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SYSTEMATIC DEVELOPMENT OF A HIGH BITUMEN CONTENT EMULSION

A Thesis
presented in fulfilment of
the requirements for the degree of
Master of Technology in Product Development
at Massey University.

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ABSTRACT

A product development approach appropriate to a medium sized civil engineering company was developed in this study. The approach was practically demonstrated to the company through the development of a high bitumen content emulsion.

Product development processes were reviewed and related to the specific company environment to develop an appropriate six stage process consisting of: Project Start, Pre-development, Laboratory Development, Mid-scale Development, Full-scale Development and Commercialisation. The high bitumen content emulsion product was taken from the Project Start stage through to the Full-scale Development stage. During each of the stages, suitable product development systems were generated to practically implement the process.

During the Project Start stage, idea capture systems were created and scoring models were developed to initiate development projects. Avenues to allow greater information collection were demonstrated to the company and a cataloguing tool was developed to assist in the organisation of a technical library useful for product development. During the Pre-development stage, marketing and technical specifications were produced to guide later development. The Laboratory Development stage had three phases: initial design, detailed design and optimisation. Experimental design was demonstrated to the company as an appropriate technique to conduct detailed design for formulated and processed products. A full factorial experimental design with four factors at two levels identified an optimum area which was further explored to produce an optimised high bitumen content emulsion at the Laboratory Development stage.

A lack of Mid-scale equipment meant scale-up was conducted on a full-scale level in this study. Two full-scale trials were run; both emulsions were stable when exiting the production mill however the emulsion was unable to tolerate conditions it encountered upon entering the spray tanker. Possible causes as to the observed instability were rapid cooling of emulsion, dilution with water, excessive shearing during circulation, incompatibility with bitumen or the formulation. All of these factors were investigated

with no conclusive results. It is recommended to further investigate the composition of bitumen used by Higgins as to its suitability for high bitumen content emulsions. A means to test emulsion application variables on a laboratory or mid-scale level are also recommended in order to investigate and solve the problems.

The tailored product development approach and supporting systems developed in this study can be used by the company in future to carry out systematic product development.

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GLOSSARY

<i>Aggregate</i>	A hard inert material such as gravel, crushed rock slag or sand.
<i>Binder</i>	Material which secures aggregate to road surface. Comprising of bitumen, solvent, polymers or other solid material.
<i>Break</i>	The destabilisation of an emulsion resulting in the separation of emulsified phases (demulsification).
<i>Chip seal</i>	A road surface consisting of a stone chip layer held by a binding material.
<i>Cut-back</i>	Bitumen liquefied by blending with petroleum solvents.
<i>Cutter</i>	Petroleum based solvent such as kerosene, turpentine or naphtha.
<i>Ionise</i>	Chemical interaction leading to the formation of ions.
<i>Hydrophilic</i>	Water-seeking.
<i>Hydrophobic</i>	Water-fearing.
<i>Slurry sealing</i>	A pavement sealing technique in which a mixture of bitumen emulsion, fine aggregate, mineral filler and water are overlaid on an existing pavement to maintain a uniform and skid resistant surface.
<i>Wetting</i>	The reduction of interfacial tension.

1. INTRODUCTION

Adopting a systematic approach to product development provides a company with a standard and repeatable method which enables it to more consistently create products that fulfil customer needs and generate wealth. When adopting a systematic product development approach, the approach needs to be tailored to the specific needs and environment of the company.

The goal of this study was to tailor a product development approach to a medium sized company, Higgins Contractors Limited and practically demonstrate this approach through the development of a high bitumen content emulsion product. There are therefore two aspects of development addressed throughout this study:

- The development of tailored product development systems, which make up the entire product development approach.
- The development of a high bitumen content emulsion product.

These two areas of development were intended to provide the company with a new product and an appropriately tailored product development approach which could be applied to other products in the future.

1.1 COMPANY BACKGROUND

Higgins Contractors Limited was founded in Palmerston North by Dan Higgins in 1963 as a drain laying business. Other family members joined the firm and over recent years control has been passed onto his three sons, who are directors and owners of the company. Growth of the company from drain laying has seen diversification into *aggregates*^{*}, concrete, bitumen and road construction. As a civil engineering contractor, Higgins tenders for road construction jobs offered by a variety of customers such as local bodies, Transit New Zealand and the public. When developing road construction products Higgins must not only consider these customers but also the requirements of its contractors which deliver the product and the final users of the roads.

^{*} Italicised words appear in the glossary.

Three other centres of operation have been established; these are based in Wellington, Tauranga and Napier. Rapid expansion into these areas has brought the company to a crossroads over the last five years. With the company becoming quite sizeable, it is now making the transition from a family business to a corporate organisation. Establishment of a technical centre in 1996 to undertake quality testing and product development was a part of this transition. Product development at Higgins is therefore very much in its formative years, with this study contributing to the creation of product development systems. A small development staff consisting of a single product developer and a technical manager fulfil the technical development needs of the company at this point in time. A small staff means rapid implementation and adaptation of product development systems is possible as there is little bureaucracy.

Company infrastructure development is under way to establish marketing, human resources and information systems departments. The current absence of these formal departments is also a factor to be considered in the tailoring of product development systems within the company.

The road construction market is very mature and largely constrained by the allocation of government funding. Innovative products and products which provide better, lower costing solutions to customers allows for more competitive tendering within this market. Developing a systematic product development approach provides a formal means by which these more innovative and lower costing products can be created to keep the company competitive.

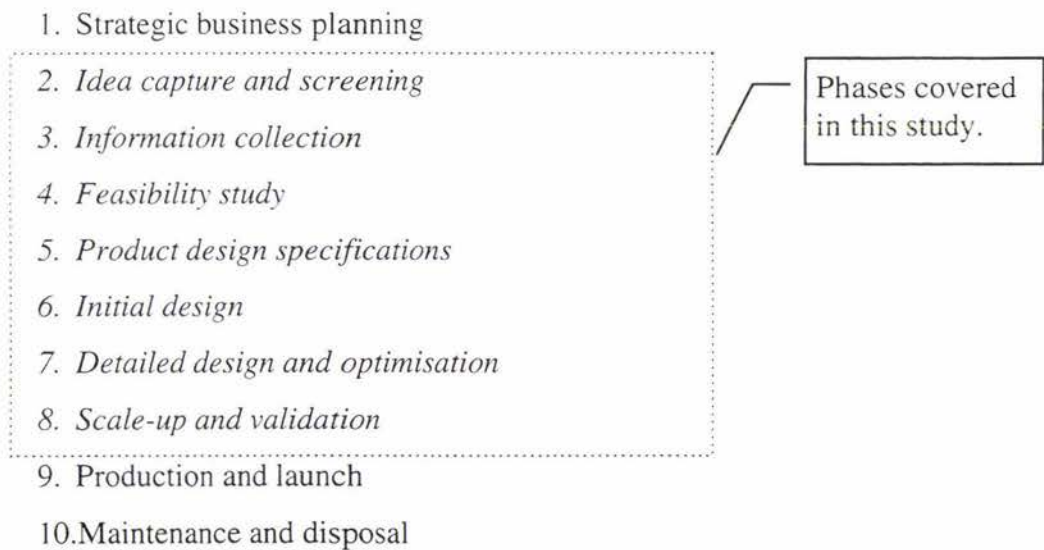
Environmental factors which need to be considered in creating appropriate product development systems include: company control structure, rapid growth, infrastructure development, a recently established technical department and market forces.

1.2 PRODUCT DEVELOPMENT PROCEDURE

Many suggested approaches to product development exist with five appropriate processes reviewed in Chapter 2: Haas, (1989); BS7000, (1989); McGrath et al., (1992); Cooper, (1993); Earle, (1997). Each of these processes have a different focus mainly

due to the environment in which the systems are used and the nature of the products being produced. When tailoring a product development approach the underlying principles from the many different product development approaches need to be extracted and then applied to the specific environment at hand. Although many product development processes exist most included similar phases. Figure 1-1 illustrates common phases in a product development approach and the phases this work focused on. Research covered phases from idea capture and screening through to scale-up and product validation with particular focus on four key areas; idea capture and screening (Chapter 5), product design specification (Chapter 6), detailed design and optimisation (Chapter 7), scale-up and validation (Chapter 8). The specific product development systems created in these four areas are discussed as well as the work carried out to develop the high bitumen content emulsion.

Figure 1-1 Common Phases In Product Development



A brief overview of the basic phases covered during the research work provides a understanding of the development path followed.

1.2.1 Idea Capture and Screening

The product development process is initiated by ideas. For the best set of ideas to be developed into profitable products the ideas first need to be systematically collected, screened and prioritised. Idea collection systems are usually simple to use, able to capture the essence of the potential opportunity and be widely adopted across the company.

Such idea collection methods include suggestion boxes, idea collection sheets, brain storming sessions and idea contests (Cooper, 1993).

Ideas screening needs to be unbiased so as to correctly identify the best allocation of resources to maximise the benefits to the company. Screening needs to be based on set criteria to assess both accurately and consistently all key areas of project viability. Scoring models, decision trees and profile modelling are a few suggested screening methods (Balachandra, 1989; Hass, 1989). Regular reviews of new and old ideas are carried out to ensure that the best set of projects are being developed at any one time.

Key product development system areas in the idea collection and screening phase include a good documentation system, regular reviews and an appropriate screening method.

1.2.2 Information Collection

A product developer uses research skills to collect information from numerous sources to carry out more informed product development; generally resulting in lower costs, shorter development time and better performing products. Increasing the ability to research and gather information enables the product developer to carry out product development more effectively. The information gathered during here will be the basis on which many later decisions will be made; the feasibility and design specification stage relying heavily on the information gathered during this phase to determine project viability and set development direction. The technical knowledge built up through research will also guide design decisions. Product development systems required in this phase include, a means of cataloguing past research, an efficient means of accessing new information and the ability to remain up-to-date. These systems provide the means to carry out research based product development.

1.2.3 Feasibility Study

A feasibility study quantitatively establishes project viability. Three key areas of feasibility are investigated; these are market, technical and financial feasibility. Demonstrated viability in these areas will justify further allocation of resources. Market feasibility is the key to on-going product success. A study conducted by Cooper (1993)

marketing specification and a technical specification (Cross et al., 1992). Both specifications begin by researching and prioritising customer requirements to ascertain what is wanted from the product. The technical specification translates the customer requirements into quantifiable metrics. The metrics describe in a technical sense the desired product characteristics which can be measured and tested. Target levels for the metrics are set, based on competitive product analysis. To obtain a product advantage a number of these metric levels should exceed competitors' levels. The sum of these metrics accurately describes in a technical sense the product the customer requires.

The marketing specification contains more of the qualitative factors related to the product including:

- Target market and users.
- Benefits of product use.
- Positioning of the product within the company's range and within the market.
- Initial marketing and promotional strategies.

The two specifications fully describe why the product is to be developed, who the product is aimed at and what is to be achieved during development. The process by which these design specifications were developed specifically for Higgins is discussed in Chapter 6.

1.2.5 Initial Design

Having established clear design direction through the design specification initial design can begin. Initial design is an exploratory phase of design where an understanding of important product variables is gained and many concepts are investigated. The scope of work is purposefully kept broad to avoid potential solutions being eliminated too early (O'Hare, 1993). Initial prototypes are screened against criteria set out in the design specification to identify a concept which possesses the greatest potential for further development.

1.2.6 Detailed Design and Optimisation

Detailed design takes the most promising concept identified during initial design and systematically explores the many design possibilities within this concept; out of the

numerous design possibilities a preferred design is identified. The method in which detailed design is carried out will vary greatly depending on the nature of the product; formulated products such as the high bitumen content emulsion require a different approach to manufactured and consumer goods. Formulated products require investigation into both the mixture components and the processing conditions, as different combinations of these variables will produce different products. Formulated products may use mixture design, experimental design or central composite design experiments to carry out detailed design (Montgomery, 1985; Barnes; 1997). A detailed design method suitable to Higgins products is outlined in Chapter 4.

Through the systematic design process a complete understanding of the product is gained and a preferred design is identified. The preferred design is further optimised to arrive at a final design. The final prototype design is validated through scale-up.

1.2.7 Scale-up and Validation

The aim of scale-up and validation is to take the final prototype design and prove its performance in a situation more representative of final production and application. Scale-up errors can occur when moving from small prototype designs to full production, this is due to differences in processing and product behaviour. By making incremental scale-up steps the risk and cost of potential failure is reduced while more accurate predictions can be made about full scale production and costs.

Gaining the most benefit from conducting scale-up and field trials involves thorough planning of trials, clear communication with the many involved parties and accurate data collection. Good planning results in greater control of the many tasks involved in scale-up, while communicating with the many involved parties assists in producing the product designed. Data collected during validation is used to confirm specifications, correlate laboratory scale work with full manufacture and to indicate product launch or re-design. Appropriate product development systems facilitate the execution of these key activities.

1.3 APPLICATION TO BITUMEN EMULSIONS

The product development procedure outlined was demonstrated in a practical way within the company using the development of a high bitumen content emulsion.

1.3.1 Background

Bitumen emulsions containing water are used in the road construction industry to improve the materials flow properties, once emulsified the bitumen behaves more like water than viscous bitumen. The traditional means of altering bitumen's flow properties is to add solvents such as kerosene at around 3-8%, this is called "*cutback*" bitumen. The bitumen/kerosene mixture is then applied to the road with a sprayer at temperatures around 140°C; this technique is called "hot cutback spray sealing".

Emulsified bitumen is also used in spray sealing and has the advantages of lower spraying temperatures of around 85 °C and less kerosene. Once applied to the road, water in the emulsion evaporates leaving a bituminous layer similar to standard cutback bitumen. This sealing technique is called "emulsion spray sealing". Stone chips or *aggregate* are then spread over the bitumen to produce a *chip-seal*. The development of a bitumen emulsion for use in spray sealing to produce chip-seals is the area of interest to the project.

1.3.2 High Bitumen Content Emulsions

Standard bitumen emulsions contain from 55%-65% bitumen; the remainder being water, solvent, emulsifier and acid. This relatively low proportion of bitumen has limited the application of bitumen emulsions in standard chip sealing. By increasing the percentage of bitumen in the emulsion a number of these limitations are overcome:

- Lower transportation costs due to less water cartage.
- Greater application efficiency.
- Larger sized chips can be used as greater *binder* depths can be achieved.
- Reduced cure times.
- Greater productivity.
- Potential reduction of environmental impact and energy consumption.

