FORMULATION DEVELOPMENT AND MICROSTRUCTURE ANALYSIS OF A POLYMER MODIFIED BITUMEN EMULSION ROAD SURFACING

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ABSTRACT

The purpose of this research was to develop a formulation for a polymer modified bitumen emulsion road surfacing product called microsurfacing to a mid-scale prototype stage. A supplementary part of the development was to investigate the polymer-bitumen interactions and how they affected the products end properties using confocal microscopy.

The formulation development consisted of three stages: technical design specifications, initial design, detailed design. The technical specification was developed to define the product performance in quantitative measures, and set the initial formulation parameters to work within. The initial design development screened three polymers, four methods of adding polymer to the emulsion and two grades of bitumen. Experimental design techniques were used to determine the best polymer-bitumen combination and emulsion process method. Further experimental investigations consisted of screening three emulsifiers and assessing the effect of aggregate cleanliness on the surfacing abrasion and curing rate.

The detailed design used experimental factorial design to examine the effects of polymer concentration, emulsifier level, and emulsifier pH on the emulsion stability, microsurfacing wear resistance and cure rate.

The emulsion residue was observed using confocal microscopy with fluorescence light and the microsurfacing mixture using both fluorescent and reflected light.

The research showed that a emulsion using 100 penetration grade Safaniya bitumen with SBR latex polymer post added could provide microsurfacing abrasion resistance of less than 100 g/m^2; an improvement of 85% on the minimum specification. The vertical permanent deformation was less than the 10% and could not be attained without polymer addition. The use of aggregate with a high cleanliness and an alkyl amidoamine emulsifier resulted in surfacing cohesion development of 20 kg-cm within 90 minutes, which compares closely to the international specification.

Unexpected results not reported before were that the emulsion residue from biphase modified emulsions had a softening point up to 10°C higher than polymer modified hot bitumen with the same polymer concentration. The biphase emulsified binder residue also has a very different microstructure to hot modified bitumen and this structure has been proposed to help account for the improved resistance to high temperature and applied stress.

Modifications to the formulation are to improve the emulsion settlement and should focus on the density difference between the bitumen and polymer latex.

This research has shown that a microsurfacing roading product can be successfully formulated with New Zealand bitumen and aggregate sources to meet key specified performance requirements. By systematically investigating the effects of materials on the performance properties of the product, a formulation ready for a mid-scale experiment has been proposed.

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TABLE OF CONTENTS

ABSTRACT ii
ACKNOWLEDGEMENTS iv
TABLE OF CONTENTS v
LIST OF TABLES xi
LIST OF FIGURES xiii
LIST OF APPENDICES xv
GLOSSARY xvi

CHAPTERS

1. INTRODUCTION 1
- 1.1 Background 1
- 1.2 Microsurfacing Product Design 2
- 1.3 Technical Specifications 2
- 1.4 Initial Laboratory Development 3
- 1.5 Formulation Detailed Design 3
- 1.6 Confocal Microscopy Research 4
- 1.7 Research Aims and Objectives 5
  - 1.7.1 Aim 5
  - 1.7.2 Research Objectives 5
  - 1.7.3 Constraints 5

2. REVIEW OF BITUMEN EMULSION SCIENCE, POLYMER MODIFICATION, AND MICROSURFACING TECHNOLOGY 6
- 2.1 Introduction 9
- **2.2 Bitumen Emulsification 9**
- 2.3 Advantages of Bitumen Emulsions 10
- 2.4 Emulsion Classification 11
- 2.5 Raw Materials 12
  - 2.5.1 Bitumen 12
  - 2.5.2 Emulsifiers 17
  - 2.5.3 Water 19

