Copyright is owned by the Author of the thesis. Permission is given for a copy to be downloaded by an individual for the purpose of research and private study only. The thesis may not be reproduced elsewhere without the permission of the Author.
FORMULATION DEVELOPMENT AND
MICROSTRUCTURE ANALYSIS OF A POLYMER
MODIFIED BITUMEN EMULSION ROAD
SURFACING

A Thesis presented
in partial fulfilment of
the requirements for the Degree of
Master of Technology in Product Development
at Massey University, Palmerston North, New Zealand.

ALLAN R FORBES
2000
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/m²; 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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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).

* 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
- 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.