Difficulties however arise when increasing bitumen content, including:

- Exponential increase in viscosity causing difficulties with handling and spraying.
- An emulsion which rapidly *breaks*.
- Short storage life.
- Potential inversion from an 'oil in water' emulsion to a 'water in oil' emulsion.

A number of ways have been developed to increase bitumen content to 75%-80% and overcome these problems (Duval and Beranger, 1989; Holleran, Booth and Maccarrone, 1993; Marchal, 1995). These methods along with basic bitumen science and technology are reviewed in Chapter 3. Based on this research and the design work conducted, this study attempted to develop a high bitumen content emulsion for production and application by Higgins for New Zealand conditions.

The development of the high bitumen content emulsion followed the systematic product development process developed. Information was gathered to gain an understanding of the science and technology of the industry and to establish project feasibility. Customer research was undertaken to formulate a design specification which would guide the design phases. Scale-up field trials were conducted using the finalised laboratory formulation in an attempt to demonstrate final product performance.

1.4 PROJECT AIMS AND OBJECTIVES

1.4.1 Project Aim

The aim of the project was to develop, within a specific company environment, an appropriate product development approach with supporting systems. The approach would be used to develop a high bitumen content emulsion for use in spray sealing applications.

1.4.2 Objectives

- Understand science & technology related to high bitumen emulsion production.
- Develop performance criteria relevant to New Zealand conditions.
- Produce an optimum product which meets performance criteria.

- Conduct large scale field trials.
- Correlate laboratory development with field performance.
- Provide a tailored product development approach applicable to current and future projects.
- Develop product development systems for the various stages within the entire approach.

1.4.3 Constraints

1.4.3.1 Product constraints

- Achieve a bitumen content of 75% or greater.
- Meet relevant industry specifications and standards.
- Not infringe patent laws.
- Product performance be acceptable to the customer, contractor and user.

1.4.3.2 Processing constraints

- Prototypes to be produced on Higgins laboratory size colloid mill.
- Optimum formulation to be produced using existing production plant and technology.
- Commercially available raw materials to be used.

2. REVIEW OF PRODUCT DEVELOPMENT PROCESSES

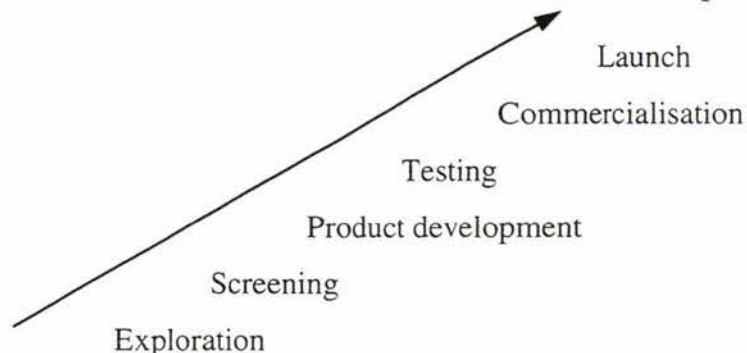
2.1 INTRODUCTION

The purpose of this chapter is to review a number of different product development processes. These processes were used to develop a tailored product development process for Higgins Contractors Limited. The review begins with a brief history of the product development process followed by an overview of four relevant product development processes. An in-depth analysis of a product development process most relevant to Higgins is also reviewed to provide a basis to formulate a tailored development method.

2.2 PRODUCT DEVELOPMENT PROCESS EVOLUTION

Product development is a fairly new discipline in itself with the first formalised study being conducted in 1968 by Booz-Allen and Hamilton. A large number of companies were investigated to determine the type and order of activities carried out to bring new products to market. The management of this process was also investigated. Findings revealed the more successful companies carried out product development in a systematic and structured way with definite stages. These successful companies also placed more resources into product development. The 1968 Booz-Allen and Hamilton study identified six stages in the product development process; refer Figure 2-1. This rather primitive outline provided the first insights into product development as a defined process.

Figure 2-1 Booz-Allen and Hamilton's 1968 Product Development Process



Many of these early product development processes were carried out in companies that were highly functionally structured. This often meant stages were carried out in sequence by single departments and then passed onto the next with little continuity and communication. For example screening and exploration was often carried out by marketing personnel then the product was handed onto technical staff for development and then to production staff for testing and process development. Much re-design occurred due to the lack of communication and understanding of the needs of the other departments. This type of system was used in the 60's by NASA and served to double development time (Cooper, 1993). Modern product development practise favours more of an integrated team approach with greater consultation.

Many studies into the product development process continued over the next few decades with the 1968 Booz-Allen and Hamilton process expanded and refined. Kerr, (1991) cites the following additions to the basic product development process. Earle developed a process in 1971 which included specific Go-No-go points at which the details of activities and gathered information were reviewed. The Earle process also integrated greater market research. The importance product development played in the strategic direction of the company was recognised and a 'Product Strategy' step was added by Booz-Allen and Hamilton (1982). This strategy guided the company's product development activities and eventual business direction.

A focus on conducting good ground work was also established with more investigation into project feasibility and into drafting design specifications (Cooper, 1983). Multi-functional teaming and a concentration on consumer input were also trademarks of the 1980's. The International Association of Product Development also recognised in 1990 the requirement of product developers to design products for responsible product disposal. This was driven by environmental legislation and product life-cycle analysis theory (Dimancescu & Dwenger, 1996). These advances in product development have refined early processes to the modern practise of product development.

2.3 PRODUCT DEVELOPMENT PROCESSES

Five appropriate product development processes relevant to Higgins operations will be reviewed to gain a broad understanding of various product development processes.

2.3.1 Hass's Industrial Product Development Process

Hass (1989) presented a process for product development suited to industrial products which is consistent with the nature of Higgins operations. Hass presented a five stage process shown in Figure 2-2. with three streams running in parallel: marketing, engineering and management. This illustrates the required interplay between these three areas for the product's development.

The five distinct steps were: (1) Basic screening- a preliminary investigation to decide the potential of an idea with respect to company objectives. (2) The decision to develop product design specifications- determining what product is to be made and the time frame it is to be made in. (3) The decision to release for development- the development of a product prototype and laboratory testing. (5) The decision to commercially produce the product-commercial product launch.

The process, although industrially focused, appears to be more suited to manufactured goods rather than processed products which Higgins produce, this is indicated by the use of terminology such as 'engineering' and 'tooling'. The general structure is, however, useful and adaptations to the process can be made to suit the formulated and processed nature of Higgins products.

2.3.2 BS-7000 Product Development Process

The British Standard (1989) sets out a framework which is designed to be applicable to a range of industries and addresses product development at both the corporate level and the project level. The standard provides practical guidelines for the implementation and management of product design and development. Figure 2-3 illustrates the BS-7000 product development process.

The BS-7000 method approaches product development more from of a life cycle analysis point of view, describing products as they go through the process as unused, installed, used and recycled. This view broadens the concept of product development beyond product launch to include disposal. This increases the constraints the product developer must consider when designing a product.

Figure 2-2 Hass's Industrial Product Development Process.

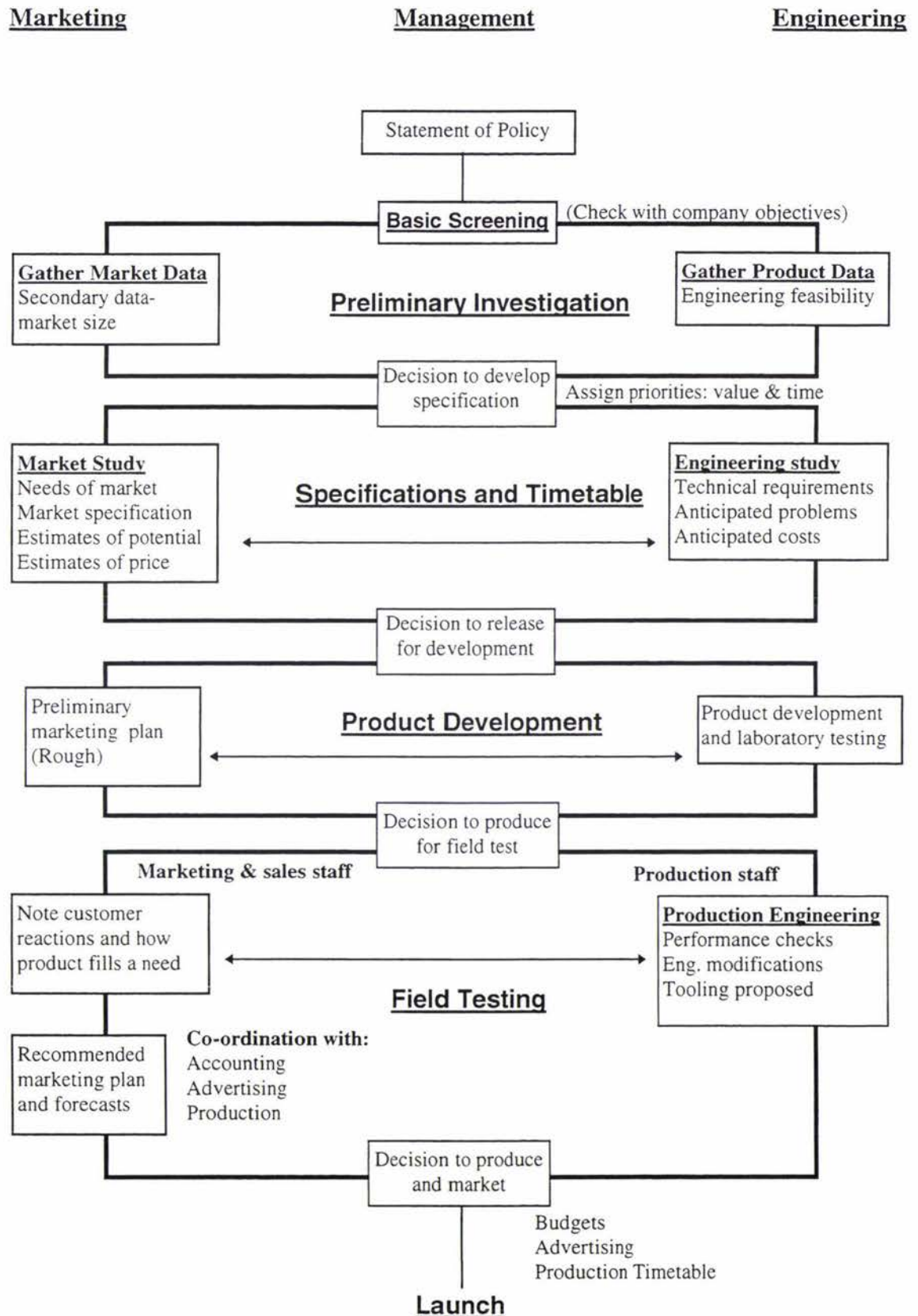
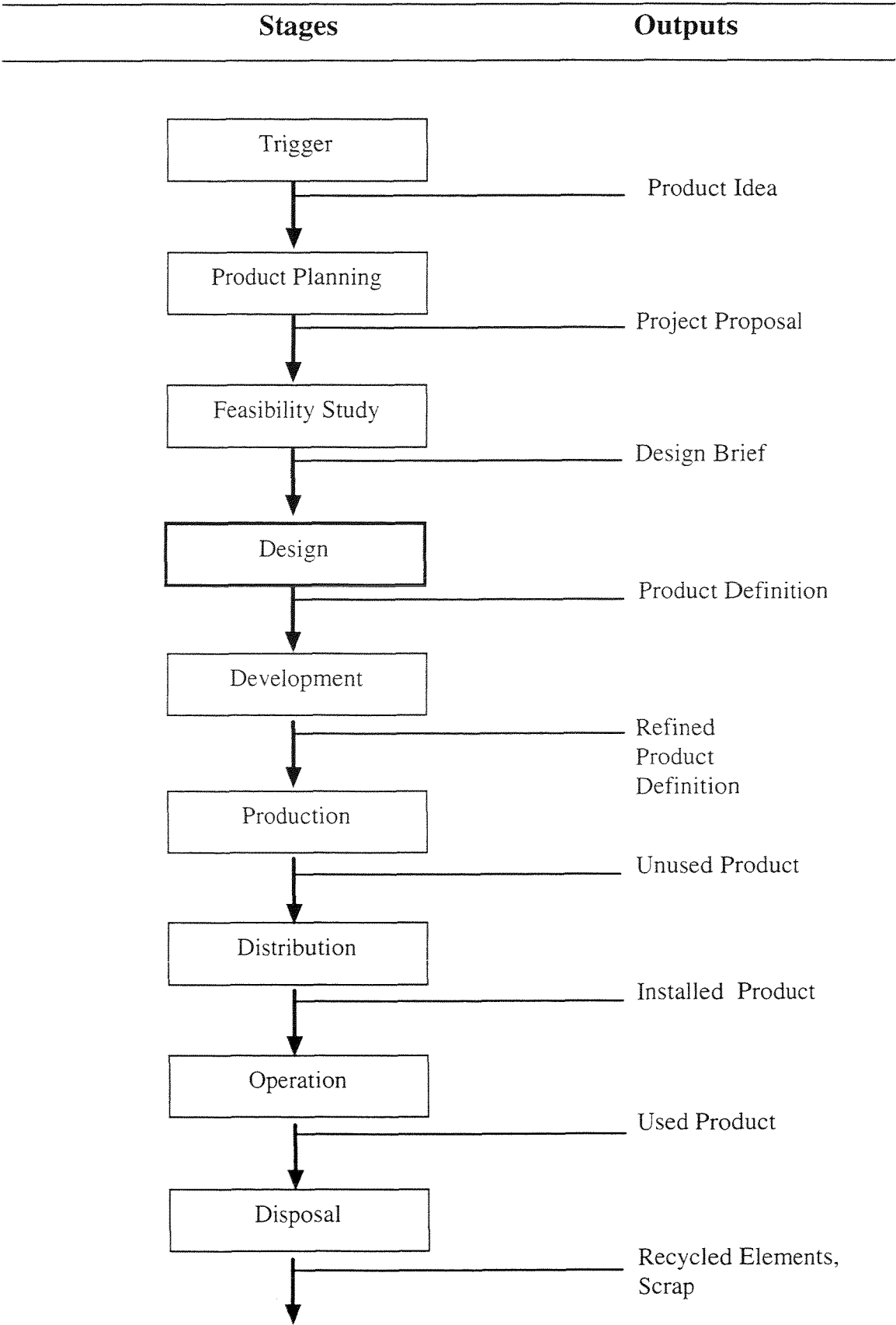


Figure 2-3. BS-7000 Product Development Process.