2.5.4 Acids 19

2.5.5 Additives 19

2.6 Emulsion Production 20

2.6.1 Stirring 21

2.6.2 Homogenizers 21

2.6.3 Colloid Mills 22

2.6.4 Processing Variables 22

2.7 Emulsion Formulation Characteristics 23

2.7.1 Emulsion Stability 24

2.7.2 Breaking Process 25

2.7.3 Emulsion Adhesion 26

2.7.4 Cohesion 26

2.7.5 Viscosity 26

2.8 Characteristics of General Emulsion Stability 27

2.8.1 Low Interfacial Tension 28

2.8.2 Zeta Potential 28

2.8.3 Electrical Double Layer Repulsions 28

2.8.4 Narrow Droplet Size Distribution 28

2.8.5 High Viscosity 28

2.9 Modification of Bitumen Emulsion Properties 28

2.9.1 Increasing the Viscosity of the Emulsion 29

2.9.2 Decreasing the Viscosity of the Emulsion 29

2.9.3 Changing the Emulsion Breaking Rate 30

2.9.4 Storage Stability 30

2.10 Polymer Modified Bitumen and Emulsions 32

2.10.1 Polymer Modified Bitumen 32

2.11 Types of Polymer Used 33

2.11.1 Plastomers 33

2.11.2 Elastomers 33

2.12 Interactions during Manufacture 34

2.12.1 Grade of Polymer 35

2.12.2 Physical Form of Polymer 35

2.12.3	Nature and Grade of Bitumen	35
2.12.4	Mixing Equipment	35
2.13	Elastomeric Polymers	36
2.13.1	Styrene Butadiene Styrene (SBS)	36
2.13.2	Styrene Butadiene Rubber (SBR)	38
2.13.3	Neoprene (Polychloroprene)	39
2.13.4	Natural Rubber (Polyisoprene)	39
2.14	Plastomeric Polymers	40
2.14.1	Ethylene Methyl Acrylate (EMA)	40
2.14.2	Ethylene Vinyl Acetate (EVA)	41
2.15	Bitumen/Polymer Compatibility	42
2.16	Microscopic Investigations of Bitumen/Polymer Blends	44
2.17	Polymer Modified Emulsions	45
2.18	Modified Emulsion Definitions	46
2.19	Manufacturing Processes	46
2.20	Polymer Modified Emulsion Properties	48
2.20.1	Storage Stability	48
2.20.2	Breaking Process	49
2.20.3	Choice of Bitumen	49
2.21	**Microsurfacing Technology**	**50**
2.22	Introduction	50
2.23	History and Relevance of Microsurfacing to New Zealand	50
2.24	Manufacture of Microsurfacing	51
2.25	Materials	52
2.25.1	Polymer Modified Emulsion	53
2.25.2	Aggregate	53
2.25.3	Mineral Filler	54
2.25.4	Water	54
2.25.5	Additives	55
2.26	Mix Design	55
2.27	Rate of Application	56
2.28	Weather Limitations	56

2.29 Chapter Conclusions 56

3. PRODUCT TECHNICAL SPECIFICATIONS AND REQUIREMENTS 58

3.1 Introduction 59

3.2 Suitability and Addition Levels of Polymers for Emulsification 59

3.3 Product Attributes 60

3.4 Preliminary Technical Specification for Microsurfacing 61

3.4.1 Scope 61

3.4.2 Description 61

3.4.3 Materials 61

3.4.4 Manufacturing Requirements 64

3.4.5 Mix Design 65

3.4.6 Test Requirements 66

4. MATERIALS AND METHODS 67

4.1 Materials 68

4.2 Processing Variables 70

4.2.1 Colloid Mill 70

4.2.2 Bitumen Phase Temperature 71

4.2.3 Soap Phase Temperature 71

4.2.4 Flow Rates 71

4.3 Preparation of Monophase Modified Emulsions 71

4.4 Preparation of Biphase Modified Emulsions 72

4.5 Test Methods 72

4.5.1 Emulsion pH 73

4.5.2 Viscosity 73

4.5.3 Binder Residue 73

4.5.4 Emulsion Settlement 74

4.5.5 Sieve Residue 74

4.5.6 Softening Point 74

4.5.7 Laser Scanning Confocal Microscopy (CLSM) 74

4.5.8 Wet Track Abrasion 75

4.5.9 Loaded Wheel Test 75

4.5.10 Microsurfacing Cohesion 75
4.5.11 Mix Time 76
4.6 Process Capability and Test Repeatability 76
4.7 Development Methodology 78
4.7.1 Initial Design 78
4.7.2 Emulsifier Investigation 81
4.7.3 Aggregate Assessment 82
4.7.4 Formulation Detailed Design 83

