTER 7 - EVALUATION OF WARM MIX ASPHALT MOISTURE
SENSITIVITY USING SURFACE FREE ENERGY

7.1	Introduction	223
7.2	Selection of Test Methods to Evaluate SFE of Binder and Aggregate	224
7.2.1	Evaluation of Asphalt Binder SFE Using Contact Angle Device	225
7.2.2	Evaluation of Asphalt Binder SFE Using Dynamic Contact Angle Analyzer	227
7.2.3	Evaluation of Aggregate SFE Using Universal Sorption Device	229
7.3	Preparation of Goniometer Test Specimen	231
7.4	Sessile Drop Method	232
7.5	Preparation of Wilhelmy Plate Device Test Specimen	234
7.6	Selection of Probe Liquid	234
7.7	Algorithm Developed for the Calculation of SFE Components	236
7.7.1	Pseudo Code	237
7.8	Work of Adhesion	240
7.9	Coefficient of Spreadability	241
7.10	Compatibility Ratio	241
7.11	Results of Sessile Drop Method Using Goniometer	242
7.11.1	Effects of Surfactant-based Additive on the Work of Adhesion	245
7.11.2	Effects of Surfactant-based Additive on the Spreadability Coefficient	247
7.11.3	Effects of Surfactant-based Additive on the Compatibility Ratio	248

7.12	Dynamic Wilhelmy Plate Device Test Result	250
7.12.1	Effects of Surfactant-based Additive on the Work of Adhesion	257
7.12.2	Effects of Surfactant-based Additive on the Spreadability Coefficient	258
7.12.3	Effects of Surfactant-based Additive on the Compatibility Ratio	260
7.13	Summary	262
CHAPTER 8 - CONCLUSIONS AND RECOMMENDATIONS		
8.1	Conclusions	264
8.2	Recommendations	268
LIST OF REFERENCES		270
APPENDIX A ASPHALT BINDER AND MIXTURE EXPERIMENTAL DATA		291
APPENDIX B QUANTIFICATION OF MIXTURE ADHESION FAILURE AND IMAGES		327
APPENDIX C BINDER AGGREGATE SUBSTRATE ADHESION FAILURE RESULTS AND IMAGES		362
APPENDIX D SURFACE FREE ENERGY COMPONENTS EXPERIMENTAL DATA		401
LIST OF PUBLICATIONS		406

LIST OF TABLES

		Pages
Table 2.1	Mechanisms of Stripping in Asphalt Mixtures (Talyor and Khosla, 1983)	19
Table 2.2	Details of Standard and Non-Standard Laboratory Test Approaches	24
Table 2.3	Approaches Used for Aggregate-binder Constituent Moisture Damage	29
Table 3.1	Properties of PG-64 and PG-76 binders	80
Table 3.2	Physical Properties of Aggregates	81
Table 3.3	Physical and Chemical Properties of PMD and OPC	82
Table 3.4	Physical and Chemical Properties of WMA Additive	83
Table 3.5	Dosage, Mixing Time and Temperature of Additive	84
Table 3.6	Mixture Design Properties for Mix Type AC 14 (PWD, 2008)	91
Table 4.1	Dosage, Mixing Time and Temperature of Additive (DTT and ITS mixture)	106
Table 4.2	ANOVA Results Showing the Effect of Viscosity on Unaged PG-64 Binder	110
Table 4.3	ANOVA Results Showing the Effect of Viscosity on Short Term Aged PG-64 Binder	110
Table 4.4	ANOVA Results Showing the Effect of Viscosity on Unaged PG-76 Binder	111
Table 4.5	ANOVA Results Showing the Effect of Viscosity on Short Term Aged PG-64 Binder	111
Table 4.6	One-way ANOVA on the Effects of Cecabase Content on G*	115

Table 4.7	G*/sin δ (kPa) of Unaged and STA Binders at Different Temperature	116
Table 4.8	HMA and WMA Construction Temperatures of Mixture Design	119
Table 4.9	Regression Results on Accumulated Compaction Energy and Compaction Energy Indices (PG-64 Binder)	133
Table 4.10	Regression Results on Accumulated Compaction Energy and Compaction Energy Indices (PG-76 Binder)	134
Table 4.11	ANOVA Results Showing Effects of Cecabase Content on Degree of Compaction (PG-64 Mixture)	135
Table 4.12	ANOVA Results Showing Effects of Cecabase Content on Degree of Compaction (PG-76 Mixture)	136
Table 5.1	Mixing and Compaction Temperatures of Asphalt Mixtures	143
Table 5.2	Mixture Designation (DTT and ITS)	144
Table 5.3	Kappa Coefficient Using Minimum and Maximum Likelihood Classification	154
Table 5.4	Two-Way ANOVA on the Effects of the Percent Adhesion Failure (PG-64 Mixture)	162
Table 5.5	Two-Way ANOVA on the Effects of the Percent Adhesion Failure (PG-76 Mixture)	162
Table 5.6	t-Test: Paired Two Sample for Means (DTT, Dry)	163
Table 5.7	t-Test: Paired Two Sample for Means (DTT, 1 F-T)	163
Table 5.8	t-Test: Paired Two Sample for Means (DTT, 3 F-T)	163
Table 5.9	Two-way ANOVA on the Effect of the Percent Adhesion Failure (PG-64 Mixture)	170
Table 5.10	Two-way ANOVA on the Effect of the Percent Adhesion Failure (PG-76 Mixture)	171
Table 5.11	t-Test: Paired Two Sample for Means (ITS, Dry)	171
Table 5.12	t-Test: Paired Two Sample for Means (ITS, 1 F-T)	171

Table 5.13	t-Test: Paired Two Sample for Means (ITS, 3 F-T)	171
Table 6.1	Mixture Designation (AST and PATTI Test)	187
Table 6.2	A Two-way ANOVA on the Effects of Percent Adhesion Failure (PG-64 Binder and Granite, AST)	199
Table 6.3	A Two-way ANOVA on the Effects of Percent Adhesion Failure (PG-76 Binder and Granite, AST)	199
Table 6.4	A Two-way ANOVA on the Effects of Percent Adhesion Failure (PG-64 Binder and Limestone, AST)	199
Table 6.5	A Two-way ANOVA on the Effects of Percent Adhesion Failure (PG-76 Binder and Limestone, AST)	199
Table 6.6	t-Test: Paired Two Sample for Means (AST Granite, Unconditioned)	200
Table 6.7	t-Test: Paired Two Sample for Means (AST Granite, Conditioned)	200
Table 6.8	t-Test: Paired Two Sample for Means (AST Limestone, Unconditioned)	200
Table 6.9	t-Test: Paired Two Sample for Means (AST Limestone, Conditioned)	200
Table 6.10	A Two-way ANOVA on the Effects of Percent Adhesion Failure (PG-64 Binder and Granite, PATTI)	212
Table 6.11	A Two-way ANOVA on the Effects of Percent Adhesion Failure (PG-76 Binder and Granite, PATTI)	212
Table 6.12	A Two-way ANOVA on the Effects of Percent Adhesion Failure (PG-64 Binder and Limestone, PATTI)	212
Table 6.13	A Two-way ANOVA on the Effects of Percent Adhesion Failure (PG-76 Binder and Limestone, PATTI)	212
Table 6.14	t-Test: Paired Two Sample for Means (PATTI Granite, Unconditioned)	213
Table 6.15	t-Test: Paired Two Sample for Means (PATTI Granite, Conditioned)	213