Comparing this method with the Hass process, the multi-functional nature of the process and the definite decision points where progress is reviewed are not emphasised. A strength of the BS-7000 process however is its view of design. The BS-7000 breaks the design phase down into four further stages which is useful for the management of this critical step.

1. Conceptual design:

A large number of ideas are conceived and general working principles are outlined.

2. Embodiment design:

The process where structured development of the preferred concept is carried out.

3. Detail design:

The process in which the precise product specifications are confirmed.

4. Design for manufacture:

The process where the detailed design is related to the manufacturing process.

Most R&D projects spend at least a third of total project time and effort in the design phase, it is therefore important to manage this step well (Anderson & Hein, 1987). By breaking down the design activity, monitoring and control of this phase is made more exact. The BS-7000 view of design is also focused towards manufactured products rather than processed products but application of the principles can be made to processed products at Higgins.

2.3.3 Earle's Product Development Process

Earle, 1997 offers a four stage product development process. Each stage is divided into a number of activities which produce outcomes on which management make decisions. Between each stage definite decision points must be passed to proceed with development. The four broad stages presented are: 1. Product strategy and planning 2. Creation and development of product 3. Production process, marketing strategy, quality assurance and commercial product 4. Launch and post launch. The process can be seen in Appendix 2.

The approach considers the entire product life cycle from strategic business planning to the evaluation of existing mature products. When inspected, this method acknowledges the multi-functional approach needed to generate the various outcomes such as

marketing, financial, technical and processing plans. The process provides practical insights into what is to be achieved and decided during the entire process.

2.3.4 P.A.C.E Product Development Approach

P.A.C.E is an approach to product development developed by McGrath et al. (1992) based on research conducted on 100 projects from numerous companies. P.A.C.E is an acronym for Product And Cycle-time Excellence. The management philosophy behind P.A.C.E has seven elements, one of the elements is a product development process. The five phases of the P.A.C.E product development process are:

- Phase 0. Concept evaluation
- Phase 1. Planning and specification
- Phase 2. Development
- Phase 3. Testing and evaluation
- Phase 4. Product release

The P.A.C.E approach appears to have been influenced by manufacturing process improvement evidenced in the application of manufacturing cycle time theory to the product development process. The entire product development process is likened to a manufacturing process which can be broken down into smaller and smaller tasks with estimated times for completion or cycle times. The sum of these task times produces a total product development cycle time. The five development phases are broken into 15-20 steps, these steps are broken into 10-30 tasks each with 30-40 activities. Steps and tasks are project independent while activities are project specific. The cycle times allocated to each activity are then used to plan resources. Improvements to the product development process can be achieved by examining individual tasks or complete steps to determine how to make them more efficient. This view of product development and its improvement is similar to theories applied to manufacturing processes (Patterson, 1993).

Decision making in the P.A.C.E approach is managed through an appointed Product Review Committee (PRC). The committee consists of upper management with authority to allocate resources. A core project team pools a number of cross functional managers who are responsible for the practical development of the product. The nature

of the structure promotes communication and parallel development by many parties, there-by increasing the rate of product development.

The core team enters one of the five development phases to produce outputs which are presented to the product review committee for evaluation. The committee reviews the information presented to it and makes a decision on the further allocation of resources. The review committee makes its decisions based on set criteria and the company's product strategy which guides the strategic direction of the companies products.

The P.A.C.E approach tends to focus on planning and scheduling the many activities in the process. The main criticism with the approach is the tendency to be over focused on developing elaborate plans which are time consuming and take the focus off more valuable product development activities. Personnel can get focussed more on keeping to the schedule rather than conducting work thoroughly, while management get frustrated when schedules are not adhered to. The many unknown variables in product development can also lead to inaccurate and misleading plans with unforeseen delays occurring. Planning is key to good project management but must remain a tool and avoid becoming burdensome.

The P.A.C.E system provides an outline of good project management and team structure. The concept of applying cycle times to the various stages and steps is also a useful way to assist in product development improvement and resource planning.

2.4 COOPER'S STAGE-GATE PROCESS

Cooper's stage-gate product development process is a widely used and well recognised product development approach, it is applicable to both industrial and consumer goods product development. It has been implemented in small and large companies alike, including Proctor and Gamble, Exxon chemicals, Dupont and Polaroid. Cooper's process for product development incorporates many of the strengths of the previously reviewed methods into one process making it an appropriate base process to develop a specific product development process for Higgins and is reviewed in depth. A number of points made this process more suitable.

- Applicable to industrial processed products
- Easy to understand, use, and implement
- Able to be used in a small company
- Flexible to future growth and change
- Widely recognised and proven approach
- Incorporates much of product development best practise

2.4.1 Product Development Stages

Cooper sets out five distinct stages:

1. Preliminary Investigation
2. Detailed Investigation
3. Development
4. Testing and validation
5. Full production

The five stages are separated by five decision gates; Figure 2-4 illustrates the overall process. In order to move onto a new development stage, the product concept must pass certain criteria at the decision gates. Go/Kill decisions can be made at any one of the gates to halt the project if it becomes un-viable. The purpose and activities of each stage and gate from Figure 2-4 are outlined to provide a more detailed understanding of the process.

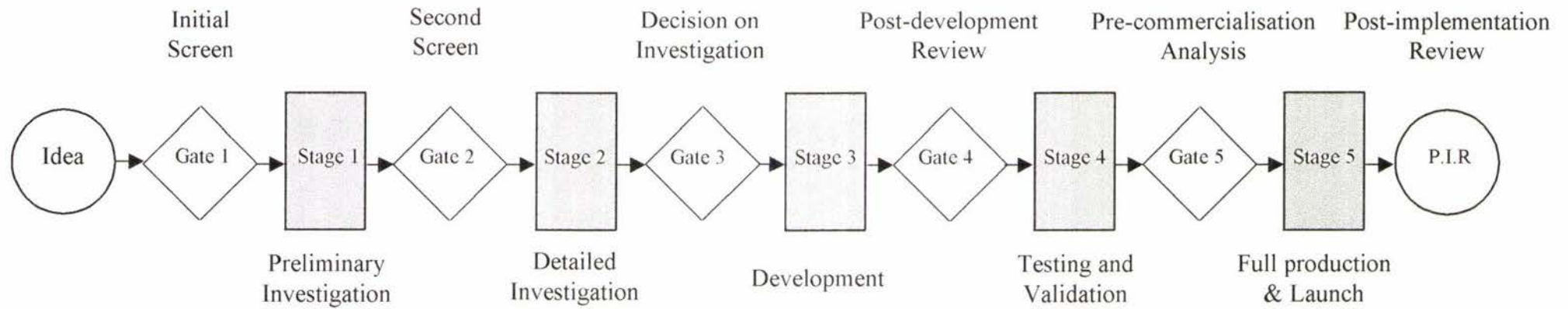
2.4.1.1 Idea

The project is initiated through an idea. The ideas coming from customers, staff, basic research or creativity techniques. An environment which encourages creativity, aids in a healthy flow of potential development projects.

2.4.1.2 Gate 1. Initial Screen

Gate 1 is a gentle screening process where often checklists are used, containing 'must have' criteria and 'should have' criteria. A lack of a single 'must have' criteria will kill the idea as a project. Scoring models can also be used to weight and score ideas. At this stage financial assessments are not generally included. A decision to accept the project is the first commitment to funds.

Figure 2-4 Cooper's Stage-gate Product Development Process



2.4.1.3 Stage 1. Preliminary Investigation

The aim of a preliminary investigation is to gather market and technical data quickly and inexpensively. The preliminary market assessment may involve a search of current products, library search and discussions with key customers. The preliminary technical assessment may involve appraisal of the idea with technical and manufacturing personnel to identify possible risk areas. The pareto rule applies here, 80% of the most significant data can be obtained in a short space of time to allow clearer decision making. This stage is usually done in less than 1 month.

2.4.1.4 Gate 2. Second Screen

The same criteria from the initial screen are used at the second screen but applied more rigorously. A preliminary financial assessment is made, this is usually a pay-back type calculation and not a detailed net present value analysis of cashflows. This gate provides a checkpoint to identify or redirect inappropriate development direction.

2.4.1.5 Stage 2. Detailed Investigation

This stage clearly defines the product to be developed. Activities required include:

- Definition of the target market.
- Positioning strategy determined.
- Product benefits, features and requirements are outlined.
- Market research and competitive analysis are undertaken.
- Detailed financial, technical and manufacturing assessments.
- Development strategy planned.

Initial designs can be explored to assist in establishing feasibility but no detailed design is embarked upon. This is mainly where a business case is built up to fully justify where and what development will be focused on.

2.4.1.6 Gate 3. Decision on Investigation

This gate provides the final assessment of the project before heavy spending and a large time commitment is made to the project. The thoroughness of the investigations needs to be assessed and any grey areas clarified before proceeding. At this gate the business case containing feasibility and product specification information is signed off as formal agreement of direction and authorisation to begin development.

2.4.1.7 Stage 3. Development

The emphasis of this stage is on technical work to fulfil the planned specification. This stage is often a lengthy process which should be broken down through the attainment of milestones. Reviews are best conducted at milestones rather than time intervals as clearer decisions can be made at a more complete stage. These milestone reviews are not Go/Kill decisions but provide good project management and control to evaluate results as they come to hand. During this stage marketing will be preparing detailed plans, while manufacturing will be preparing for the testing and validation phase.

2.4.1.8 Gate 4. Post-development Review

This checkpoint assesses the continued attractiveness of the project. An assessment is made on the product's ability to perform as set out in the product design specification. Development work is reviewed to ensure all required steps have been carried out to a high standard. Marketing and manufacturing plans are reviewed with the expectation of execution. Financial measures are also reviewed with a clearer estimate of cost being possible.

2.4.1.9 Stage 4. Testing and Validation

This stage validates the entire project as the production process, user acceptance and financial aspects can be more accurately judged. Typical activities to be conducted during the stage include:

- In house testing- Extended laboratory trials.
- Limited pilot trials- To test production variables.
- Trial sell- Initial user tests to gauge reaction to product.
- Revised financial analysis- Continued checking of project viability.

Demonstrated performance in all areas under end-use conditions will be the main criteria being assessed.

2.4.1.10 Gate 5. Pre-commercialisation Analysis

This gate opens the door to full commercialisation, launch and production. This is the final kill point for the product with the key focus on financial return based on a proven product. The final marketing and operations plans are also reviewed and approved.

2.4.1.11 Stage 5. Full production and Launch

This stage sees the implementation of the marketing, launch and production plans. Good planning and sufficient resources make this stage run smoothly.

2.4.1.12 Post-implementation Review

After the product has been in use for around 6-18 months an idea of the products performance and acceptance can accurately be gauged. This is an appropriate time to review the project. Projections are measured against actual performance to guide future predictions; problems are reviewed and improvements to both the product and the development process are discussed. This stage is a time for reflection to improve performance and provides an opportunity to identify any further design improvements for future generation products. The review also marks the completion of the project with the team disbanding and the product officially becoming a routine product.

2.4.2 Management Issues

Cooper sets out management goals to support the systematic process and provide a total approach to product development:

- Quality of execution
- Sharper focus
- Managed risk
- Parallel development
- Multi-functional teaming
- Strong market orientation
- Strong pre-development activity

The quality, completeness and consistency of executing product development was stressed by Cooper. The fact was highlighted that no matter what form the product development process takes, if gaps were present or the execution of steps was poor the result are longer development and poorer products.

Sharper focus is brought through the 'Gates' in the process as decision making is concentrated at specific points. The gates need to have tough criteria which are applied consistently across all projects to eliminate unprofitable and time wasting developments.

Stringent gate criteria will result in a sharper focus on more profitable projects while solving the dilemma of too many projects and not enough resources.

The idea of risk and uncertainty is applied to the allocation of resources throughout the process. As the product takes shape through the stages there is less uncertainty about the product and the risk of failure decreases. The demonstrated reduction in risk at each gate means more resources can be safely allocated. Allocating resources in smaller lots reduces the chance of large scale losses.

Parallel development provides a faster rate of development as a number of facets of the product mix are being developed concurrently. Cooper likened this to a rugby game where all players are running down field in the same direction passing the ball amongst themselves. At certain points the team converges in a huddle or scrum and then move forwards again. The ball was likened to the product and the scrums to the decision gates. Parallel development implicitly requires the use of a multi-functional team. If a number of different facets of the product are to be produced at once, interaction between the areas needs to be structured. A multi-functional team provides the structure to produce a cohesive development approach.

Strong market orientation is the most critical factor for product success according to Cooper. Numerous studies were cited to back this including the SAPPHO project, Booz-Allen and Hamilton's study of 700 firms and Cooper's own research into 203 companies. At the top of all these studies, market need and an orientation towards customer requirements was placed as the most critical factor for success. A strong market focus needs to be built into the product development process for consistent product success. This means carrying out better market evaluation and greater consumer testing along the development path.

More time spent on pre-development activities was also shown in Cooper's studies to produce more successful products. Information gathering, feasibility and design specification are all carried out prior to development and are relatively inexpensive exercises compared to later development. These activities focus and clarify development making the design activity a less risky and lengthy phase.

The early investigations will mean the right product is developed in the right way for the right reasons.

2.5 SUMMARY AND CONCLUSIONS

Analysis of the various product development processes revealed several recurring themes; Cooper's product development process incorporated many of these key themes into one process and was the most appropriate process for use at Higgins. Reflecting on the main points gathered from the literature provides an understanding of the aspects to be included in the approach developed for Higgins which is detailed in Chapter 4.

Product development is guided by a product strategy formulated by top management. This strategy guides the companies product range and precedes any development work. Project ideas are screened by various methods according to the product strategy and attractiveness criteria. This was highlighted by Earle's product development process. Market need is a key factor for successful product development. Understanding the needs of customers can be gained by obtaining their input throughout all key stages of development to judge product performance and indicate likely acceptance by the market. Without a demand for a product little success can be expected.