5. LABORATORY DEVELOPMENT RESULTS 86
5.1 Introduction 87
5.2 Initial Design Investigation 87
5.2.1 SBR Latex Modified Biphase Emulsion Results 87
5.2.2 Monophase Polymer Modified Emulsion Results 95
5.2.3 Conclusions 102
5.3 Emulsifier Investigation Results 104
5.3.1 Emulsion Viscosity 104
5.3.2 Emulsion Settlement 105
5.3.3 Emulsion Sieve Residue 105
5.3.4 Binder Properties 105
5.3.5 Microsurfacing Abrasion Resistance 106
5.3.6 Microsurfacing Cohesion 106
5.3.7 Mix Time 107
5.4 Aggregate Investigation Results 107
5.4.1 Microsurfacing Abrasion Resistance 108
5.4.2 Microsurfacing Cohesion 108
5.4.3 Conclusions 109
5.5 Detailed Design Investigation Results 110
5.5.1 Final Emulsion pH and Binder Residue 110
5.5.2 Emulsion Viscosity 113
5.5.3 Emulsion Settlement 114
5.5.4 Emulsion Sieve Residue 115
5.5.5 Binder Softening Point 115

5.5.6 Microsurfacing Abrasion Resistance 117

5.5.7 Microsurfacing Permanent Deformation 119

5.5.8 Microsurfacing Cohesion 119

5.5.9 Microsurfacing Mix Time 122

5.6 Determining an Optimum Formulation Range 128

6. CONFOCAL MICROSCOPY INVESTIGATIONS **131**

6.1 Introduction 132

6.2 Unmodified Bitumen 132

6.3 Hot Polymer Modified Bitumen and Resulting Emulsion Binders 133

6.3.1 SBS Modified Binder 133

6.3.2 SBR Modified Binder 134

6.3.3 EMA Modified Binder 136

6.4 Observations of Biphase Emulsions Containing SBR Latex 137

6.4.1 Resistance of Microstructure to Higher Temperature 139

6.4.2 Binder Resistance to Applied Stress 140

6.5 Observations of Microsurfacing 141

7. DISCUSSIONS AND CONCLUSIONS **144**

7.1 Introduction 145

7.2 Initial Product Specification 145

7.3 Initial Design 146

7.3.1 SBR Latex Biphase Emulsion Investigation 146

7.3.2 Monophase Polymer Modified Emulsion Investigation 148

7.3.3 Emulsifier Investigation 150

7.3.4 Aggregate Investigation 152

7.4 Detailed Design Investigation 153

7.5 Confocal Microscopy 156

7.6 Recommendations and Further Work 157

7.7 Conclusions 159

BIBLIOGRAPHY **160**

APPENDICES **169**

LIST OF TABLES

Tables

Table 2-1	Comparison of Viscosity for Polymer Modified Bitumen	47
Table 2-2	Aggregate Gradings for Microsurfacing	54
Table 2-3	Microsurfacing Mix Design Guidelines	56
Table 3-1	Polymer Addition Methods and Concentrations Suitable	60
Table 3-2	Product Attributes and Material Solutions for Microsurfacing	60
Table 3-3	Possible Polymers for Microsurfacing	62
Table 3-4	Possible Quick Setting Emulsifiers	62
Table 3-5	Grading of Type II Aggregate	63
Table 3-6	Grading of Type III Aggregate	63
Table 3-7	Potential Production Characteristics	65
Table 3-8	Approximate Mix Design Components	65
Table 3-9	Acceptance Criteria for Microsurfacing Emulsion and Binder	66
Table 3-10	Acceptance Criteria for Microsurfacing Slurry	66
Table 4-1	Microsurfacing Emulsifiers	69
Table 4-2	Grading of Aggregate used for Investigation	70
Table 4-3	Variation in Laboratory Production Process and Testing Methods	77
Table 4-4	Initial Emulsion Formulation	79
Table 4-5	Initial Microsurfacing Mixture Design	79
Table 4-6	Experimental Design Matrix for SBR Latex Biphase Emulsions	80
Table 4-7	Initial Design Runs for Producing Monophase Polymer Modified Emulsions	80