Table 6.16	t-Test: Paired Two Sample for Means (PATTI Limestone, Unconditioned)	213
Table 6.17	t-Test: Paired Two Sample for Means (PATTI Limestone, Conditioned)	213
Table 6.18	Correlation Between AST and PATTI	215
Table 7.1	Surface Free Energy Characteristics of Probe Liquids	236
Table 7.2	Surface Free Energy Characteristics of Aggregate	236
Table 7.3	Contact Angle Results (Example Solution)	239
Table 7.4	Contact Angle of Asphalt Binders Measured With Goniometer	243
Table 7.5	SFE Components of Asphalt Binders Using Goniometer Contact Angles	244
Table 7.6	Advancing Contact Angle of Asphalt Binders (DWPD)	252
Table 7.7	Receding Contact Angle of Asphalt Binders (DWPD)	253
Table 7.8	SFE Components of Asphalt Binders (DWPD Advancing Contact Angle)	254
Table 7.9	SFE components of asphalt binders (DWPD Receding Contact Angle)	255

LIST OF FIGURES

		Pages
Figure 2.1	Different Aspects of Moisture Damage Standardization	34
Figure 2.2	Asphalt Pavement Moisture Damage Process	35
Figure 2.3	Chemical Nature of Road Aggregates (Akzo Nobel, Surface chemistry)	47
Figure 2.4	Pore Pressure Histories at Different Depths (Dong et al., 2007)	51
Figure 2.5	Sketch Map of Specimens Subject to the Coupled Water/loading Action (Solaimanian et al., 2007)	53
Figure 2.6	Typical 3D Image of Extracted Air Voids (Khan et al., 2013)	66
Figure 2.7	Failure Modes in Porous Asphalt (Kringos, 2007)	70
Figure 2.8	Simplified Schematic of Life Time Optimization Tool (LOT) Design Strategy (Huurman et al., 2010)	71
Figure 3.1	Research Methodology Flowchart	78
Figure 3.2	Aggregate Gradation, JKR AC14 (PWD, 2008)	81
Figure 3.3	Differences in Intermolecular Forces (Little et al., 2006)	100
Figure 4.1	Rotational Viscosity versus Temperature (PG-64 Binder)	108
Figure 4.2	Rotational Viscosity versus Temperature (PG-76 Binder)	109
Figure 4.3	Aging Index of Hot and Warm Mix Asphalt	109
Figure 4.4	Dynamic Shear Modulus and Phase Angle of Cecabase Modified Binders	114
Figure 4.5	Mixture Design of HMA (PG-64 binder)	120
Figure 4.6	Mixture Design of WMA (PG-64 binder, 0.2%)	121
Figure 4.7	Mixture Design of WMA (PG-64 binder, 0.4%)	122

Figure 4.8	Mixture design of HMA (PG-76 binder)	123
Figure 4.9	Mixture design of WMA (PG-64 binder, 0.2%)	124
Figure 4.10	Mixture design of WMA (PG-76 binder, 0.4%)	125
Figure 4.11	Degree of Compaction Using PG-64 Binder (HMA Compacted at 150°C)	128
Figure 4.12	Degree of Compaction Using PG-76 Binder (HMA Compacted at 170°C)	129
Figure 4.13	Accumulated Compaction Energy Using PG 64 Binder (HMA Compacted At 150°C)	131
Figure 4.14	Accumulated Compaction Energy Using PG 76 Binder (HMA Compacted At 170°C)	132
Figure 4.15	Compaction Energy Index of Hot and Warm Mix Asphalt	135
Figure 5.1	Sample Fixed with Special-Designed Molding Cap Failed in Direct Tension	146
Figure 5.2	Original Images of WO130 Fractured Surfaces (DTT)	148
Figure 5.3	Transformed Images of WO130 Fractured Surfaces (DTT)	148
Figure 5.4	Theme Map of WO130 Fractured Surfaces	153
Figure 5.5	Adhesion Failure Results of Image Analysis on Fractured Surfaces (DTT)	155
Figure 5.6	Percent Broken Aggregate on Surfaces Fractured by DTT	158
Figure 5.7	Direct Tensile Strength Results of Fractured Samples	160
Figure 5.8	Original Images of WO150 Fractured Surfaces (ITS)	164
Figure 5.9	Transformed Color Images of WO130 Fractured Surfaces (ITS)	164
Figure 5.10	Adhesion Failure Results of Image Analysis on Fractured Surfaces (ITS)	166
Figure 5.11	Percent Broken Aggregate on Surfaces Fractured by ITS	168

Figure 5.12	Direct Tensile Strength Results of Fractured Samples	169
Figure 5.13	Correlation Between Percent Adhesion Failure of DTT and ITS (Dry, PG-64 Mixtures)	172
Figure 5.14	Correlation Between Percent Adhesion Failure of DTT and ITS (1 F-T, PG-64 Mixtures)	173
Figure 5.15	Correlation Between Percent Adhesion Failure of DTT and ITS (3 F-T, PG-64 Mixtures)	173
Figure 5.16	Correlation Between Percent Adhesion Failure of DTT and ITS (Dry, PG-76 Mixtures)	174
Figure 5.17	Correlation Between Percent Adhesion Failure of DTT and ITS (1 F-T, PG-76 Mixtures)	174
Figure 5.18	Correlation Between Percent Adhesion Failure of DTT and ITS (3 F-T, PG-76 Mixtures)	175
Figure 6.1	Detailed Drawing-I of Fabricated Aggregate Substrate Mold	184
Figure 6.2	Detailed Drawing-II of Fabricated Aggregate Substrate Mold	185
Figure 6.3	Original Images of PG-64 Binders on Granite Aggregate Substrate	189
Figure 6.4	Transformed Images of PG-64 Binders on Granite Aggregate Substrate	189
Figure 6.5	Original Images of PG-64 Binders on Granite Aggregate Substrate	189
Figure 6.6	Transformed Images of PG-64 Binders on Granite Aggregate Substrate	189
Figure 6.7	Percent Adhesion Failure of DTT Results on Granite Substrates	192
Figure 6.8	Percent Adhesion Failure of DTT Results on Limestone Substrates	194
Figure 6.9	Direct Tensile Strength Test Results of Granite Substrates	196
Figure 6.10	Direct Tensile Strength Test Results of Limestone Substrates	197

Figure 6.11	Granite and Limestone Aggregate Substrates Prepared for PATTI Test	202
Figure 6.12	Original Images of PG-64 Binders on Granite Aggregate Substrate	203
Figure 6.13	Transformed Images of PG-64 Binders on Granite Aggregate Substrate	203
Figure 6.14	Original Images of PG-64 Binders on Granite Aggregate Substrate	203
Figure 6.15	Transformed Images of PG-64 Binders on Granite Aggregate Substrate	203
Figure 6.16	Percent Adhesion Failure of PATTI Test Results on Granite Substrate	206
Figure 6.17	Percent Adhesion Failure of PATTI Test Results on Limestone Substrate	207
Figure 6.18	Bitumen Bond Strength Test Results of Granite Substrate	209
Figure 6.19	Bitumen Bond Strength Test Results of Limestone Substrate	210
Figure 6.20	Correlation between Percent Adhesion Failure of AST and PATTI (PG-64 Binder, Limestone Aggregate, Unconditioned)	215
Figure 6.21	Correlation between Percent Adhesion Failure of AST and PATTI (PG-64 Binder, Limestone Aggregate, Conditioned)	216
Figure 6.22	Correlation between Percent Adhesion Failure of AST and PATTI (PG-64 Binder, Granite Aggregate, Unconditioned)	216
Figure 6.23	Correlation between Percent Adhesion Failure of AST and PATTI (PG-64 Binder, Granite Aggregate, Conditioned)	217
Figure 6.24	Correlation between Percent Adhesion Failure of AST and PATTI (PG-76 Binder, Limestone Aggregate, Unconditioned)	217
Figure 6.25	Correlation between Percent Adhesion Failure of AST and PATTI (PG-76 Binder, Limestone Aggregate, Conditioned)	218
Figure 6.26	Correlation between Percent Adhesion Failure of AST and PATTI (PG-76 Binder, Granite Aggregate, Unconditioned)	218