A structured development process is used which does not vary greatly across different projects. This increases the rate of product development as new methods do not need to be made up for each new project. Template documentation can be used to reduce paperwork, increase the efficiency of reporting and speed up the decision making process. The process is divided into stages which are separated by management review. This reduces risk by ensuring the product remains attractive throughout the entire development process and remains correctly focused. It also provides good project management and accountability.

Five broad stages were identified and best summarised by Cooper's product development process.

Stage 1. Project initiation

- Idea capture and screening.

Stage 2. Investigation

- Initial investigation to outline the proposed idea.
- Detailed feasibility investigation and design specification.

Stage 3. Design and development

- Initial design to determine a preferred concept
- Detailed design to refine and optimise preferred concept

Stage 4. Testing and Validation

- Scaling-up product to actual processing facility and conducting in-use testing.

Stage 5. Full production and product launch

- Producing consistent product and establishing it in the market.

A large number of product ideas and concepts are gradually and deliberately focused through the design and development process to emerge with a validated design. A large number of concepts are started with so potential solutions are not eliminated too early, as the process proceeds more commitment can be placed into the better design solutions.

Development is a crucial and often lengthy phase which can be broken into a number of distinct parts to exert greater control over the process.

Validation and testing of the design must prove the ability to produce a product on full scale equipment that is acceptable to customers. Customer acceptance and financial return are the key validation criteria to be met before moving to product launch.

The entire product development process can be broken into smaller elements for greater resource planning and to aid process improvement.

A multi-functional team approach is more suitable in product development. Multi-functional teams allow the rapid development of a number of areas at once by specialists with less oversights. Technical, marketing and financial functions need key involvement.

Incorporating these principles into a tailored product development approach will provide Higgins with a quality product development approach which will produce more consistent success.

3. REVIEW OF BITUMEN EMULSION SCIENCE AND TECHNOLOGY

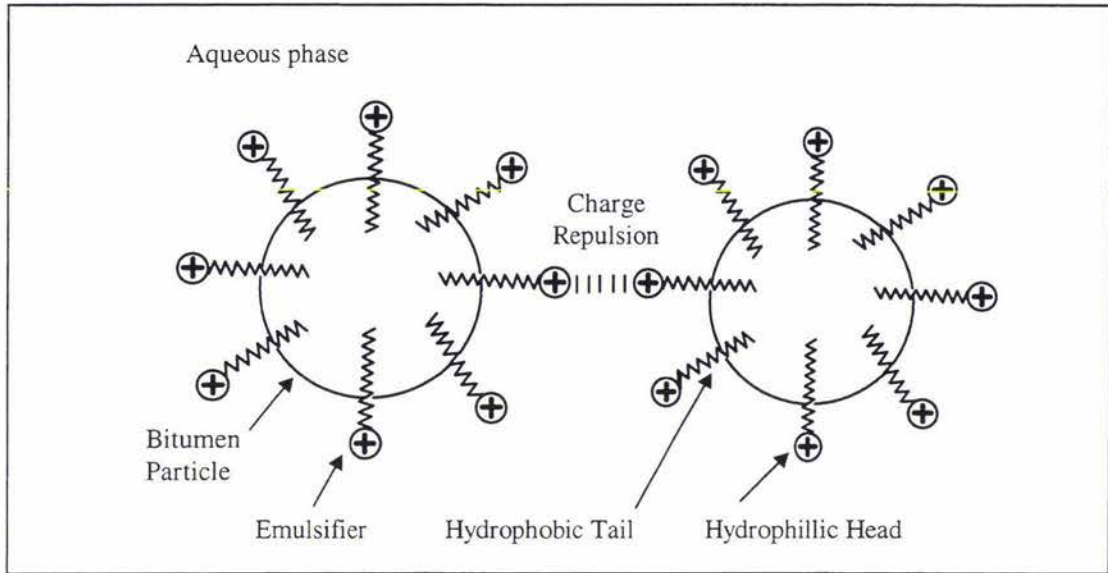
3.1 INTRODUCTION

The review covers emulsification principles, raw materials, production and application methods of general bitumen emulsions. Several processes with regards to high bitumen content emulsions are also reviewed. Knowledge in these areas assist in making informed decisions in later product design.

3.2 BITUMEN EMULSIFICATION

An emulsion is defined as a dispersed system in which the phases are immiscible or partially miscible liquids (for example; Shaw, 1992). Emulsification is the process which allows the stable mixing of the liquids through the aid of an emulsifier; an emulsifier being defined as chemical substances which stabilises or preserves an emulsion (for example; Chambers, 1991). In bitumen emulsions, bitumen particles are dispersed in water through the aid of an emulsifier. The water phase is also referred to as the aqueous, soap, continuous, or dispersing phase.

The emulsifier generally consists of a long chain hydrocarbon molecule with *hydrophilic* and *hydrophobic* ends. The hydrophobic tail of the molecule is attracted to the bitumen, while the hydrophilic head is attracted to the water. The head of the emulsifier molecule generally has an ionic charge; positive for cationic emulsifiers, negative for anionic emulsifiers. A number of emulsifier molecules attach themselves to bitumen particles producing charged globules. When many of these like charged globules are in solution they repel one another. The particle repulsion produces the separation required to stabilise the emulsion. Figure 3-1 illustrates in a simplified manner bitumen emulsion stabilisation.

Figure 3-1 Basic Representation of Emulsified Particles

3.3 RAW MATERIALS

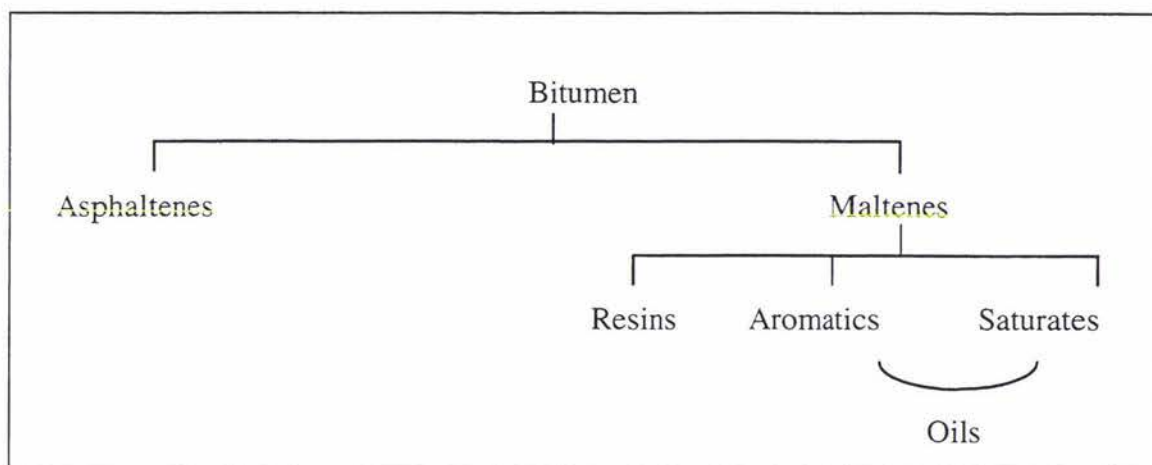
Bitumen emulsions generally consist of six main components; bitumen, solvents, emulsifier, acid or alkali, water and additives.

3.3.1 Bitumen

Fractional distillation is used to refine crude oil into bitumen. Lighter hydrocarbon fractions are distilled off to leave a residuum in the bottom of the distillation tower; this is the starting material for bitumen. This fraction is further refined with the distillation of heavy gas oils under vacuum to produce straight run bitumen. Harder grades of bitumen are produced by air blowing or solvent extraction processes. Intermediate grades are produced by blending the harder grades with the straight run bitumen.

Depending on the source of crude oil, bitumen chemistry and properties will vary (Harrington, 1993). Bitumen used in New Zealand is sourced from the Middle East and therefore has unique properties compared to bitumen from other nations such as Venezuela and the United States. Understanding the properties of bitumen used in New Zealand assists in more informed product design. Figure 3-2 illustrates the main constituents present within bitumen. Bitumen constituents can be divided into two main classes, solid asphaltenes and fluid maltenes. The relative ratios of these components plays a major role in the quality and performance of the final emulsion (Roberts et al., 1991).

Figure 3-2. Main Chemical Constituents of Bitumen.



The asphaltene fraction within bitumen used in New Zealand has the greatest impact upon emulsions developed here. A relatively high asphaltene content characterises locally used bitumen compared to that from other sources such as Venezuela (Harrington, 1993). Asphaltenes are harder to emulsify and therefore will impacts on the level and type of emulsifier required. Increases in this fraction also has the greatest effect on rheology, producing harder bitumen with higher viscosities (Roberts et al, 1991).

The Middle Eastern bitumen used in New Zealand also has lower acid values and higher salt contents compared to other bitumens which make the bitumen more difficult to emulsify (West 1985).

3.3.2 Solvents

Solvents used in the industry include kerosene and turpentine and are often referred to as '*cutter*'. Cutters temporarily alter bitumen viscosity and are added to emulsions to improve stone *wetting*. Improved wetting allows stronger adhesive bonds to be formed at the chip/bitumen interface resulting in better stone retention (Reed & Holleran, 1996). Cutters will eventually evaporate from the binder. Automotive gas oil (AGO) or diesel is another solvent used in the industry which permanently softens bitumen and is known as a fluxing agent. At low temperatures, fluxes prevent bitumen from becoming brittle at which point stones can be easily torn from the road surface.

3.3.3 Emulsifiers

Emulsifiers can be divided into four main categories based on their ionic disassociation in water: anionic, cationic, amphoteric and nonionic (Asphalt Institute, 1992). Anionic and cationic emulsifiers are the most common types used in the roading industry.

Anionic emulsifiers were commonly used in the 1930's and 40's. The reduction in their present day usage is due to the nature of the chemistry involved. Anionic emulsifiers disassociate to produce negative charged ions which do not chemically react with similarly charged aggregates. Cationic emulsifiers however disassociate to produce positively charged ions which react readily with negatively charged aggregates; accelerating emulsion cure and improving adhesion (Neaylon & Gillespie, 1992). Anionic emulsifiers are solely dependant on water evaporation for curing and therefore have been increasingly replaced by cationic emulsifiers.

Cationic emulsifiers may have between one and three amino-nitrogen groups and are classed accordingly. The three groups are monomaniacs, diamines and tertiary amines (Johnson, 1979). The structure of the emulsifier impacts on the ability to emulsify or impart charge onto bitumen particles to produce stable particle separation. The preferred cationic emulsifiers for high bitumen content emulsions are from the diamine family (Emoleum, 1990).

Generally the emulsifier will be added at around 0.1- 0.5% based on total emulsion weight. Beyond these levels the emulsifier is unable to be adsorbed onto the bitumen particles and will remain in the aqueous phase increasing viscosity and lowering adhesion (Marchal et al., 1992).

3.3.4 Salts

Addition of salts to bitumen emulsions improves the stability by promoting ionic structure in the aqueous phase. Common salts used in the industry are sodium, calcium and other metal-halide salts and are generally added at approximately 0.1% on total emulsion weight. Over-addition of salt however results in the ions adversely interacting with the emulsifier stabilisation mechanism causing the emulsion to break down (Menon & Wasan, 1988).

Various natural salts are present in bitumen. These salt molecules attract water into the bitumen and in the process a pressure differential is set up between the bitumen and aqueous phases; the pressure differential is known as osmotic pressure. Water attracted to the salt molecules become trapped inside the bitumen particles causing an increase in osmotic pressure. This reduces the apparent water content in the aqueous phase and increases viscosity. Adding salt to the aqueous phase restores the osmotic pressure by causing the water to migrate back from within the bitumen to the aqueous phase (SFERB, 1991).

3.3.5 Water

The presence of ions is the single most important factor concerning water quality for emulsion production. Calcium and magnesium ions assist in making a more stable cationic emulsion, whereas the presence of phosphate and carbonate ions form insoluble salts with amine hydrochlorides present in cationic emulsifiers (Dybalski, 1985). Water used at the Higgins production facility located in Napier uses artesian bore water generally higher in calcium and magnesium ion concentrations.

3.3.6 Acids

Acids are used to *ionise* cationic heads of emulsifiers to allow dispersion in water. Hydrochloric acid is the acid most frequently used in the industry but other mineral acids such as phosphoric and sulphuric acid are also used. Acidity and pH have a major impact on emulsion properties such as adhesion and cohesion and is therefore a critical factor to be controlled during production.

3.3.7 Additives

Other additives are placed in the emulsion to improve performance, listed below:

- Anti-stripping agents- Prevent bitumen from being removed from the stone.
- Viscosity builders- Improve spraying and application performance.
- Anti-oxidants- Improve the ageing characteristics of bitumen.

Polymers however are the biggest growth area in relation to additives (Exxon, 1996). Polymer modification reduces bitumen temperature susceptibility allowing better performance at higher and lower temperatures. The main advantage however of polymer modification is the increase in strength of the bitumen. The greater strength widens the applications for bitumen and provides better performance in high stress areas

such as roundabouts and corners (Beiter & Dean, 1992). Polymers can be broken into two main classes: elastomeric and polymeric. Elastomeric polymers provide greater resistance to torsional stresses and provide flexibility to a pavement to avoid cracking. Common examples of elastomeric polymers include: Natural Rubber (NR) Styrene Butadiene Styrene (SBS), Styrene Butadiene Rubber (SBR). Polymeric polymers provide greater strength and stiffness to a binder to reduce rut formation. Examples of polymeric polymers include: Ethylene Vinyl Acetate (EVA) and Ethylene Methyl Acrylate (EMA).