Table 4-8	Formulations for Monophase Polymer Modified Emulsions using 130/150 Bitumen	81
Table 4-9	Formulations for Emulsifier Experiment	82
Table 4-10	Experimental Plan for Aggregate Assessment	82
Table 4-11	Factors and Levels for Experimental Detailed Design	83
Table 4-12	Full Factorial Experimental Design Matrix for Three Factors at Two Levels	84
Table 4-13	Experimental Design Treatment Combinations for Three Factors and Two Levels	84

Table 4-14	Emulsion Formulation and Processing Conditions for Detailed Design	85
Table 4-15	Microsurfacing Mixture Design for Detailed Design Formulation	85
Table 5-1	Summary of Test Results from SBR Latex Biphase Emulsion Investigation	89
Table 5-2	Significance of Variables from SBR Latex Biphase Emulsion Investigation	90
Table 5-3	Summary of Test Results from Monophase Emulsion Investigation	98
Table 5-4	Slurry Mix Design Results for Monophase Emulsion Investigation	102
Table 5-5	Summary of Test Results from Emulsifier Investigation	104
Table 5-6	Test Results from Aggregate Investigation	107
Table 5-7	Summary of Test Results for Emulsion Detailed Design	111
Table 5-8	Significance and Effects of Variables Controlling the Emulsion and Microsurfacing Properties	112
Table 5-9	Summary of Significant Effects Controlling the Microsurfacing Properties	128
Table 5-10	Refined Emulsion Formulation for Mid-Scale Trial	130
Table 5-11	Microsurfacing Mixture Design for Mid-Scale Trial	130

LIST OF FIGURES

Figure

Figure 1-1	Product Design Stages to Develop the Microsurfacing Formulation	2
Figure 2-1	Emulsifier Behaviour on a Bitumen Particle	10
Figure 2-2	Main Chemical Constituents of Bitumen	13
Figure 2-3	Cationic Emulsifier	18
Figure 2-4	Schematic Diagram of an Emulsion Production Process	20
Figure 2-5	Manufacturing Process of Monophase Modified Bitumen Emulsion	46
Figure 2-6	Manufacturing Process of a Biphase Modified Bitumen Emulsion	48
Figure 2-7	The Microsurfacing Process	52
Figure 5-1	Five Day Emulsion Settlement for SBR Biphase Emulsions	92
Figure 5-2	Slurry Permanent Deformation for SBR Biphase Emulsion Investigation	94
Figure 5-3	Main Effects Plot for Emulsion Viscosity	113
Figure 5-4	Main Effects Plot for Emulsion Settlement	114
Figure 5-5	Main Effects Plot for Emulsion Sieve Residue	116
Figure 5-6	Softening Point Comparison between SBR Polymer Modified Emulsion Binder and Hot SBR Polymer Modified Bitumen	116
Figure 5-7	Main Effects Plot for Abrasion Resistance	118
Figure 5-8	Changes in Microsurfacing Permanent Deformation by Increased Polymer Addition	119
Figure 5-9	Main Effects Plot for Microsurfacing Cohesion after 60 minutes	120
Figure 5-10	Regression Plot for Microsurfacing Cohesion after 60 minutes	121
Figure 5-11	Regression Plot for Microsurfacing Cohesion after 90 minutes	122
Figure 5-12	Main Effects Plot for Microsurfacing Mix Time	123
Figure 5-13	Interaction Plot for Mix Time of Polymer and Emulsifier	124
Figure 5-14	Interaction Plot for Mix Time of Emulsifier and Soap pH	124
Figure 5-15	Contour Plot for Mix Time of Polymer and Emulsifier Level	126
Figure 5-16	Contour Plot for Mix Time of Emulsifier and pH Level	127