Figure 6.27	Correlation between Percent Adhesion Failure of AST and PATTI (PG-76 Binder, Granite Aggregate, Conditioned)	219
Figure 7.1	Young's Contact Angle Formula	226
Figure 7.2	Metallic Strip and Hook Designed for DCA Test Specimen	228
Figure 7.3	Schematic Layout of the Universal Sorption Device (USD) (Little et al., 2006)	230
Figure 7.4	Typical Adsorption Isotherm Measured Using USD (Little et al., 2006)	231
Figure 7.5	Asphalt Binder Samples and Binder Coated Glass Slides	232
Figure 7.6	Schematic Layout for The Sessile Drop Method (Little et al., 2006)	233
Figure 7.7	Output Snapshot of the Developed Algorithm Using Microsoft Visual Studio	240
Figure 7.8	Total Surface Free Energy of Asphalt Binder in Unaged Condition	245
Figure 7.9	Work of Adhesion Energy of PG-64 Asphalt Binder	246
Figure 7.10	Work of Adhesion Energy of PG-76 Asphalt Binder	246
Figure 7.11	Spreadability Coefficient of PG-64 Asphalt Binder	248
Figure 7.12	Spreadability Coefficient of PG-76 Asphalt Binder	248
Figure 7.13	Compatibility Ratio of PG-64 Asphalt Binder	249
Figure 7.14	Compatibility Ratio of PG-76 Asphalt Binder	250
Figure 7.15	Correlation Between Surface Free Energy (Advancing)	256
Figure 7.16	Correlation Between Surface Free Energy (Receding)	256
Figure 7.17	Work of Adhesion Energy of PG-64 Asphalt Binder (DWPD)	258
Figure 7.18	Work of Adhesion Energy of PG-76 Asphalt Binder (DWPD)	258

Figure 7.19	Spreadability Coefficient of PG-64 Asphalt Binder (DWPD)	260
Figure 7.20	Spreadability Coefficient of PG-76 Asphalt Binder (DWPD)	260
Figure 7.21	Compatibility Ratio of PG-64 Asphalt Binder (DWPD)	262
Figure 7.22	Compatibility Ratio of PG-76 Asphalt Binder (DWPD)	262

LIST OF PLATES

		Pages
Plate 3.1	RTFO Oven to Simulate Short Term Aging of Asphalt Binder	85
Plate 3.2	PAV and Vacuum Degassing Oven to Simulate Long Term Aging of Binder	86
Plate 3.3	Brookfield Rotational Viscometer	87
Plate 3.4	DSR And Temperature Control Unit	88
Plate 3.5	Servopac Gyrotory Compactor	90
Plate 3.6	Vacuum Saturation of Asphalt Mixture	92
Plate 3.7	Shimadzu Universal Testing Machine Used to Conduct DTS test	94
Plate 3.8	Accelerated Laboratory Vacuum Saturator	95
Plate 3.9	Pneumatic Pull-off Tensile Testing Instrument with Modified Stub (Moraes et al., 2011)	96
Plate 3.10	Goniometer Device Used to Measure Contact Angle	101
Plate 3.11	Metallic Strip Coated with Binder	102
Plate 3.12	Dynamic Contact Angle Device	103
Plate 6.1	Pneumatic Adhesion Tensile Testing Instrument	181
Plate 6.2	Core Cutting Machine	182
Plate 6.3	Diamond Cutter	183
Plate 6.4	Stone Polishing Machine	183
Plate 6.5	Granite and Limestone Aggregate Substrates (50 mm Diameter)	185
Plate 6.6	Aggregate Substrates Fixed in Moldings	186

LIST OF ABBREVIATIONS

ALVS	Accelerated Laboratory Vacuum Saturator
ACV	Aggregate Crushing Value
AST	Aggregate Substrate Test
AI	Aging Index
AASHTO	American Association of State Highway and Transportation Officials
ASTM	American Society for Testing Materials
ANOVA	Analysis of Variance
ANA	Anti-Stripping Agent
APA	Asphalt Pavement Analyzer
AAPT	Association of Asphalt Paving Technologist
BBS	Bitumen Bond Strength
CEI	Compaction Energy Index
CR	Compatibility Ratio
CT	Computerised Tomography
CN	Condition Number
CA	Contact Angle
CAA	Coarse Angular Aggregates
DTS	Direct Tensile Strength
DTT	Direct Tension Test
DMA	Dynamic Mechanical Analyzer
DSR	Dynamic Shear Rheometer
DWP	Dynamic Wilhelmy Plate

DWPD	Dynamic Wilhelmy Plate Device
ENVI	Environment for Visualizing Images
ECS	Environmental Conditioning System
FHWA	Federal Highway Administration
FTIR	Fourier Transform Infrared Spectroscopy
F-T	Freeze and Thaw
GPR	Ground Penetrating Radar
GVOC	Good-Van-Oss–Chaudhury
g	Gravitational Force
GHG	Greenhouse Gas
HWTD	Hamburg Wheel-Tracking Device
HMA	Hot Mix Asphalt
ITS	Indirect Tensile Strength
ITSR	Indirect Tensile Strength Ratio
ITT	Indirect Tension Test
IGC	Inverse Gas Chromatography
JKR	Jabatan Kerja Raya
LTA	Long Term Aging
MEPDG	Mechanistic Empirical Pavement Design Guide
MPa	Mega Pascal
MIST	Moisture Induced Stress Tester
NAPA	National Asphalt Pavement Association
NCAT	National Center for Asphalt Technology
NCHRP	National Cooperative Highway Research Program

NMAS	Nominal Maximum Aggregate Size
NMR	Nuclear Magnetic Resonance
OBC	Optimum Binder Content
OPC	Ordinary Portland Cement
PMD	Pavement Modifier
PI	Penetration Index
PG	Performance Grade
PATTI	Pneumatic Adhesion Tensile Testing Instrument
PSV	Polished Stone Value
PMB	Polymer Modified Bitumen
PA	Porous Asphalt
PAV	Pressure Aging Vessel
PWD	Public Works Department
RoAM	Raveling of Asphalt Mixes
RAP	Reclaimed Asphalt Pavement
ROI	Region of Interest
RSM	Response Surface Methodology
RTFO	Rolling Thin Film Oven
RV	Rotational Viscosity
SATS	Saturated Aging Tensile Stiffness
SEM	Scanning Electron Microscope
STA	Short Term Aging
SC	Spreading Coefficient
SBS	Styrene-Butadiene-Styrene

SEAM	Sulfur Extended Asphalt Modified
SFE	Surface Free Energy
TTS	Tack Test System
MMLS3	Third-Scale Model Mobile Load Simulator
TRB	Transportation Research Board
UK	United Kingdom
USD	Universal Sorption Device
UTM	Universal Testing Machine
UKM	Universiti Kebangsaan Malaysia
USM	Universiti Sains Malaysia
UiTM	Universiti Teknologi Mara
VFA	Voids Filled Asphalt
VMA	Voids in Mineral Aggregates
WRI	Western Research Institute