3.4 EMULSION PRODUCTION

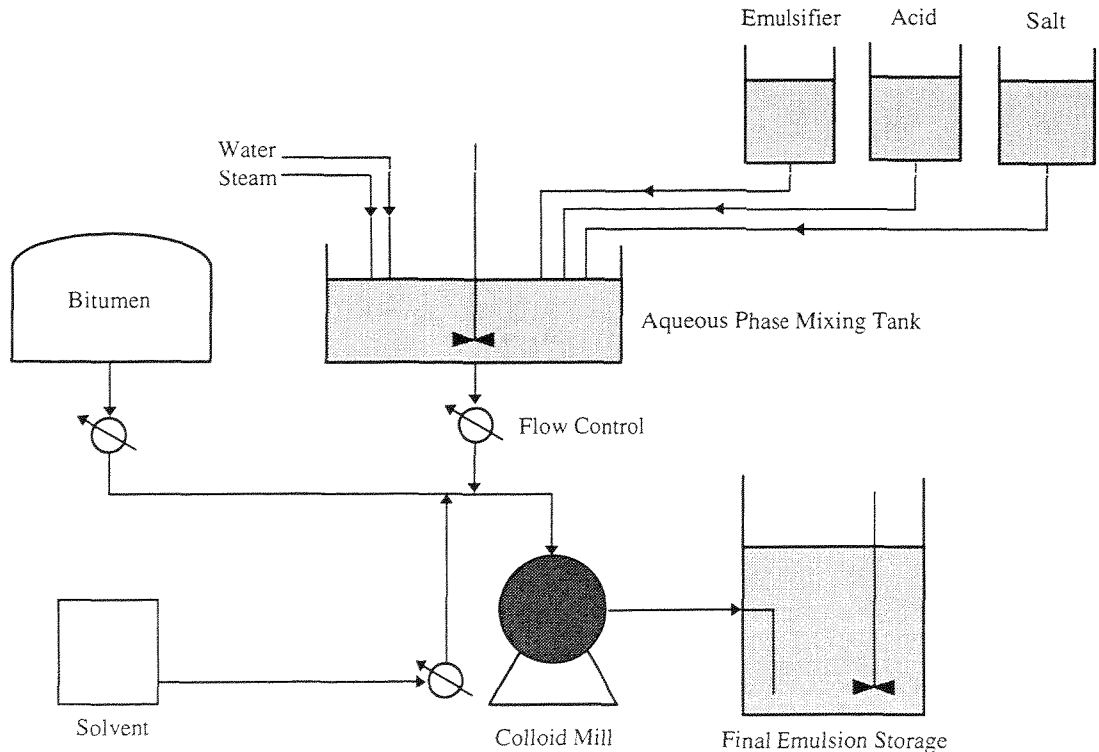
The production process takes raw materials and combines them in such a way to produce the desired product. By simply varying processing conditions an emulsion with very different end properties can be produced using the same formulation and raw materials. This highlights the need for good process control and an understanding of production variables.

3.4.1 General Production Process

Production of a bitumen emulsion begins with the preparation of the aqueous phase. A concentrate 'soap' solution is prepared which generally contains the cationic emulsifier, acid and salt. Other additives such as viscosity modifiers, polymers and antistripping agents can also be added in the aqueous phase. A percentage of the total water is added to the mixing tank and heated. Salts and a proportion of the acid are then added followed by the emulsifier; the acid must be added prior to the emulsifier to aid dispersion. Once the concentrate is thoroughly mixed the remaining water is added and the pH is adjusted to the required level by further addition of acid.

Bitumen and solvent make up the dispersed phase (also known as the bitumen phase) and together are known as *binder*. The three streams (bitumen, solvent and soap solution) are fed separately but simultaneously into the colloid mill to form the emulsion; this is referred to as co-milling. Addition of components before or after milling is referred to as pre-blending or post-addition. A schematic diagram of the production process is shown in Figure 3-3.

Figure 3-3. Schematic Diagram of Emulsion Production Process.



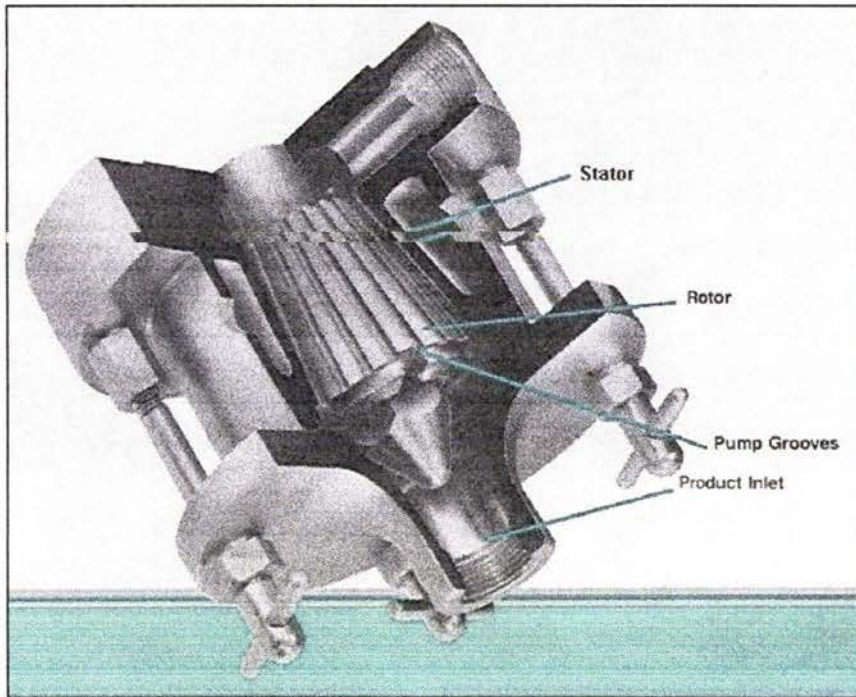
3.4.2 Colloid Mill

A colloid mill provides the high shear force required to obtain a uniform dispersion of the binder amongst the aqueous phase. The mechanical action creates fine particles onto which the emulsifier molecules are adsorbed to maintain the particle separation. The Charlotte colloid mills used in the laboratory and in the Napier production facility consist of a moving rotor and a stationary casing called the stator. Grooves are machined into these components to create pressure differentials aiding the dispersing process. Figure 3-4 illustrates the main components of the Charlotte colloid mill.

3.4.3 Processing Variables

Processing variables such as mill gap, mill pressure, bitumen temperature, soap temperature and exit temperature are significant variables to be controlled during production.

Figure 3-4 Internal Components of a Charlotte Colloid Mill



Photograph from Charlotte Colloid Mills, 1989.

- **Mill gap.** The gap between the stator and rotor has a large influence on the particle size and distribution created. This in turn influences final product characteristics such as viscosity and storage stability. The mill gap is generally between 0.25mm and 0.5mm and will create an emulsion with an average bitumen particle size of 0.005 mm (Asphalt Institute, 1992).
- **Mill pressure.** Colloid mills can be operated above atmospheric pressure by increasing the pressure on the exit line. This increases the boiling points of components to gain better control of particle size and viscosity. Higher mill pressures produce finer and more stable emulsions (Duval and Beranger, 1989). Mills operated under pressure generally require a heat exchanger downstream to cool emulsions below 100 °C to avoid degradation by boiling.
- **Bitumen temperature.** Bitumen temperature affects the viscosity of the material as it enters the mill and thus affects the resultant particle size (Whiteoak, 1990). Higher bitumen temperatures reduce bitumen viscosity making the dispersion of the material easier. Bitumen temperatures range from 120°C to 150°C depending on the asphalt grade and required exit temperature.

- Soap temperature. Soap phase temperature is partly dependant on the temperature at which the emulsifier will disperse and the required exit temperature. Solid emulsifiers require higher temperatures to aid dispersion compared to liquid emulsifiers. A soap temperature between 30°C and 60°C is typical.
- Exit temperature. Exit temperature is not a processing variable which can be directly controlled but is dependant on the mass and temperature contributions of the bitumen and soap phases entering the mill. This variable is carefully monitored during production as it is a key indicator of final emulsion viscosity (Booth et al. 1992). Exit temperatures between 85 and 90 °C are acceptable.

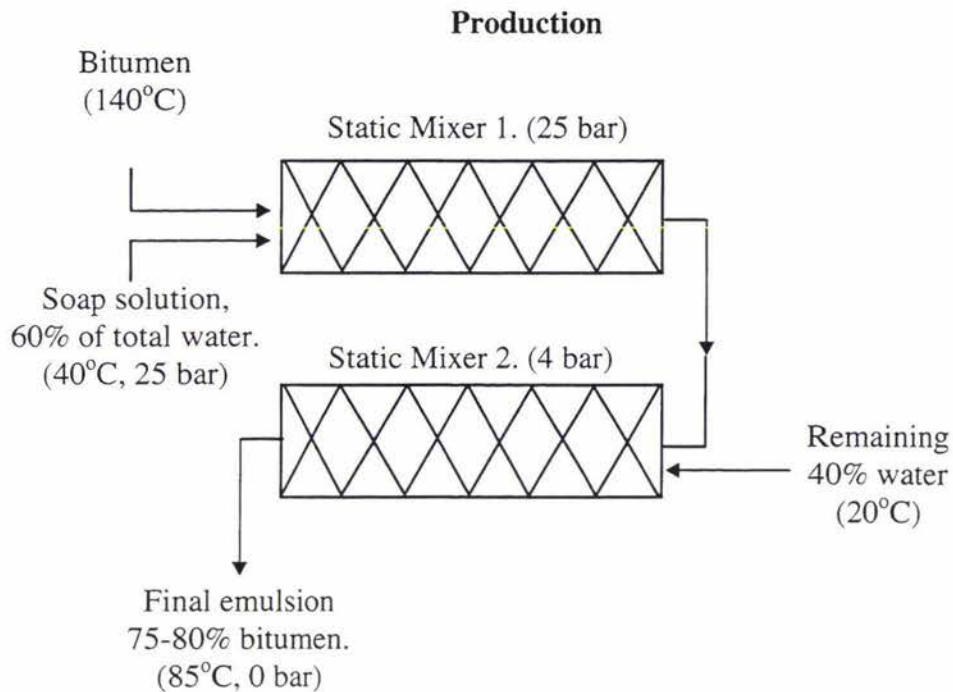
3.4.4 Production of High Bitumen Content Emulsions

A number of production methods have been developed to successfully produce stable high bitumen content emulsions. These include a multi-stage milling process, developed by Esso in France (Marchal, 1991); a stabilisation system using undisclosed additives developed by another French company, SCR (Duval and Beranger, 1989); and thirdly creating a bimodal response in the particle size distribution to increase bitumen packing ability (Booth et al., 1993).

3.4.4.1 Multi-stage milling

This method uses a number of pressurised batch or static mixers arranged in series to blend the various components of the emulsion. Figure 3-5. illustrates the process. Firstly bitumen (140 °C, 25 bar) is mixed with a soap solution (40°C, 25 bar) containing 60% of the total water in the first mixer. The increased pressure prevents vaporisation of the water and allows a fine particle size to be obtained. The mixture is then fed into a second batch mixer at a lower pressure and the remaining 40% of the water at ambient temperature is added. The emulsion emerges from the second mixer at atmospheric pressure with an exit temperature between 80-90°C. Emulsions resulting from this process have a bitumen content in the order of 75-80%.

Figure 3-5 Multi-stage milling Process For High Bitumen Content Emulsion



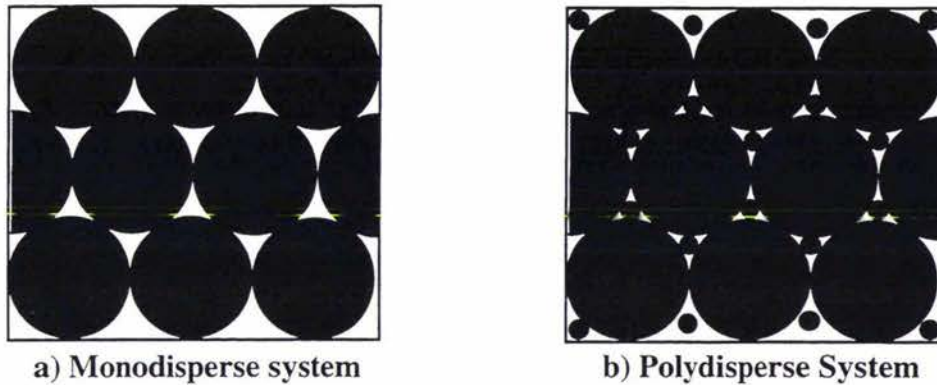
3.4.4.2 Stabilisation system

An emulsion stabilisation system created by SCR is based on the careful selection of an emulsifier system combined with the addition of prepared additives (Duval & Beranger, 1989). The exact nature of additives is proprietary information and has not been disclosed. Standard components of hydrochloric acid, emulsifier and calcium chloride are included and the colloid mill is operated under pressure with a heat exchanger. Few details have been disclosed on the exact nature of the stabilisation mechanism.

3.4.4.3 Bimodal particle size response

The amount of bitumen that can be held in solution is limited by the space between particles and the ability for bitumen particles to be packed close together. Packing theory states that if all particles are spherical and of the same size a maximum packing efficiency of 74% can be achieved (Lissant, 1974). To achieve a higher packing ability a particle size distribution with a range of particle sizes (polydisperse distribution) needs to be created. Polydispersity increases packing as smaller particles are able to fill voids between larger particles. This is illustrated in Figure 3-6.

Figure 3-6 Polydisperse Packing To Increase Bitumen Content



To achieve the polydisperse packing two distinct particle size distributions need to be created. When these distributions are combined a bimodal particle size distribution is achieved. Achieving the correct ratio of small particles to large particles is the key to maximising particle movement and minimising viscosity.

From a production point of view the bimodal response can be created in two ways. In the first method a standard 60% bitumen emulsion is created and this emulsion is co-milled into a second emulsion, further refining a proportion of the bitumen particles. A final emulsion with increased packing and a bitumen content of 80% is obtained. This is a batch type process.

The second and more efficient way to create the bimodal response is to introduce a recycle loop into the process. A proportion of emulsion is taken and re-milled to create the second finer distribution. This is a continuous process without the need for a pre-emulsion. Both systems were patented by Emoleum Australia LTD. and detailed by Holleran, (1993).

3.4.5 Discussion

The appropriateness of the various methods were evaluated against the current technology and capability of the company. The multistage milling process requires completely different mixing technology than the company currently possesses. Purchasing of new emulsifying plant and equipment was outside the scope of this study. This ruled out the use of this type of process in the project. Few technical details were disclosed on the stabilisation system providing little insight into possible development

techniques. The principles underlying the bimodal response and polydispersity along with the careful selection of additives and emulsifier systems contributed the greatest to the development work. Emulsions are rarely monodisperse and it is therefore possible to increase the packing density slightly beyond 74% without causing instability (Chirinos, 1985).

Based on the information reviewed, a bitumen content of 75% could be achieved with current technology through creating a polydisperse particle size. Achieving a bitumen content greater than this without breaking patent laws would be difficult.

Achieving a 75% binder content would also fulfil new New Zealand bitumen emulsion specifications (Transit, 1996). Specifications are in the process of being redrafted with the inclusion of a new category for high bitumen content emulsions (binder contents of 75% and greater). Producing an emulsion with a bitumen content of 75% would provide conformance to this category when the final specifications are introduced.