Figure 6-1	CLSM Fluorescence Image of Unmodified Bitumen (1000x Magnification)	132
Figure 6-2	CLSM Fluorescence Image of Unmodified Bitumen Emulsion Residue (1000x Magnification)	133
Figure 6-3	CLSM Fluorescence Image of Bitumen Modified with 3% SBS Polymer (400x Magnification)	134
Figure 6-4	CLSM Fluorescence Images of 3% SBR Latex Pre-blended into Hot Bitumen a). The Pre-blended SBR-Bitumen Residue after Emulsifying b). (1000x Magnification)	135
Figure 6-5	CLSM Fluorescence Images of 3% EMA Modified Bitumen a). EMA-Bitumen Binder Residue after Emulsifying b). (1000x Magnification)	136
Figure 6-6	CLSM Fluorescence Images of 3% SBR Latex Modified Biphase Emulsion Binders (1000x Magnification)	138
Figure 6-7	CLSM Fluorescence Image of Biphase Emulsion Residue after Heating at 109°C (1000x Magnification)	139
Figure 6-8	CLSM Fluorescence Image of Biphase Emulsion Binder under Shear Strain (1000x Magnification)	140
Figure 6-9	CLSM Depth Scan Image of Microsurfacing under Fluorescent Light (100x Magnification)	141
Figure 6-10	CLSM Depth Scan Image of Microsurfacing under Reflected Light (100x Magnification)	142
Figure 6-11	CLSM Composite Depth Scan Image of Microsurfacing with Combined Fluorescence/Reflected Light (100x Magnification)	142

LIST OF APPENDICES

Appendix

2-1	Comparison of Chemical Fractions within Bitumen Sources	169
5-1	Emulsion and Microsurfacing Results from SBR Latex Addition Method Investigation	170
5-2	Microsurfacing Mix Design Results for SBR Latex Biphase Emulsion Investigation	171
5-3	Emulsion and Microsurfacing Properties from Monophase Modified Emulsion Investigation	172
5-4	Emulsion and Microsurfacing Properties from Detailed Design Investigation	173
5-5	Contour Plot of Abrasion Loss from Detailed Design for Emulsifier and Soap pH Level	174
5-6	Contour Plot of Abrasion Loss from Detailed Design for Polymer and Soap pH Level	175
5-7	Experimental Error Results from Detailed Design Experiment	176

GLOSSARY

Aggregate	A hard inert mineral material, such as gravel, crushed rock, or sand.
Binder	Material which secures aggregate to road surface. Can comprise of bitumen, polymers, solvent or other solid material.
Biphase Emulsion	Polymer modified bitumen emulsion characterised by a dispersed phase made up of two types of droplets: bitumen and polymer.
Break	The destabilisation of an emulsion resulting in the separation of emulsified phases (demulsification).
Copolymer	A polymeric structure that is composed of at least two different monomers in alternating sections or a coupling group of low molecular weight.
Curing	The development of mechanical properties of the bitumen binder. This occurs after the emulsion has broken and the emulsion particles coalesce and bond to the aggregate.
Cut-back	Bitumen liquefied by blending with petroleum solvents.
Emulsifier	The chemical added to the water and bitumen that keeps the bitumen in stable suspension in the water.
Elastomer	Polymers that can easily undergo large elongation at relatively low stress levels and rapidly return to approximately its original size.
Latex	An aqueous, stable, colloidal emulsion of a polymer substance.
Microsurfacing	A mixture of polymer modified bitumen emulsion, crushed graded aggregate, mineral filler, additives, and water. Microsurfacing provides thin resurfacing of 10 to 20 mm to the pavement and returns traffic use in 1 to 1.5 hours under average conditions.
Monophase Emulsion	Polymer modified bitumen emulsion characterised by a dispersed phase composed of only polymer modified bitumen droplets.
Residue	The bitumen binder that remains after the emulsion has broken and cured.
Wetting	The reduction of interfacial tension.