LIST OF SYMBOLS

γ^{AB}	Acid-Base Component
ρ_{air}	Air Density
G_{mb}	Bulk Specific Gravity
$CaCO_3$	Calcium Carbonate
cP	Centipoise
G^*	Complex Shear Modulus
CT	Computerised Tomography
$^{\circ}C$	Degree Celsius
ρ_L	Density of The Liquid
ΔF	Difference Between Weight of Plate in air and Partially Submerged in Probe Liquid
γ^+	Lewis Acid Component of Surface Interaction
γ^-	Lewis Base Component of Surface Interaction
γ^{LW}	Lifshitz-Van Der Waals Component
E^*	Modulus of Elasticity
P_t	Perimeter of The Binder Coated Strip
δ	Phase Angle
M_r	Resilient Modulus
SiO_2	Silica
Na_2CO_3	Sodium Carbonate
$G^*/\sin \delta$	Superpave Rutting Factor
G_{mm}	Theoretical Maximum Density
γ^T	Total Surface Energy

γ_L	Total Surface Energy of The Probe Liquid
V_{im}	Volume of Solid Immersed in the Liquid
W	Work of Adhesion
W_{AS}^{dry}	Work of Adhesion In Dry
W_{WAS}^{wet}	Work of Adhesion In Wet
W_{AA}	Work of Cohesion of Binder

KESAN KEGAGALAN REKATAN KE ATAS KEROSAKAN LEMBAPAN ASFALT BERSUHU SEDERHANA YANG MENGANDUNGI CECABASE

ABSTRAK

Campuran asfalt bersuhu sederhana (WMA) adalah teknologi yang membolehkan pengurangan ketara suhu pencampuran dan pemadatan campuran asfalt panas lazim. Teknologi ini boleh menjimatkan kos, meningkatkan keboleherjaan, mengurangkan kesan pengeluaran gas rumah hijau dan mesra alam. Walau bagaimanapun, WMA mudah terdedah kepada kerosakan lembapan sebagai akibat suhu pengeluaran yang lebih rendah. Hal yang demikian menyebabkan kegagalan rekatan, dan seterusnya pelucutan pengikat asfalt daripada agregat. Dalam kajian ini, bahan tambah campuran suam Cecabase digunakan untuk menurunkan suhu pengeluaran dan meningkatkan keboleh-rekatan asfalt dengan agregat. Pengikat jenis PG-64 dan PG-76 digunakan untuk menyediakan spesimen ujian. Bertindak sebagai surfaktan apabila dicampurkan dengan pengikat asfalt, Cecabase menggalakkan rekatan pada antara muka pengikat asfalt dan agregat. Keputusan ujian makmal secara keseluruhannya menunjukkan bahawa penambahan Cecabase tidak memberi kesan yang ketara ke atas reologi bahan pengikat dan kandungan pengikat optimum. Pendekatan baru melalui analisis imej digunakan untuk mengklasifikasikan kerentanan kegagalan rekatan dalam campuran asfalt sebagai akibat kerosakan lembapan. Hasil kajian menunjukkan kegagalan rekatan meningkat dengan bilangan kitaran beku dan cair dan campuran yang mengandungi pengikat PG-76 mempamerkan kegagalan rekatan yang lebih rendah berbanding pengikat PG-64. Ujian tegangan langsung substrat pengikat-agregat dan ujian tarik-keluar dijalankan untuk menilai kegagalan rekatan. Peralatan makmal vakum tepu (ALVS)

penyesuaian lembapan digunakan untuk menyediakan spesimen pengikat-agregat. Keputusan ujian menunjukkan pengikat asphalt yang didedahkan kepada pengusiaan jangka pendek dan jangka panjang, lebih mudah terdedah kepada kerosakan lembapan apabila ditindaki ALVS. Untuk mendapatkan gambaran asas, Tenaga Permukaan Bebas (SFE) pengikat terubahsuai Cecabase dinilai menggunakan sudut sentuh Goniometer dan peranti plat dinamik Wilhelmy. Pengukuran analitik berdasarkan keputusan SFE menunjukkan Cecabase meningkatkan kebolelsebaran pengikat untuk lebih mudah menyaluti permukaan zarah agregat. Tambahan pula, kerja rekatan meningkat dengan penambahan Cecabase. Nisbah keserasian menggambarkan pengaruh lembapan dan menunjukkan bahawa rintangan terhadap kegagalan lembapan agregat granit adalah lebih rendah berbanding agregat batu kapur.

EFFECTS OF ADHESION FAILURE ON MOISTURE DAMAGE OF WARM MIX ASPHALT CONTAINING CECABASE ADDITIVE

ABSTRACT

Warm Mix Asphalt (WMA) is a technology that allows significant reduction in mixing and compaction temperatures of conventional hot mix asphalt. It is a cost effective technology that can improve mixture workability, reduces greenhouse gas emissions, and is environmental friendly. However, WMA is susceptible to moisture damage due to its lower production temperature. This can cause adhesion failure, hence stripping of asphalt binder from the aggregates. In this research, Cecabase warm mix additive was used to lower the production temperature and enhance the asphalt binder adhesion properties with aggregate. Two binders, PG-64 and PG-76, were used to prepare the test specimens. As a surfactant and when blended with asphalt binder, Cecabase promotes adhesion at the binder-aggregate interface. Therefore, the overall laboratory test results showed that addition of Cecabase had no significant effects on binder rheology and optimum binder content. A novel approach using image analysis was used to measure the asphalt mixture adhesion failure susceptibility due to moisture damage. The results showed that adhesion failure increased with the number of freeze and thaw cycles and mixtures prepared with PG-76 binder exhibited lower adhesion failure compared to PG-64 binder. To assess the adhesion failure, binder-aggregate substrate direct tensile and pull-off tension tests were carried out. An accelerated laboratory vacuum saturator (ALVS) moisture conditioning was fabricated to condition the binder-aggregate specimens. The results indicated that short term and long term aged binders when subjected to

ALVS, were susceptible to moisture damage. In order to gain fundamental insight, the Surface Free Energy (SFE) of Cecabase-modified binder was evaluated using contact angle Goniometer and dynamic Wilhelmy plate device. The analytical measurements based on SFE results showed that Cecabase improved the spreadability of asphalt binder over the limestone aggregate particles. In addition, the work of adhesion improved with the addition of Cecabase. The compatibility ratio is an indicator of moisture susceptibility and indicated that the granite aggregates were less resistant to moisture damage compared to limestone aggregates.

CHAPTER 1

INTRODUCTION

1.1 Preface

Warm mix asphalt (WMA) is gaining increasing popularity all over the world. It is used to lower the production and laying temperature of asphalt mixtures. In addition, it has several advantages such as very low environmental impact in terms of green-house gas emission, less energy required during mixing and can also be compacted in cooler conditions (Hamzah et al., 2014). Another major benefit is that the workers involved in production and laying process are not exposed to toxic fumes compared to conventional hot mix asphalt.

There are three main methods to reduce production and laying temperatures of HMA (Capitão et al., 2012). Firstly, reducing binder viscosity using foaming processes, which can either be water-based (direct method technologies) or water-containing (indirect method technologies) (Rubio et al., 2012b). Secondly, usage of organic or synthetic additives such as Sasobit and Asphamin increases wax content in the binder to reduce viscosity. The third method incorporates chemical additives such as Cecabase® to modify binders, which contains combinations of emulsification agents, surfactants, polymers and adhesion promoter or also known as anti-stripping agents. Such additives help to improve coating of the aggregate particles, workability and ease of compaction (Rubio et al., 2012b).