3.5 EMULSION PROPERTIES

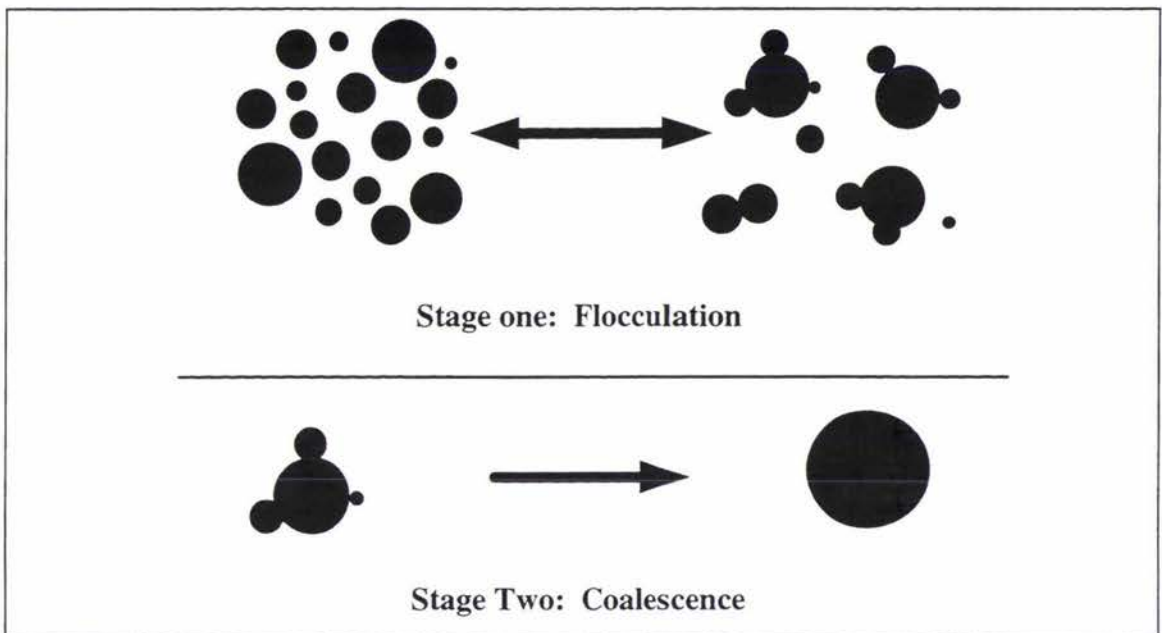
Good control during processing assists in the production of an emulsion with a desired set of emulsion properties. Ideal emulsion properties will depend on the end-use of the emulsion. An emulsion used in chip sealing requires different properties compared to emulsion used in mixes for patching pot-holes. Important emulsion properties include break, cure, cohesion, adhesion, storage stability and handling and spraying ability.

3.5.1 Break

Breaking is the process by which the emulsion is destabilised and changes from its dispersed state back to its separate phases. Once applied to the road the emulsion begins to interact with aggregates and water begins to evaporate off which destabilises the system and causes the emulsion to break. This process is necessary to separate the aqueous phase from bitumen particles in the emulsion to leave the bituminous layer which provides aggregate binding and pavement waterproofing.

Breaking is a two stage process: firstly flocculation occurs and then coalescence. Flocculation is the agglomeration of a number of particles into groups, this stage is reversible with particles separating again under gentle agitation. The second stage of the breaking process involves the irreversible merging of flocculated clusters into larger particles, these large particles are no longer able to be held in solution and settle out (Holleran, 1993). Figure 3-7 illustrates the two stage process.

Figure 3-7 Emulsion Breaking Process: Flocculation and Coalescence



When this process is repeated throughout the entire emulsion the emulsion is said to have broken. The breaking of bitumen emulsions is visually characterised by a colour change from brown to black. An ideal emulsion will not coalesce in storage but when applied to the pavement it will remain unbroken for long enough to allow good chip wetting and then break quickly to provide a strong bond between binder and aggregate.

3.5.2 Cure

Curing is to be distinguished from breaking. Breaking is the initial process where the emulsion is destabilised leaving the separate phases whereas curing is the full hardening of the bitumen layer. These processes are linked but unique, generally a rapid breaking emulsion will be rapid curing. Cure rate is generally an evaporative process which is highly dependant on the following factors (Redelius & Stewart 1992):

- Pavement temperature
- Air temperature
- Humidity
- Wind
- Rain
- Breaking rate
- Emulsifier type
- Aggregate type

Fast cure is important to allow traffic back onto the newly laid surface without damaging the seal.

3.5.3 Adhesion

Adhesion is defined by Chambers, (1991) as: “Intermolecular forces which hold matter together, particularly closely contiguous surfaces of neighbouring media, e.g., liquid in contact with a solid”. In relation to the current work, adhesion is the bonding force which secures the aggregate to the binder; the magnitude of these forces are important for good chip retention. There are two types of adhesion: active and passive (Schevenin, 1994). Active adhesion gives immediate bonding between the aggregate and binder; this type of adhesion is very strong for cationic rapid setting emulsions due to the strong ionic interaction between emulsifier and aggregate. Passive adhesion is the bonding force created by weak intermolecular charges which provide final adherence between aggregate and binder. Passive adhesion take a longer time to impart strength compared to active adhesion.

Active and passive adhesion are influenced by a number of factors which include: aggregate type, bitumen acidity and emulsifier type (Holleran, 1995).

Aggregates. Aggregates are a composition of many minerals, some having greater net charge than others. Aggregates with high silica content such as Greywacke and Quartzite have greater net negative charge than Dolomite and Limestone and therefore interact more strongly with cationic emulsifiers to give greater adhesive strength.

Bitumen acidity. Bitumen is generally acidic which means it has a greater affinity for more basic aggregates. Granite and Basalt are more basic than Greywacke and river gravel therefore adhesion is greater in these types of chip. Variation in bitumen acidity may also correspond to variation in adhesion levels.

Emulsifier type. As previously mentioned cationic emulsifiers have a greater interaction

with negative aggregates than anionic emulsifiers. Simple adhesion tests can be carried out to establish the compatibility of various aggregates with different emulsifiers

Other influences. The use of excessively dirty chip will also reduce adhesion, as the formation of bonds between the binder and stone are weakened by dust particles. Holleran, (1989) also showed that the formation of a skin or “cheesy” layer in cool, windy conditions reduced the emulsion’s wetting ability preventing good adhesion.

3.5.4 Cohesion

Cohesion is defined by Chambers, (1991) as: “Attraction forces by which particles are held together to form a body”. Relating this to bitumen, cohesion is the attractive forces between bitumen constituents which hold the material itself together. A binder with high cohesion is generally able to withstand greater applied forces. Harder grades of bitumen have higher cohesion while addition of cutters reduces cohesive strength.

Cohesive failure is more common with emulsions than cutback bitumen due to the time required to evaporate water from the system to achieve full cure and high internal strength. Allowing traffic onto the surface too early will result in cohesive failure.

3.5.5 Storage Stability

An emulsion must be stable so it can be stored if required without separation or coalescence. If particles are able to settle they begin to flocculate and coalesce into larger particles which are unable to be held in suspension. To create an emulsion which is stable during storage, particle settling needs to be minimised. Particle settling is governed by Stokes Law (SFERB, 1991); by considering the variables in Stokes equation an understanding of how to control particle settling can be gained.

Stokes Law:
$$V = \frac{2 \cdot g \cdot r^2 (\rho_1 - \rho_2)}{9 \cdot \eta}$$

V = Particle settling velocity.

g = Acceleration due to gravity.

r = Particle radius.

ρ_1 = Density of dispersed (Bitumen) phase.

ρ_2 = Density of aqueous (Soap) phase.

η = Viscosity of aqueous phase.

The following measures minimise settling velocity (V) and maximise storage stability.

- Particle size during production to be as small as possible. Small r^2 .
- Lower the difference in densities between dispersed and aqueous phases. Softer and less dense grades of bitumen tend to make more stable emulsions.
- Increased viscosity of aqueous phase by addition of salts and stabilisers.

Other factors which should be considered when storing bitumen emulsions include:

- Avoid heating above 100°C thereby boiling off water and causing the emulsion to break.
- Avoid freezing which also causes the emulsion to break.
- Maintain gentle circulation to ensure a uniform consistency.

3.5.6 Viscosity

Viscosity is defined as a fluid's resistance to flow under shear (SFERB, 1991). This is a critical parameter in emulsion application and handling. If an emulsion is not viscous enough when sprayed it will run off the road and end up in the gutter. If it is too viscous difficulties are encountered in obtaining a uniform spray distribution and streaking occurs. Pumping and handling also become difficult. Viscosity is controlled by a number of factors (Whiteoak, 1990):

- | | |
|----------------------------------|--------------------------|
| • Particle size and distribution | • Salt content |
| • Emulsifier type and content | • Bitumen content |
| • Solvent or cutter content | • Flow-rate through mill |

Emulsion viscosity also changes with temperature and shear rate. Bitumen emulsion viscosity reduces with increased shear; this thixotropic nature plays a key part in the product's application. When emulsion is sprayed through small nozzles, high shear rates are created causing the emulsion to thin, allowing a more even application. Once the shearing forces are removed and the emulsion hits the ground the viscosity increases due to its thixotropic nature. This means thicker layers of emulsion can be built up without the concern of run-off. With thicker emulsion layers larger aggregates can be held.

3.5.7 Summary of properties

A bitumen emulsion provides the binding material to which aggregate can adhere producing a skid resistant and waterproof surface. An ideal emulsion will have the correct viscosity to be evenly applied. When applied it will break at the appropriate speed yet allow chips to be fully wetted. It will then have sufficient strength in the form of adhesive and cohesive forces to initially retain the stones. Cure will then be rapid to allow traffic to resume as soon as possible to provide a long lasting road surface. Finally the emulsion should be stable enough to be stored for the correct length of time. These are the desired characteristics of an emulsion for use in chip sealing work.

3.6 EMULSION APPLICATION

Production of a good emulsion with the correct properties is only part of producing a high quality chip-seal. Just as essential is the correct application of the emulsion to the road (Holleran, 1997). The basic operations in application include:

- Spraying of emulsion.
- Spreading of chips over emulsion.
- Rolling to re-orientate and press chips into the binder.
- Brooming to remove excess chips.

3.6.1 Spraying

A spray tanker is used to apply an exact quantity of bitumen emulsion in a uniform way. The sprayer consists of a temperature controlled tank, pumps and a spray bar located at the truck's rear fitted with a number of nozzles. See Figure 3-8.

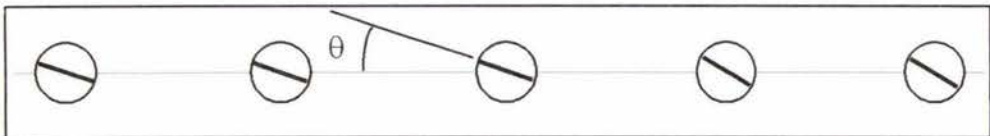
Two important variables related to the spray bar need to be controlled. These are nozzle angle and spray bar height (Asphalt Institute, 1992). Nozzle type also influences spraying characteristics. However this was a fixed constraint for the project with V-slotted jet nozzles being used.

Figure 3-8 Spray-tanker for Bitumen Emulsion Application



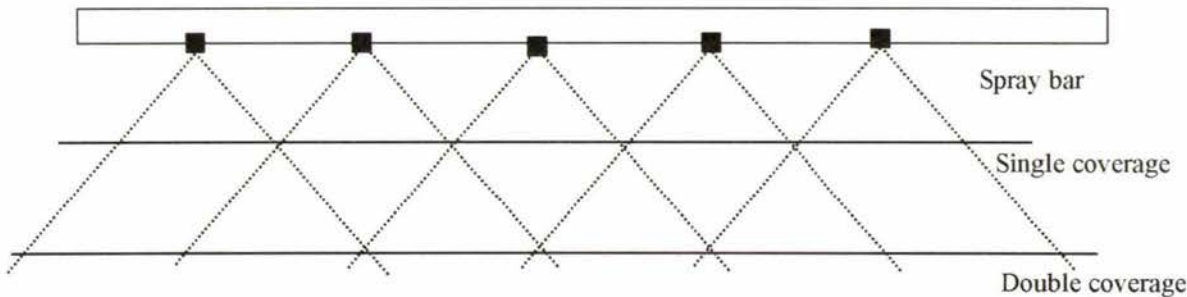
Nozzle Angle. Nozzle angle needs to be set so that the individual spray fans do not interfere with each other to produce streaking. Figure 3-9 illustrates nozzle angle. Generally the nozzle angle is set between 15-30 °.

Figure 3-9 Spray-nozzle Angle



Spray bar height. Correct height from the spray bar to the ground is essential for uniform coverage. If the spray bar is too high the spray pattern is prone to interference by wind; if it is too low there is insufficient coverage. As spray bar height increases the spray bands overlap to a greater degree. This is illustrated in Figure 3-10. The best coverage is achieved with a double overlap.

Figure 3-10 Overlap of Spray Bands With Height Increase



Spray temperature is a further factor to be considered in spraying. The temperature at which the emulsion is sprayed will affect the emulsion's viscosity. A high spray temperature reduces viscosity and can cause the emulsion to 'run-off' the road. A low spray temperature can cause the emulsion to increase in viscosity which is then unable to be dispersed easily resulting in an uneven distribution. A temperature which balances these constraints must be found. This is usually around 85°C for high bitumen content emulsions.

3.6.2 Chip Spreading

The chip spreader provides a uniform cover of aggregates over the sprayed emulsion. A number of systems are used including, tailgate vane spreaders, mechanical spreaders and tailgate hopper spreaders (Asphalt Institute, 1992). The most common being the tailgate hopper spreaders, illustrated in Figure 3-11. Tip trucks are mounted with hoppers on the tailgate, the rate of aggregate application being controlled by a feed roller which is activated by small wheels driven by the truck's wheels. The spreader is driven in reverse so that a cover of aggregate is laid protecting the truck's tyres from picking up the tacky emulsion.