1. INTRODUCTION

1.1 Background

The use of polymer modified bitumen emulsions for road sealing maintenance has the potential to be an important product area for New Zealand contractors. Unmodified bitumen softens under increased temperatures and this results in the pavement deforming (Whiteoak, 1990; Transit, 1993; Asphalt Institute, 1994). Common problems encountered are loss of stone chips and formation of wheel tracking ruts that cause an uneven surface. The loss of stone chips reduces tire traction. Wheel ruts in roads can cause vehicles to aquaplane due to water build-up and reduce braking effectiveness. These problems can be reduced by the addition of polymer modifiers to the bitumen to increase its strength and elasticity (Whiteoak, 1990; Transit, 1993; Bahia et al., 1998; Swanston & Remtulla, 1998).

But the only product alternatives in New Zealand to solve these problems are polymer modified hot-mix asphalt, or polymer modified hot *cut-back** bitumen as a sprayed layer covered with graded *aggregate* (Transit, 1993). Asphalt is expensive and must be laid in thick layers. Cutback bitumen contains petroleum solvent to reduce the temperature needed to lower the viscosity to a sprayable level. But, the spraying temperature is still around 160°C. Another drawback of solvent is that it also reduces the softening point of the bitumen, making it more susceptible to heat. The combination of high temperature and solvent present a safety risk for workers, high energy costs and environmental concerns over solvent evaporation (Asphalt Institute, 1994; Reed, 1996). Both of these options also require the whole section of road to be resurfaced even though in many cases it is only the wheel ruts that may be the problem.

In particular the *microsurfacing* product, which uses a polymer modified bitumen emulsion mixed with aggregate, has important benefits. The advantage of bitumen emulsions is that they are applied at ambient temperature, and generally require no solvent. In the USA and several countries in Europe the microsurfacing product is common and rapidly gaining acceptance (Asphalt Institute, 1994; Holleran, 1997).

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* Italicised words appear in the glossary.

Microsurfacing imparts protection to the underlying pavement and provides renewed surface friction. Wheel ruts of up to 40 millimetres can be easily filled using this product. Microsurfacing is quick setting, which allows traffic rapidly on the pavement. It can also be applied in the early evening or even at night-time.

1.2 Microsurfacing Product Design

The basic formulation aspects of a microsurfacing consists of:

1. Polymer modified bitumen emulsion
2. Graded aggregate
3. Setting additives
4. Extra water to wet the aggregate

The most challenging part of designing a microsurfacing is the emulsion formulation (Asphalt Institute, 1994; Holleran, 1997). The experimental work undertaken in this research focuses mainly on this part of the product. But, it is important to recognise the whole microsurfacing system and the experimental work also includes the emulsion-aggregate interactions in detail. The formulation development followed a common product design approach. The product design approach used in this experimental research consisted of the phases shown in Figure 1-1.

Figure 1-1. Product Design Stages to Develop the Microsurfacing Formulation

- Technical design specifications
- Initial design – material screening
- Detailed design

← Phases covered in this research

- Scale-up and validation
- Optimal design
- Production and launch

1.3 Technical Specifications

Developing a set of technical specifications helps to define the product performance in quantitative measures, set the initial formulation parameters to work within and the process method to use. A set of preliminary specifications for the product was prepared to help guide the initial formulation development. This included suitable materials, process method and processing parameters. Performance criteria to compare the experimental products against were selected from technical literature. The technical specification developed is discussed in chapter 3.