Of these three main methods, the use of chemical additives has been found to be more practical and convenient as they can be added directly to the bitumen prior to mixing without any modification of the asphalt plant. It is also claimed that unlike foaming or organic based WMA additives, the chemical surfactant-based additive

does not significantly affect the mechanical and rheological properties of the bitumen, mixture stiffness and low temperature properties (Oliveira et al., 2012).

In asphalt mixtures, the bitumen binds the aggregates particles together and transfers the traffic loading stresses during its service life. Good adhesion or bonding between bitumen and aggregate surface is therefore very important and any mechanism that reduces this bond will reduce the life of the asphalt mixture layer. The presence of water either contained within the aggregate particle or external to the bitumen coated particle is probably the main cause for failures at this critical interface (Bhasin and Little, 2009).

One of the main concerns relating to the durability of WMA is the potential for water or moisture induced failure of the bond. This is primarily due to issues with the lower temperatures involved during mixing that may not adequately reduce the aggregate moisture contents to an acceptable level. Despite extensive research to understand the behavior of WMA and its obvious beneficial characteristics, the risk of moisture damage related failure still remains a problem and requires further investigations.

1.2 Problem Statement

Asphalt pavements are exposed to environmental conditions soon after its construction. The environmental conditions such as the effects of moisture on the effectiveness of asphalt mixtures are the main causes of distress. A detailed review on the identification of moisture damage in asphalt mixtures reveals that there are no established test methods that can be used to quantify moisture damage which truly reflects the materials susceptibility to moisture damage.

Since the WMA are produced and laid down at lower temperature compared to HMA, there is more chance of moisture damage during its service life. Research shows that due to the insufficient drying of aggregates during the low temperature production of WMA, some moisture remained trapped in aggregates that are responsible for WMA moisture damage. In addition, the trapped moisture can diffuse through binder onto the asphalt aggregate interface and ultimately causing the stripping of asphalt binder and this needs an in-depth investigation.

Generally, moisture damage in asphalt mixtures can be determined based on the quantitative or qualitative measurements. The quantitative evaluation is based on the mechanical strength properties such as indirect tensile and direct tensile strengths of compacted asphalt mixtures. On the other hand, the qualitative measure depends on the assessment of loose mixtures and its resistance against moisture damage is evaluated in terms of visually ranked adhesion failure. The quantitative and qualitative parameters are very important to address the material susceptibility to moisture damage. Therefore, in the context of asphalt mixture subjected to moisture damage, the use of only compacted material avoiding loose mix requires additional tools to address both parameters. A novel method using image analysis technique was adopted which fractured the compacted specimen in direct tensile or indirect tensile followed by examining the fractured surface. This method provides a more precise quantification compared to the conventional method of visual inspection. Such a development can facilitate to test both qualitative and quantitative properties using only one material, in addition to saving time and cost involved in the preparation and testing of loose mix.

The adhesion failure or adhesive bond strength of asphalt mixture components that are asphalt binder and aggregate can be determined using the

Pneumatic Adhesion Tensile Testing Instrument (PATTI) as modified by Kantipong and Bahia (2003), Kantipong and Bahia (2004), Kantipong and Bahia (2005). The test evaluates the susceptibility of asphalt binder to adhesion failure at a defined thickness. The pull-off stubs are designed to test the binder specimens over the aggregate surface. The reduction in pull-off strength due to moisture conditioning is referred to as moisture damage evaluation of asphalt binder over aggregate. The failed surfaces are further evaluated visually to classify the mode of failures that are “Cohesive, Adhesive or Cohesive and Adhesive”.

The precise measurement of percent adhesion failure can be more viable to predict material behavior towards moisture damage susceptibility in terms of adhesion failure. The use of image analysis technique can quantify the adhesion failure more precisely. Therefore, it provides additional parameter to determine the moisture susceptible materials in terms of adhesion failure quantification. In order to identify the adhesion failure at material constituent level, the binder-aggregate substrates direct tension test is performed. This can simulate the actual field conditions where the aggregates are coated by the binder or mastic.

The phenomenon of stripping is mainly dependent on the asphalt binder and aggregate individual properties (Emery and Seddik, 1997). The types of aggregate used in asphalt production are varied and reflect the many different types available. A simple classification is based on how they were formed that are igneous, sedimentary and metamorphic. Within each of these there are many different types that can be described in terms of their overall morphology, mineralogy, grain size and degree of weathering. With regards to most aggregate and bitumen research, morphology is typically used to describe the aggregate.

The surface of different types of aggregate exhibits different chemical affinities with bitumen. For example, aggregates with higher SiO₂ contents such as granite and quartz are typically acidic. These aggregates are classified as hydrophilic and due to their affinity to water, they are difficult to coat with bitumen. In contrast, basic aggregates with high CaCO₃ content such as limestone, are hydrophobic. They tend to repel water and be less affected by moisture induced problems (Tarrer and Wagh, 1991).

Terminologies such as wettability and adhesion are used to explain the bitumen and aggregate interface mechanism (Wasiuddin et al., 2008). The interface can be explained using analytical terminologies such as wettability of bitumen over aggregate, work of adhesion and solubility of adhesion bond. With respect to the chemical interactions occurring at the interface, the polar (hydrophilic) and non-polar (hydrophobic) nature of aggregate and bitumen control the wettability of bitumen over aggregate. Most types of bitumen are considered to be non-polar. Most basic and acid aggregate types have high polarity surfaces (Wasiuddin et al., 2010). Therefore, it is difficult to wet a polar aggregate surface with most non-polar types of bitumen. The wettability of most non-polar types of bitumen over polar aggregate can be improved by altering the aggregate surface from being polar to become non-polar that is hydrophilic to hydrophobic. This is best done by reducing the bitumen non-polar component and the polar component of the aggregate. The surface energy is considered as a useful tool to classify constituent materials more resistant against moisture damage (Cheng, 2002). Hence, it is important to consider the SFE characteristics of Cecabase modified binders due to its surfactant based properties.

1.3 Research Objectives

The objectives of the research are as follows:

1. To determine the moisture resistance of warm mix asphalt subjected to laboratory accelerated moisture conditioning incorporating Cecabase.
2. To quantify the adhesion failure at asphalt-aggregate interface using mixture, aggregate substrate and pull-off tensile strengths.
3. To assess the extent of adhesion failure using the image analysis technique and its application in asphalt stripping evaluation.
4. To evaluate the surface free energy characteristics of Cecabase modified binders under different aging conditions and its effects on the wettability of bitumen over aggregate, work of adhesion and the compatibility ratio.

1.4 Scope of Research

The scope of the research is limited to a study on the effects of moisture damage of asphalt mixtures incorporating Cecabase. The asphalt mixtures were prepared using crushed granite aggregate according to the Malaysian Public Works Department (PWD) gradation specifications for AC 14. The asphalt binder-aggregate constituent studies were conducted using aggregate substrates prepared with granite and limestone boulder. PG-64 and PG-76 binders were selected for the preparation of asphalt mixtures and binder-aggregate constituent specimens. Asphalt mixtures were compacted using Servopac gyratory compactor. The characterization of asphalt binders was made using a Rotational Viscometer (RV) and Dynamic Shear Rheometer (DSR). The properties of asphalt mixtures were evaluated based on the results of Marshall Stability, indirect tensile strength and direct tensile strength tests. The Optimum Binder Content (OBC) of WMA based on volumetric measurements

was determined using the Marshall method. The Compaction Energy Index (CEI) was evaluated using height versus gyration data obtained during Servopac gyratory compaction. The binder-aggregate constituent test specimens were evaluated using pull-off tension test and direct tensile test, while the binders surface free energy evaluation were determined by Goniometer contact angle and Dynamic Wilhelmy Plate (DWP) device.