Figure 3-11 Tailgate-hopper Chip Spreader



3.6.3 Rolling

Rolling follows the application of aggregate. This presses the stone into the emulsion and re-orientates chips so that they are in the most stable position to form a strong matrix. Rolling also increases the rate of cure as the mechanical action increases the breaking of the emulsion. Two types of rollers exist: steel wheeled rollers and pneumatic tyred rollers. Pneumatic tyred rollers produce the best results for chip sealing. The softer tyres do not crush the chips as do steel wheeled rollers. Figure 3-12 shows a pneumatic tyred roller.

Figure 3-12 Pneumatic Tyred Roller



3.6.4 Brooming

The final operation in the construction of the chip seal is brooming of excess chips from the surface to avoid stones becoming dangerous projectiles and damaging windscreens. A rotary broom is pictured in Figure 3-13.

Figure 3-13. Rotary Broom



3.7 CONCLUSION

The review of literature provides an understanding of the science and technology related to bitumen emulsion production and application. Bitumen is emulsified primarily to improve its flow characteristics. The bitumen emulsion is stabilised through the adsorption of charged emulsifier molecules onto finely dispersed bitumen particles. The charge repulsion between particles keeps them separated to stabilise the emulsion. Cationic emulsifiers provide greater adhesion characteristics and reduced cure times due to the stronger ionic interaction with aggregates and are more appropriate for use in high bitumen content emulsions. Bitumen composition was examined and it was found that the high asphaltene and salt contents make bitumen used in New Zealand more difficult to emulsify. Various high bitumen content emulsion processing techniques were investigated. Optimisation of the formulation and particle size distribution was the most appropriate means to achieve increased packing and a bitumen content of 75%.

Important properties to control in a spray seal emulsion included: viscosity, break rate, cure rate, cohesion, adhesion and storage stability. Correct viscosity is important for even spraying without run-off. Viscosity is largely controlled by choice of emulsifier, particle size distribution and exit temperature. Breaking is the destabilisation of the emulsion, this is necessary to liberate the emulsified bitumen on application. Curing is largely an evaporative process and is required to provide cohesive strength to the binder to enable the surface to be driven on without damage. Cohesive strength is the internal strength of the binder whereas adhesion is the bonding strength between the binder and aggregate, both are important in forming a strong bitumen/aggregate matrix. Storage stability is increased by reducing particle settling.

Understanding the emulsification principles, production methods, raw materials, desired properties and application methods related to bitumen emulsions provided a technical basis to undertake informed product design.

4. DEVELOPMENT METHODOLOGIES

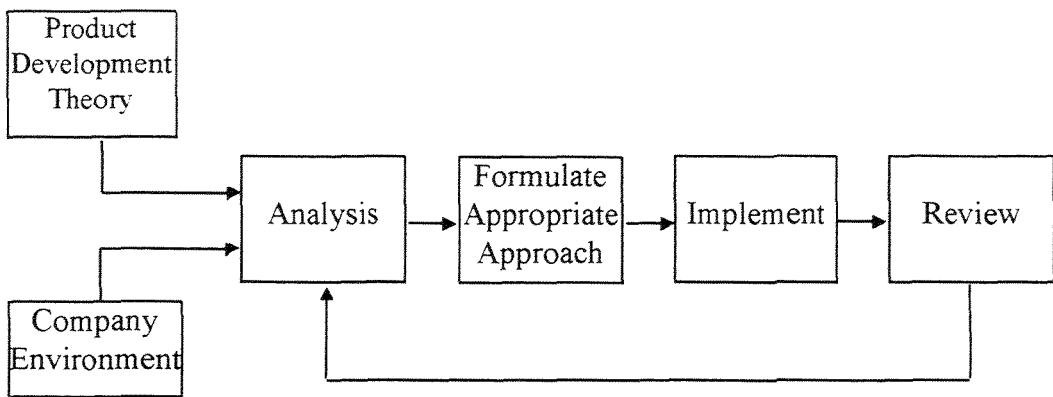
The general product development processes and principles reviewed in Chapter 2 are used to develop a tailored product development approach for the project and for future use by Higgins. An appropriate formulation design methodology is also discussed along with the raw materials, processing and testing methods used. Three aspects to methodology are therefore to be addressed:

- The research method used to create the tailored product development approach (Section 4.1).
- A description of the tailored product development approach (Section 4.2).
- The methodology used to undertake formulation development (Section 4.3).

4.1 RESEARCH METHOD

It was the researcher's task to analyse the product development theory reviewed in Chapter 2 and relate it to the people, products, structure and conditions of Higgins to design the most suitable approach to product development. An approach was developed, reviewed and necessary adjustments made. Figure 4-1 illustrates the research method used to create the product development approach suitable to Higgins.

Figure 4-1 Research Method Used to Develop Higgins Product Development Process



4.1.1 Environmental Factors

The theoretical product development approaches reviewed in Chapter 2 were unable to take into account the uniqueness of individual company environments. By analysing the

company environment the most appropriate approach for product development was found and adapted. The environmental factors outlined in Chapter 1 which affected the required product development approach included:

- Industrial marketing environment.
- Processed nature of Higgins products.
- Newly established development centre with a small development staff.
- Company infrastructure under development.
- Management structure.

4.1.2 Analysis

Analysis of the company in relation to the literature revealed Cooper's Stage-Gate process to be the most appropriate process to Higgins Contractors for the following reasons:

- Suitable to industrial and formulated products.
- Can be implemented in small or large companies.
- Incorporated most of the best practise into one process.
- Simple and clear to understand.
- Easy to implement.
- Other aspects of best product development practise could be easily incorporated into Cooper's Stage-Gate process.

4.1.3 Formulate Product Development Approach

While formulating the product development approach, weekly meetings were set up with the Group Technical Manager and the other Product Development Technologist to discuss the proposed approach. This consultation meant the most workable process would be implemented at Higgins as well as incorporating all the stages required for good product development. The creation of the product development approach was carried out in a number of steps.

- Establish the broad product development stages required.
- Establish the smaller phases required within each broad stage.
- Outline the outputs, documentation and decisions required in each phase and stage.
- Build up template documentation for the required outputs in each phase and stage.

4.2 HIGGINS PRODUCT DEVELOPMENT APPROACH

The first task to develop a suitable product development process for Higgins was to establish the broad product development stages required. Six stages were identified:

1. Project start
2. Pre-development
3. Laboratory development
4. Mid-scale development
5. Full-scale development
6. Commercialisation

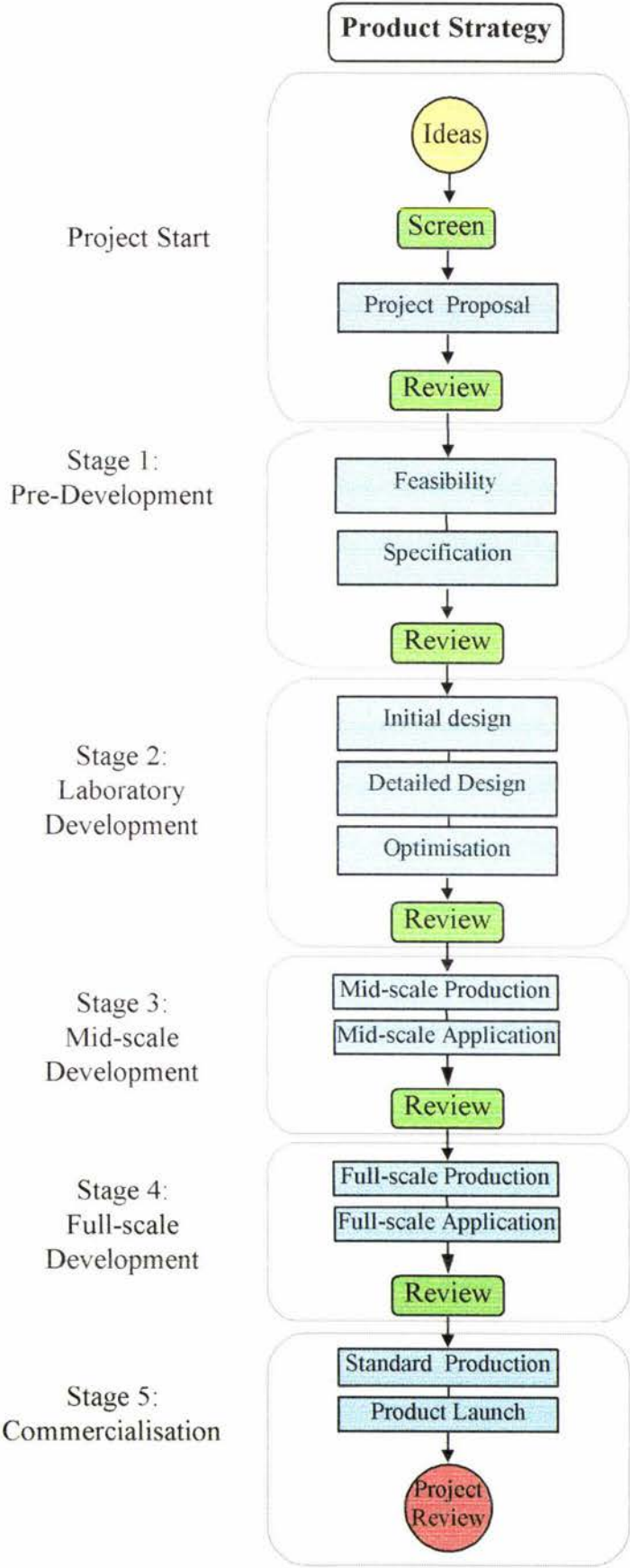
Each of the broad stages consists of a number of smaller phases. Figure 4-2 shows with more detail the Higgins product development process outlining the six broad stages and smaller phases. Stages are marked by the dotted areas in Figure 4-2 while the smaller phases are identified in the blue boxes.

Each stage in the product development process requires certain inputs, activities, outputs and decisions to be made (Rosenau, 1990; Earle, 1997). An outline of these requirements for each stage provides an increased level of detail into product development at Higgins.

4.2.1 Product Strategy

Product strategy is not a stage in itself, but is strategic policy set prior to any development activity, thus it is outside the Project start stage in Figure 4-2. A product strategy is created to guide the overall direction of all product development activity. Top-level management need to clarify in a product strategy document, the scope and direction of product development. Potential development projects can then be assessed as to whether they fit the strategic alignment of the company. Revision of the product strategy is required periodically to ensure the product strategy reflects any changes in the direction of the entire company.

Figure 4-2 Higgins Product Development Process.



4.2.1 Product Strategy continued.

Goal

- To develop strategic policy to guide all product development activity.

Required Inputs

- Strategic company policy, mission statement, company objectives.

Typical activities

- Strategic policy meeting.
- Policy reviews.

Documentation and Outputs

- Product strategy document.

Decisions

- Scope and direction of product development.

4.2.2 Project Start

The Project Start stage consists of two phases: Idea collection and Screening and Project Proposal. Ideas are gathered from various sources including conferences, contractors and literature. The ideas are documented using concept capture sheets (detailed in Chapter 5). These capture sheets are collated, reviewed and screened quarterly to identify the greatest potential projects. Once an idea has passed screening, basic information is collected on the market, technical and financial aspects of the product. This information and the projects aims and objectives are summarised in a project proposal for further evaluation by management.

4.2.2.1 Idea Capture and Screening

Goal:

- To identify greatest potential ideas for further investigation.

Required Inputs

- Product strategy objectives
- Concept capture sheets

Typical activities

- Concept review meeting
- Idea screening

Decisions

- Go/No-go. Selection of high priority projects consistent with product strategy.
- Assigning of project responsibility.

4.2.2.2 Project Proposal

Goal

- To summarise and clarify the development opportunity.
- To outline project aims/objectives and estimate required resources.

Typical activities

- Literature Search.
- Patent search.
- Product search.
- Initial market research.
- Preliminary technological assessment.
- Preliminary financial assessment.
- Preliminary production and application assessment.

Documentation and Outputs

- Project Proposal

Decisions

- Go/No-go. Assess project proposal based on initial investigation.

4.2.3 Stage 1. Pre-Development

The Pre-development stage is entered once the project proposal generated in the Project Start stage has been approved. The Pre-development stage consists of Feasibility and Specification phases. The purpose of Feasibility and Specification phases is to investigate the proposed idea in-depth to establish clear viability and specific means and intent of development. Feasibility and Specification phases can either be carried out consecutively or simultaneously depending on the size of the project. Larger more complex projects will require two distinct phases; feasibility followed by specification. Smaller and simpler projects can combine the two phases into one.

4.2.3.1 Feasibility

Goals

- To prove feasibility of key development areas to reduce development risk.
- To gain in-depth information of intended technology and market.

Required Inputs

- Project Brief.
- Approval to further investigate the product concept.

Typical Activities

- Detailed technological assessment.
- Detailed financial assessment.
- Detailed production and application assessment.
- Bench chemistry- gather starting formulations.

Documentation & Outputs

- Feasibility report- Detailing exact technologies, market and methods to be used.
- Starting formulations of product.

Decisions

- Go/No-go. Assess viability with respect to technology, cost, time and return.
- Authorise development of design specifications.

4.2.3.2 Specification

Goals

- To quantitatively specify performance levels to be met during development.
- To specify intended marketing strategy, product positioning and needs of market.

Required Inputs

- Approval to specify feasible product.

Typical Activities

- Competitive analysis and bench marking.
- Review relevant standards and industry specifications.
- Determine and rank key product attributes.
- Establish testing regime.
- Set performance targets with acceptable limits.
- Market research.
- Formulate marketing strategy, objective and product positioning.

Documentation & Outputs

- Marketing specification outlining market issues.
- Technical design specification- Quantitative description of development intent.

Decisions

- Go/No-go. Assess specifications for correct development direction. Approval indicates authorisation for laboratory development.

4.2.4 Stage 2. Laboratory Development

Laboratory development is where the planned product set out in the technical design specification is realised. Within this lengthy and critical stage the process is divided into three phases: initial design, detailed design and optimisation. Initial design is where many concepts or formulations are evaluated and a preferred starting concept is selected. Detailed design explores the concept to identify a preferred design within the selected concept. Optimisation takes the preferred design and refines it into a final laboratory formulation.

Goals

- To produce a product in the laboratory setting which meets performance levels of the design specification.
- To identification of a preferred starting concept.
- To exploration of the starting concept to identify a preferred design.
- To optimise the preferred design to produce a final laboratory developed product.