1.4 Initial Laboratory Development

The scope of the product materials and their effect on the performance properties requires a screening process to adequately assess them. The polymer type and its method of addition to the emulsion can add different performance properties to the bitumen *binder*. The polymer can be added to an emulsion in four possible ways and it needed to be determined if there were significant performance differences. Bitumen can be supplied in different grades and this directly affects the durability of the microsurfacing and also the polymer processing method. The *emulsifier* type can affect the cure rate of the microsurfacing, which determines the time frame for allowing traffic on the surfacing. Aggregate type and quality are also suggested to be very important to the durability and *curing* aspects of the surfacing (Asphalt Institute, 1994). Hence, the experiments had to investigate these aspects to understand material interactions, in order to select the viable polymer(s), bitumen, emulsifier, aggregate, and emulsion process method.

1.5 Formulation Detailed Design

The detailed design experiment took the best polymer, emulsifier, bitumen type, aggregate type and emulsion processing method determined from the initial formulation material screening. The emulsion was further investigated in detail by examining the effects of the polymer, emulsifier and emulsifier solution pH. These aspects were selected as they could affect in some way the emulsion stability, the bitumen resistance to deformation and also the microsurfacing cure rate. The aim was to refine the material addition levels to produce an optimal set of microsurfacing performance characteristics.

To investigate the overall research questions a selection of experimental design trials were used to systematically examine the performance effects of materials and refine step by step the formulation to be ready for a mid-scale trial.

1.6. Confocal Microscopy Research

Polymer modified bitumen should ideally have a microstructure that consists of a fine dispersion of polymer throughout the bitumen (Piazza et al., 1980; Bouldin et al., 1990; Morgan & Mulder, 1995; PIARC, 1999). But the addition of polymer to bitumen can cause compatibility problems in the polymer-bitumen blend. The problem can manifest itself as phase separation whereby the polymer rises to the top of the bitumen. Or the polymer can coagulate into lumps at a microscopic level giving an uneven distribution. This incompatibility is strongly dependent on the bitumen source (Morgan & Mulder, 1995; Loeber et al, 1996). Incompatible binders can cause storage stability problems and also can result in early aggregate loss from a road surfacing.

Microscopy techniques have been used in several studies to examine the compatibility of polymers with bitumen (Piazza et al., 1980; Bouldin et al., 1990; Loeber et al., 1996; Rozeveld et al., 1997; Lu et al., 1999). But there has been no reported literature regarding the compatibility of polymers with New Zealand's source of bitumen at a microstructural level. Another gap in the research literature relates to the microstructure of polymer modified bitumen emulsion binder. The modified binder after evaporation of the water phase is supposed to result in the same properties of a hot sprayed modified bitumen (Asphalt Institute, 1994). The research investigates this effect, but also goes further and investigates the way that the polymer improves the properties of bitumen, and how they resist stress in the binder and microsurfacing. A technique called confocal microscopy was used to assess the binder and microsurfacing microstructure.

Chapter 2 will cover the technical aspects of bitumen emulsions, polymer modification, and microsurfacing technology to give an overview to understand the critical parameters involved.

The research has been partially funded by the Higgins Group of Companies and Technology New Zealand, and the formulations should be treated as confidential.

1.7 Research Aims and Objectives

1.7.1 Aim

The research aim is to investigate and develop a polymer-modified emulsion based road surfacing (microsurfacing) formulation to a mid-scale prototype stage. A supplementary part of the development was to investigate the polymer-bitumen interactions and how they affect the products end properties by using confocal microscopy.

1.7.2 Research Objectives

- Identify and measure the effects of polymers to meet the performance requirements of the microsurfacing.
- Determine the required effect of emulsifiers and aggregate quality to obtain a rapidly curing microsurfacing.
- Use a combination of qualitative (microscopy) and quantitative (physical testing) techniques to understand the performance enhancing properties of polymer-modified bitumen.
- Compare and relate the test results of the modified bitumen binder and microsurfacing to results from overseas studies.
- Measure and determine the effect of varying the method of adding the polymer to the emulsion.

1.7.3 Research Constraints

Product Constraints

- Bitumen sourced from Marsden Point refinery must be used.
- Meet relevant industry specifications for performance.

Process Constraints

- Prototype emulsions produced using the Higgins laboratory colloid mill.