The fracture faces of specimens after the direct and indirect tensile strength tests were analyzed using image analysis technique. The binder-aggregate substrate specimens were tested using direct tensile and pull-off tension tests. The failed surfaces obtained after the binder-aggregate substrate direct tensile and pull-off tension tests were processed for image analysis to quantify the adhesion failure. In addition to strength evaluation, the quantification of adhesion failure was considered as an assessment criteria for the identification of moisture susceptible specimens.

The SFE evaluation of asphalt binders was performed using Goniometer and DWP device. The short-term and long-term aging of asphalt binders was also considered along with unaged binders to evaluate the effects of aging on the SFE. The analytical measurements such as spreadability, work of adhesion, work of debonding and compatibility ratio which acts as an indicator of moisture susceptibility, were also determined.

1.5 Significance of Research

This research investigates the performance of WMA incorporating Cecabase used as Malaysian local road materials against moisture damage. Therefore, the output of this research will promote the use of WMA by the Malaysian asphalt industry. This research will also enable researchers to adopt the laboratory method

used to improve the process to identify moisture damage. The imaging technique is a very useful tool to quantify adhesion failure in conjunction with qualitative and quantitative assessments. This is because previous studies and standard test methods rely on visual inspection expressed in terms of ranking specimens based on equal to or more than 95% stripping. The proposed imaging method is able to quantify more specifically the exact percentage of failure due to adhesion, cohesion and broken aggregates.

Chemical additives such as Cecabase® contain combinations of emulsification agents, surfactants and adhesion promoting (anti-stripping) additives. These help to ease coating of the aggregate particles by the binder, mix workability and compaction (Rubio et al., 2012a,b). Therefore, comprehensive information obtained from the results of this research can be encouraging for the policy and decision makers to adopt WMA technology in the context of sustainable development.

The lower production and laying temperatures of WMA are the main advantages compared with HMA. On the other hand, the low temperature can make the WMA vulnerable to moisture damage. Therefore, it is necessary to investigate the effects of moisture damage on the performance evaluation of WMA incorporating Cecabase. The results of this research provide an in-depth knowledge and investigation strategies that can be adopted as evaluation criteria to develop and upgrade the Malaysian standards for future developments.

Asphalt mixture moisture damage is evaluated on loose and compacted mixtures through qualitative and quantitative methods. A novel approach that considers the qualitative evaluation based on image analysis technique enables

precise measurement of the extent of moisture damage compared to visual inspection. Conducting direct and indirect tensile tests on compacted mixture is essential to measure the mechanical strength of asphalt mixture that can produce results based on quantitative measurements. The application of image analysis technique on the same sample fractured surface when subjected to tensile force will produce the qualitative analysis or measurement of stripping potential. Therefore, instead of conducting tests on loose and compacted mix separately to obtain quantitative and qualitative measurements, this method enables researchers to use only one material for the evaluation of asphalt moisture damage. Moreover, this method can save time, material and minimize the possibility of errors which occur during sample preparation.

This study also evaluates the adhesion failure characteristics using the pull-off tension and binder-aggregate substrate direct tensile strength test. The application of image analysis technique on the results obtained using pull-off tension test can be more useful to quantify the adhesion failure. The binder-aggregate substrate direct tensile test followed by adhesion failure quantification using image analysis technique is a unique approach to characterize the asphalt material constituent properties. Therefore, these novel test approaches will create a new era for the asphalt pavement technologists and warm mix asphalt producers as well as civil engineers towards the material design and evaluation against moisture damage.

The study also covers the surface free energy characteristics, the effects of Cecabase and aging on the binder-aggregate bond. In the process of material selection criteria, these results can provide a better understanding about different combinations of asphalt binder and aggregate that are resistant to moisture damage.

1.6 Thesis Organization

This thesis is organized into eight chapters and presented as follows:

- (i) Chapter 1 introduces WMA and describes the problem statement, research objectives, scope of work and significance of the study.
- (ii) Chapter 2 presents the previous research work done on the moisture damage in asphalt mixtures. The laboratory test methods and newly developed test techniques to evaluate moisture damage in asphalt mixture and ingredients are also highlighted in this chapter.
- (iii) Chapter 3 defines the materials characterization in accordance to test standards, specimen preparation methods, test techniques and the application of image analysis for the identification of failure in asphalt mixtures.
- (iv) Chapter 4 presents the results of rheological properties of asphalt binders modified with different Cecabase contents subjected to various aging conditions and test temperatures. This chapter also evaluates the results of mixtures design and ease of compaction.
- (v) Chapter 5 describes the moisture sensitivity analysis based on the quantitative and qualitative measurements. An image analysis technique is used to quantify moisture susceptibility in asphalt mixtures based on adhesion failure as a qualitative measure.
- (vi) Chapter 6 evaluates the moisture susceptibility of mixture constituents based on pull-off tension and binder-aggregate substrates direct tension tests. This chapter also presents the application of image analysis technique on the fracture surfaces to estimate the failure mode in a more precise manner compared with visual inspection.

- (vii) Chapter 7 investigates the effects of Cecabase on the SFE evaluation of asphalt binder under different aging conditions. The results are discussed based on the analytical measurements of SFE such as coefficient of spreadability, work of adhesion and compatibility ratio as an indicator of moisture damage.
- (viii) Chapter 8 outlines the research conclusions and recommendations for future research.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter reviewed, summarized and discussed the mechanisms of moisture damage in asphalt mixtures and different approaches used to evaluate moisture damage effects based on material selection criteria. Moisture damage in asphalt mixtures has remained a topic of debate among investigators for many years. Moisture shortens the service life of asphalt mixtures, resulting in failures such as alligator cracking, ravelling, potholing and rutting (Liddle and Choi, 2007). There are three major areas of research in asphalt moisture damage: field investigations, laboratory experiments and analytical studies. Initially, most research was limited to field observations. Later, laboratory-based testing methods combined with field investigations were developed (Mehrara and Khodaii, 2013). The laboratory approach was based mostly on the development of techniques for simulating the field conditions accurately rather than conducting a fundamental assessment of asphalt moisture damage. In contrast, analytical methods based on surface free energy (SFE) evaluation are used to characterize the fundamental properties of aggregate and binder as related to moisture damage resistance (Howson et al., 2009). This fundamental evaluation can yield input criteria for material selection and design for preventing moisture damage in the field. The production of asphalt mixtures particularly at low temperature brings the attention of many highway agencies towards the assessment of moisture damage. It is believed that, due to lower production temperature, the moisture still exists within the aggregate microspores.

The moisture diffuses towards the binder aggregate interface and finally, the stripping of asphalt aggregate takes place.

This research highlights the importance and approaches used to address the moisture damage in warm mix asphalt. The primary goal when preparing asphalt mixtures is to remove the root cause of moisture damage. Here, one key consideration is the proper identification and assessment of distress. There are two common ways to reduce pavement distresses: preventive measures based on experience and the cautionary measures based on fundamental understanding. However, a third and more profound way of addressing moisture-related problems is currently under investigation. It is suggested that a combination of in-situ testing, material selection criteria and proper mix design can be used to effectively prevent moisture damage in asphalt mixtures.

Apart from this research, for future developments and implementation it is suggested that new in-situ testing techniques can assess the expected failures in asphalt mixtures more practically and correlate well with the material selection criteria. Therefore, these techniques can minimize the need for laboratory-based simulations of field conditions such as air void interconnectivity in field samples.

2.2 Background

First observed in the early 1900s, moisture damage was identified as one of the major causes of distress in asphalt pavements (Huang et al., 2010). Traffic-generated stresses reduce the internal strength of hot mix asphalt (HMA) pavement and can result in early rutting, fatigue cracking and ravelling of the HMA layer (Kok and Yilmaz, 2009). In asphalt mixtures, the adhesive and cohesive forces within the

aggregate and binder are primarily responsible for holding the latter together. Moisture can infiltrate into an asphalt pavement layer via the permeation of rainwater, a rising of the ground water table, the absorption and adsorption of water vapor or a combination thereof (Arambula, et al., 2007). Such an ingress of moisture shortens the design life performance (durability) of asphalt pavement, resulting in high maintenance costs. Every pavement requires maintenance at some point in its service life. Maintenance is the art of ensuring that a pavement is in an operational condition, while minimizing expenditures and inconvenience for road commuters. Although inappropriate maintenance can often be worse than doing nothing, preventive maintenance is a prudent addition to the other basic forms of maintenance (Hunter and Ksaibati, 2001).

The presence of water in asphalt pavement adversely affects the durability of the pavement and as a result can lead to very complicated modes of distress that are the stiffness and structural loss of pavement. Although the presence of water does not initiate distresses such as cracking, permanent deformation and ravelling, it exacerbates their severity and extent. Local road maintenance authorities in the United Kingdom and Wales alone spend £2.5 bn annually to prevent these distresses (ALARM, 2006). Attention has shifted from making repairs to taking preventive measures because the former imposes high costs on the road authorities involved and can cause inconvenience to road commuters. The current practice among mix designers is to purchase the binder and aggregate based on individual specifications. However, because there is a lack of knowledge about these mix ingredients, it is still not clear whether they can interact favorably (Kringos, 2007).

To develop techniques for the assessment of highway performance, Ensley et al. (1984) used a method to measure the bond energy of asphalt and aggregate. Gharaybeh, (1987) investigated the available testing methods for assessing the stripping potential of asphalt mixtures. There were no comparable developments for assessing moisture susceptibility until the Strategic Highway Research Program (SHRP) funded research for the development of new testing procedures aiming to prevent asphalt moisture ingress. Al-Swailmi and Terrel (1992) developed the environmental conditioning system (ECS), while Aschenbrener and Currier (1993) introduced the concept of the Hamburg wheel-tracking device (HWTD). Comprehensive work on asphalt chemistry and its significance related to moisture damage was conducted by the Western Research Institute (WRI). The asphalt source plays an important role in the separation of asphalt polar constituents from the aggregate. Presently, WRI is working on a rapid centrifugation method to evaluate the displacement of polar constituents by moisture in asphalt binder. The concept is based on the observation that insoluble calcium salts in asphalt components form in asphalt-aggregate mixtures that are less prone to moisture damage. In addition, surface energy parameters are possible tools for the assessment of asphalt-aggregate adhesion. However, although recent research has greatly aided the selection of asphalt-aggregate mixtures, it has not considered the effect of traffic-generated stresses combined with moisture damage (WRI, 2002). To this end, the National Cooperative Highway Research Program (NCHRP) Project 9-34 has focused on the environment-traffic factors for properly simulating moisture damage in asphalt mixtures (Solaimanian et al., 2003).

2.3 Moisture Damage Mechanism

According to Caro et al. (2008) moisture damage mechanism is taken on the following steps:

- I. Moisture transport: processes by which moisture in either a liquid or vapour state infiltrates the asphalt mixture as well as the asphalt binder or mastic and reaches the asphalt binder–aggregate interface, and
- II. Response of the system: changes in the internal structure leading to a loss of load carrying capacity of the material.

There are historically, six contributing mechanisms of moisture damage identified: detachment, spontaneous emulsification, displacement, pore pressure–induced damage, hydraulic scour, and the environmental effects on the aggregate–asphalt system. It is evident that moisture damage is normally not limited to only one mechanism but is the result of a combination of processes. It is important to develop a more fundamental understanding of the moisture damage process, by taking into consideration the micro mechanisms that affect the asphalt aggregate adhesive interface and the cohesive strength and mastic durability (Little et al., 2003).

The micro and macro mechanisms are considered to be the two mainstreams studies in the stripping evaluation of asphalt mixtures. There are some theories in asphalt and aggregate that explains the adhesion and cohesion failure on a molecular scale. Some other theories explain the adhesive and cohesive failure using macro-scale mechanical theories. However, both approaches can be seen in most of the recent researches (Mehrara and Khodaii, 2013). The micro mechanisms are further classified into mechanical theory, chemical reaction theory, molecular orientation theory, surface energy theory, weak boundary theory, and micro-theories include the

six traditional mechanisms. The macro mechanisms encompass the formation of excess pore pressure in saturated pavement, hydraulic scouring, physical erosion of asphalt due to high velocity hydraulic flows (Mehrrara and Khodaii, 2013).

2.3.1 Stripping

Early efforts to classify and describe asphalt stripping date from the 1960s and 1970s (Field and Phang, 1967; Lottman, 1978). However, in the 1980s, this subject attracted the interest of highway agencies and the pavement industry across the globe (Taylor and Khosla, 1983). In a report submitted to the National Center for Asphalt Technology (NCAT), Kiggundu and Roberts (1988) listed down several definitions of stripping in asphalt mixtures from the point of view of a number of researchers (Petersen, 1982; Tunnicliff and Root, 1984).

- Deterioration or loss of the adhesive bond between the asphalt and the aggregate from the action of water.
- The physical separation of the asphalt cement from the aggregate produced by the loss of adhesions primarily due to the action of water or water vapour.
- The displacement of asphalt cement films from aggregate surfaces by water caused by conditions under which the aggregate surface is more easily wetted by water than by asphalt.
- The breaking of the adhesive bond between the aggregate surface and the asphalt cement.
- The loss of the bond between the asphalt binder and the mineral aggregate due to separation of asphalt cement coating in the presence of water.
- The progressive functional deterioration of a pavement mixture by loss of the adhesive bond between the asphalt cement and the aggregate surface and or

loss of the cohesive resistance within the asphalt cement principally from the action of water.

From the above definitions, stripping is the separation of asphalt from aggregate or the rupture of asphalt texture in asphalt mixtures under the combined, simultaneous action of cyclic traffic load and water or water vapor. According to Kiggundu and Roberts (1988), a more complete definition of stripping includes the cohesive and adhesive failures that are considered to be the main causes of moisture damage. Moisture infiltration is normally considered a primary cause of stripping in asphalt mixtures; it therefore causes the removal of asphalt binder from the aggregate surface. The stripping phenomenon leads to a pre-mature rehabilitation and higher maintenance cost (Haghshenas et al., 2015). The progressive dislodgement of aggregate can occur because of the continuous and combined action of moisture and traffic load (Kringos, 2007). Studies that have evaluated several aspects of stripping are classified based on fundamental studies, qualitative studies and quantitative or engineering-based studies (Kiggundu and Roberts, 1988).

2.3.2 Mechanisms of the Stripping Process

There are a number of mechanisms that can account for stripping in asphalt mixtures. According to these mechanisms, there must be a stripping initiation point and its subsequent propagation (McGennis, 1984; Tarrer, 1991). Stripping usually begins at the bottom of the bituminous layer where it is thought that the moisture content is high and moves upward (Graf, 1986). The mechanisms of stripping in asphalt mixtures presented in Table 2.1 are formulated and compiled by Bagampadde et al., (2004).

Table 2.1: Mechanisms of Stripping in Asphalt Mixtures (Taylor and Khosla, 1983)

Process	Theory	Mechanism
Displacement	Thermodynamic and chemical reaction	Water with lower surface energy and higher dipole moment than bitumen displaces it from aggregate surfaces.
Detachment	Thermodynamic and chemical reaction	Water with lower surface energy and higher dipole moment than bitumen detaches it from the aggregate surface.
Spontaneous emulsification	Electrostatic	Emulsion formation, due to presence of agents like clay coatings, weakens the bonding at the interface.
Pore Pressure	Mechanical break	High pore water pressure in undrained conditions causes a break in bitumen film allowing water to enter the interface.
Chemical disbonding	Chemical reaction and electrostatic	Chemical and electrostatic interaction between water and some aggregates favour removal of bitumen from them.
Microbial activity	Bacterial metabolism	Microbial metabolic processes at the interface give by-products that break adhesion at the interface.
Osmosis	Diffusion	Concentration gradient across the bitumen film causes water to be transported to the interface.

2.3.3 Adhesion Failure as a Major Contributing Factor

The failure mechanism of the asphalt and aggregate adhesion bond has remained a topic of debate among researchers. It is thought to be related to one or both of the following phenomena.

- First, moisture may interact with the binder, causing a reduction in cohesive strength and subsequently a reduction in mixture stiffness.
- Second, water can gain access to the spaces between the asphalt film and aggregate, breaking the adhesive bond and finally stripping the asphalt binder from the aggregate.

In both failure mechanisms, the moisture may diffuse through the asphalt binder to the interface, or it may already exist in the aggregate micropores due to the low-temperature production of asphalt mixture in accordance with WMA (Zaniewski and Viswanathan, 2006). According to Hicks (Hicks, 1991), adhesion is defined as “the physical property or molecular force by which one body sticks to a body of another nature”. The asphalt-aggregate adhesion is influenced by many factors including the interfacial tension between the asphalt binder and the aggregate, the aggregate temperature, the chemical composition of the asphalt binder and the interfacial moisture content present at the time of mixing.

Adhesion is a fundamental property of the asphalt-aggregate interfaces. Research has established the importance of adhesion to asphalt moisture susceptibility and its relation to pavement durability and quality (Kringos, 2008; Al-Qadi, 2006). The molecular forces between adhesive and substrate play a large role in every adhesive and adherent system. The physical and chemical behaviors of wetting and interlocking are strongly affected by the molecular forces. Therefore, the adhesion strength of road materials strongly depends on the interaction, affinity and attraction between the asphalt and aggregate. Hence it is believed that the chemical nature of asphalt and aggregate governs adhesion (Merusi et al., 2010). There are basically four general theories of adhesion that attempt to explain the asphalt-aggregate adhesion. These include the mechanical interlocking theory, the chemical

reaction theory, the surface energy theory and the molecular orientation theory. However, these theories can only partially explain the nature of adhesion (Hicks, 1991; Johnson, 2002).

2.3.4 Asphalt-aggregate Interface

Moisture can accelerate the damage due to different types of distress in asphalt mixtures (Cho and Kim, 2010). The response of asphalt mixtures to different distresses is influenced by the mechanics of aggregate-binder interface bonding, which is affected by moisture damage conditions. Moisture at the asphalt-aggregate interface is a major contributing factor to the debonding of asphalt and aggregate (Moraes et al., 2011).

There are many possible mechanisms by which the water can access the asphalt-aggregate interface. These include migration through pinholes and diffusion through the asphalt matrix, local inhomogeneities, defects and pores in asphalt films. It is evident that during situations where the interface is exposed to a high water concentration for a short time or there is a thin water layer at the interface of thick asphalt films, water transport to the interface from the outside occurs through the hydrophilic or water-soluble regions of the asphalt film. The areas covered by the highly polar groups of asphalt molecules or water-soluble impurities (ions and salts) in the asphalt film are considered hydrophilic regions (Lu and John, 2005). Each water-soluble impurity is probably linked to a polar site in the asphalt. Therefore, it is possible that water-soluble impurities and polar groups of asphalt molecules are present in hydrophilic regions of an asphalt film (Nguyen et al., 1996; Nguyen et al., 2003). In summary, water-soluble materials, which can be transferred from the environment, transferred from the asphalt film or present at the interface (that is,

transferred from both asphalt and aggregate), form a water-sensitive layer at the asphalt-aggregate interface. This results in the formation of a water layer, many thick monolayers at the interface and finally in the stripping of asphalt or loss of adhesion of asphalt at the siliceous aggregate interface (Nguyen et al., 2005). The polar constituents at the asphalt-aggregate interface form a bond between the asphalt and aggregate surface. The bonding force between asphalt and aggregates decreases because of the loss of these polar constituents in asphalt; this weakening could ultimately accelerate the adhesion failure in asphalt pavements. Early adhesion failure at the asphalt-aggregate interface may also be caused by preferential binding of the aggregate to acetate anions, which are more polar than the asphalt molecules. Furthermore, acetate anions can weaken the bond between asphalt and aggregate, leading to different forms of distress such as ravelling and stripping, which normally occurs in moisture-damaged asphalt pavements (Pan et al., 2008).

2.4 Laboratory Testing Methods

Since the 1920s, efforts have been made to develop laboratory-based testing methods to assess the performance of mixtures with respect to stripping (Solaimanian et al., 2003).

2.4.1 Standard and Non-standard Laboratory Test Methods

The laboratory-based testing methods are categorized according to the viewpoints of different investigators and are presented in Table 2.2 (Mehrara and Khodaii, 2013).

1. Tests on loose mixtures and mixture components.
 - (a) Qualitative measures of stripping

- (b) Indirect quantitative measures
 - (c) Energy based methods
 - i. Mechanical tests, measure of adhesion and cohesion
 - ii. Energy based indices
 - iii. Non- mechanical test
 - (d) Advance techniques
2. Tests on compacted mixtures
- (a) Destructive mechanical test on compacted mixtures
 - (b) Non-destructive mechanical test on asphalt concrete
 - (c) Non-destructive non-mechanical tests.

Table 2.2: Details of Standard and Non-standard Laboratory Test Approaches

Category	Test Method	Test Description
Tests on Loose Mixtures and Mixture Components		
Qualitative measures of stripping	Static immersion	Percent of aggregates surface that have maintained their asphalt coatings after static immersion in water
	Dynamic immersion	Percent of aggregates surface that have maintained their asphalt coatings after being agitated in water
	Boiling water	Percent of stripped aggregates after immersion in boiling water
	Methylene Blue	The amount of harmful clays of the smectite (montmorillinite) group, organic matter and iron hydroxides present in fine aggregates
	Quick and Rolling Bottle test	Measuring the adhesion capability of asphalt to Ottawa sand
Indirect quantitative measures	Net adsorption	A quantitative index based on the difference of the adsorbed asphalt to aggregate surface in the presence and absence of moisture
	Chemical immersion	A quantitative index based on the concentration of a chemical material for the initiation of moisture damage
	Surface reaction	A quantitative index based on the pressure of produced gas due to reaction of a chemical with the stripped surface of aggregates
	Tack Test System (TTS)	Measuring the required force to cause cohesive failure in asphalt