Required Inputs

- Initial concepts/formulations
- Technical design specification

Typical activities

- Screening-emulsifiers, materials, process variables and formulations.
- Initial design- Identification of preferred starting formulation.
- Detailed product design- Detailed exploration of preferred formulation.
- Optimisation- Further exploration to produce a final product.
- Refine technical design specification.
- Yard trials.
- Estimate product tolerances.
- Intellectual property considerations.

Documentation and Outputs

- Laboratory development plan.
- Results and analysis of screening, development and optimisation.
- Production specification and other production documentation.
- Marketing plan with forecast of discounted cashflows.
- Fully optimised laboratory formulation.

Decisions

- Go/No-Go. Performance has been demonstrated by laboratory scale product to justify further development.
- Approval to move to mid-scale development.

4.2.5 Stage 3. Mid-Scale Development

Making incremental scale-up steps reduces the size and risk of potential failure. Before moving to full-scale production and application performance is to be proven on a medium scale. This is less costly than full-scale development and provides useful information of variables unable to be evaluated in the laboratory setting.

Goals

- To quantify production and application variables unable to be simulated in laboratory setting.
- To gain a clearer understanding of final product performance and cost.

Required Inputs

- Optimised laboratory product.
- Mid-scale production specification.

Typical activities

- Mid-scale production and application.
- Evaluate mid-scale product performance.
- Build correlations between mid-scale trials and laboratory work.
- Reformulation.
- Refine technical specifications.
- Begin production of promotional material.
- Estimate product cost.

Documentation and Outputs

- Mid-scale development plan.
- Mid-scale trial evaluation report.
- Refined technical specification.
- Successful completion of mid-scale trial.

Decisions

- Go/No-go. Satisfactory product performance demonstrated on a mid-scale level.

4.2.6 Stage 4. Full-Scale Development

Information gained during mid-scale development trials is used to move into full-scale trials to assess the actual production, application and performance of the product.

Goals

- To demonstrate an ability to successfully produce and apply product in a full-scale field trial.

Required Inputs

- Refined production specification.
- Refined technical specification.

Typical activities

- Staff training.
- Produce product literature.
- Determine testing regime for field trial.
- Plan and conduct full scale production.
- Evaluate field trial.
- Correlate field trial to laboratory and mid-scale trials.
- Evaluate production variables.
- Establish quality control measures.
- Finalise launch strategy.

Documentation and Outputs

- Full scale field trial evaluation report
- Finalised product specification
- Promotional material
- Launch proposal
- Marketing and tendering resulting in contracts requiring product.

Decisions

- Go/No-go Proven product performance on full-scale.
- Decision to commence standard production.

4.2.7 Stage 5. Commercialisation

Commercialisation consists standard production and product launch phases.

Goal

- To produce consistent quality product for commercial sale

Required Inputs

- Finalised product documentation.
- Marketing and tendering resulting in contracts requiring product.

Typical activities

- Standard production procedure established.
- Standard application procedure established.
- Quality control checks.
- Storage and transportation.
- Product application.
- Customer feed back.
- Project evaluation and review.

Decisions

- Plan production of product and required quantities.
- Necessary product adjustments

Documentation and Outputs

- Project evaluation report.
- Fully developed and launched product.

4.2.8 Template Documentation

Template documentation was then required to be built up to practically implement the developed product development process. This is a large and on-going task but examples of some documentation were produced, this included:

- Project brief
- Concept capture sheets (Appendix 5.1)
- Product design specification (Appendix 6.1)
- Development plans
- Laboratory development reports
- Field trial plan

4.2.9 Discussion on Product Development Process

Changes to the classical product development processes reviewed in Chapter 2 to produce the Higgins product development process illustrated in Figure 4-2 and described in sections 4.2.1-4.2.9 were made based on the needs and focus of the company; a discussion of the tailoring process follows.

A 'Product strategy' step was added to the standard Cooper process to emphasise the need for guiding direction in product development. Although Cooper recognises this step it is not presented in his process. This incorporates best practise from the processes presented in Chapter 2 by Earle, (1997) and Hass (1989).

Product development stages were named using terminology the company was more familiar with and which better reflected the nature of work. Cooper's 'Initial investigation' was named 'Project Proposal' while the 'Detailed investigation' was named 'Feasibility and Specification'.

The laboratory development stage was divided into three phases: initial design, detailed design and optimisation. This was to exert more control over the design process and incorporated the principles from the BS-7000 product development process.

The Higgins product development process expanded Cooper's testing and validation stage into two distinct stages, mid-scale and full-scale development. The processed nature of Higgins products meant more emphasis on scaling-up developments was required; breaking scale-up and validation into two distinct stages reflected this emphasis.

4.2.10 Discussion and Recommendations

Having established a product development process relevant to the current Higgins environment, consideration was also given to the future of product development at Higgins. A discussion of the current environment at Higgins highlights a number of recommended changes to the current product development approach.

Currently product development at Higgins is largely a technical activity with the input from other functions called on informally and as required by product development staff. This approach is dependant on the individual product developer's foresight and ability to gather the input of other functions rather than the product development process itself. Greater formalised input from other functions throughout the process will allow a more accurate estimation of problems by those with a greater understanding of specific areas. The input from other functions can be formalised with a team approach. A core team comprising of key functions such as contracting and production could be assigned when initiating a project. These different functions could formally advise and input into the project through regular progress meetings. This would better facilitate the flow of communication between functions to incorporate the requirements of production and contracting personnel. It would also serve to integrate product development to a greater degree into the company.

As there is currently only one development staff member an elaborate project review procedure is inappropriate. Review points are currently assessed by the Group Technical Manager who reports progress quarterly and annually to company directors. This is an appropriate form of management at the current time but may need re-evaluation with further growth. The use of project teams could be examined in future with progress being reviewed by a review committee, similar to the P.A.C.E approach reviewed in Chapter 2.

Currently there is a lack of specialised personnel to carry out marketing and promotion however plans are in place to strengthen this area. Marketing of newly developed products will therefore need to be incorporated into the product development effort in future.

Currently there is little formal assessment of the market or contact with customers in developing products. Assessment of the market and discussions with customers will assist in establishing product opportunities which have a genuine market demand. A greater market and customer focus may be achieved with the assistance of marketing personnel. It is recommended that the marketing aspects of product development be delegated to marketing personnel, these activities could include:

- Conduct market research with customers to determine product opportunities and requirements.
- Market feasibility analysis.
- Develop launch and promotional plans.
- Conduct financial analysis of market.
- Gain customer feedback on product performance.

Project costs and the assessment of a project's financial return are currently not formally evaluated. Increasing the assessment of the costs related to projects will provide greater accountability of development funds. While increasing the level of financial assessment will establish whether the revenue to be generated by a product will justify its development and aid in screening projects. Although development projects are currently small, establishing budgets and systems for project costings readies the company for larger developments without misappropriation of funds.

As product development is a relatively new function in the company, development work is generally product maintenance, line extensions or copying competitor's products. These type of products are necessary at this stage of product development at Higgins to develop a competitive product range. However numerous studies have shown that more innovative products enjoy greater market and financial success (for example; Douglas et al, 1978; Cooper, 1993).

Goals are recommended to be set at Higgins to begin to move towards more innovative product development. To provide products which are not available to the local market to obtain greater financial and market success; recommended goals include:

- Establish a systematic product development approach.
- Become proficient with the product development approach.
- Establish a competitive product range.
- Establish product innovation leadership in local market.

4.2.11 Conclusion

Analysis of the various product development processes in the light of company environmental factors revealed Cooper's product development process to be the most appropriate base process to begin tailoring a product development process for Higgins. Various adaptations were made to incorporate aspects of a number of other systems and allow an appropriate process to be tailored. A process was created and included the following six stages:

1. Project start
2. Pre-development
3. Laboratory development
4. Mid-scale development
5. Full-scale development
6. Commercialisation

This tailored product development process was used by the project to guide development of the high bitumen content emulsion and provides a template for future projects. Details of the required activities and outputs to be produced from each stage were generated. Details of the tailored product development systems developed within each of the stages from project start through to full-scale development are covered in Chapters 5, 6, 7 and 8.

The developed product development process considered the current environment and recommendations for future change to product development at Higgins were made. Recommendations included: a greater team approach, increased marketing effort, focus on market needs, increased financial assessment and obtaining proficiency in product development. These measures are recommended to achieve a competitive product range and begin leading innovation in the local market.

4.3 FORMULATION DEVELOPMENT METHODOLOGY

Formulation development carried out in the Laboratory Development stage of product development at Higgins is a critical stage where the product characteristics set out in the technical design specification are realised. Methods used in this critical stage relating to the formulation of the high bitumen content emulsion product included:

- Overall design methodology.
- Raw materials and processing conditions.
- Test methods used in product evaluation.
- Detailed design methodology.

4.3.1 Overall Development Methodology

Three phases were undertaken during laboratory development:

1) Initial design

- Process capability and test repeatability study.
- Raw material screening.
- Investigation of processing variables.
- Establish tests to be conducted.

2) Detailed design

- Detailed exploration of formulation using experimental design techniques.

3) Optimising

- Isolating an optimum formulation area and determining the best possible set of product characteristics within this area.

A similar development method was used by Uaphithak (1991) using experimental design techniques in the optimisation of a hand cream emulsion. Kerr (1995b) cites a similar template method for formulated food products. Moskowitz (1983) provided further evidence of the appropriateness of using factorial experimental designs in formulated products.

4.3.2 Initial Design

A focusing of the raw materials, processing conditions and testing was achieved in the initial design phase. The goal of this first phase in Laboratory Development was to

identify a preferred concept for continued development. The initial design phase consisted of three steps:

1. Establish testing to be conducted.
2. Screen raw materials.
3. Establish processing variables.

4.3.3 Test Methods

The first step in the initial design phase was to establish the set of tests to be used to evaluate the developmental emulsions. The testing regime for the study included the following seven tests.

- pH of final emulsion
- Residue
- Viscosity
- Settlement
- Break index
- Adhesion
- Cohesion

pH and residue were tested as production checks and not for formulation optimisation. The five remaining tests summarised the main response variables to be optimised during the detailed design phase. Emulsions were stored for 24 hours after production in a 60°C oven before conducting the various tests, unless otherwise stated.

4.3.3.1 pH

Emulsion was stored at 60°C for 24 hours after production to allow an equilibrium pH to be achieved. pH was then measured at 60°C using an Orion 210A pH meter and an Orion 91165BN pH electrode coupled with a Mettler Toledo ATC probe.

4.3.3.2 Residue by Evaporation

Residual binder was tested to monitor the quality of all emulsions to verify that binder content was in fact between 75-76%. Residual binder content was tested immediately after production using a Mettler LP16 infra-red moisture meter. A 10 g sample of emulsion was exposed to a 120°C heat until all water evaporated and the weight remained constant for a 30 second interval.

4.3.3.3 Viscosity

Viscosity was measured immediately after production using a Brookfield RVT Syncro-Lectric dial viscometer. Measurements were taken in compliance with TNZ M/1:1996 specifications. The following sampling conditions were used:

- Emulsion temperature $70^{\circ}\text{C} \pm 2^{\circ}\text{C}$.
- Spindle #1 (Appropriate for low viscosity measurement.)
- Spindle speeds of 5, 10 and 20 rpm were used.
- The spindle was allowed to spin for 1 minute before a reading was taken.

4.3.3.4 Settlement

Settlement is an indication of storage stability and was tested in accordance to the standard ASTM D244:45-50 method. A 500 ml. sample of emulsion was left to settle in a glass cylinder over a 24 hour period and the difference between the residual bitumen of the top 50 mls. and the bottom 50 mls. determined. To be judged as stable no more than 1% difference in residual bitumen is permissible per day. High binder contents increased viscosity and made sampling emulsion more difficult compared to lower content emulsions.

4.3.3.5 Break Index

Break index provides an indication of the breaking speed and stability of an emulsion. The break index test was based on a French test method NF T66-017. In this test small amounts of sand were mixed into 20 g of emulsion until the mixture turned into a solid lump, at this point the emulsion was fully broken. The break index is a ratio of the mass of emulsion to sand required to fully break the emulsion. Break index is generally to be minimised for high bitumen content emulsions.

4.3.3.6 Adhesion

Adhesion was tested 24 hours after production according to the French test method NFT 66-018. 100g of aggregate was placed in 150g of emulsion at 60°C and left to stand for 5 minutes. Excess emulsion was decanted off and the aggregate gently washed till the

water ran clear. Aggregate was then judged by eye for a percentage coating. Subjective evaluation of the coating reduced the accuracy of the test.

4.3.3.7 Cohesion

There was no established test method within Higgins to assess cohesion. Therefore it was necessary to find an appropriate method. A number of test methods were reviewed including the spin test method (Redelius & Stewart, 1992), cure indexing (Booth et al., 1993) and the frosted marble cohesion tester (Benedict, 1992). The test developed by Benedict was selected as it used current testing equipment and was simple to use.

Marbles that have been scoured with carborundum to produce a uniform and slightly rough surface are used in the frosted marble test. Five frosted glass marbles were placed on a metal plate with a 1 L/m^2 coverage of residual binder. Three plates were left to cure, a plate was taken for measurement after 1, 2 and 3 hours to obtain three separate data points for cohesion. Cohesive strength was measured at each cure interval by the average torque required to dislodge the five marbles using a stand mounted torque meter. Cure conditions varied according to ambient laboratory conditions.

4.3.3.8 Process Capability and Test Repeatability Study

Variance of measurements are an important aspect in testing product properties. Determining the variance in testing establishes whether observed changes in product properties are from deliberate changes to inputs, random process variation or variation in the testing methods (Barnes, 1996a). Knowledge of the variance involved in the process and in testing assists in the management of experimental error and provides an indication of the level of confidence attached to test results. Therefore before design work was embarked upon an estimate of testing and process variance was necessary. The investigation was set up in two parts: process capability and test repeatability.

To test the process capability of the laboratory mill, three identical formulations were made to determine the ability of the process to reproduce samples. The three emulsions were then tested for pH, viscosity, break index and residue. Test methods for

settlement, adhesion and cohesion were yet to be established at this stage and could not be included in the study.

Test repeatability was assessed using a single emulsion, the various tests were repeated five times each using the standard emulsion. 95% confidence intervals were calculated on the averages of both process capability and test repeatability data to provide an indication of variation. Table 4-1 shows the percentage variation calculated from the analysis.

Table 4-1. Variation in Laboratory Production Process and Testing Methods

	Process Error ± %	Test error ±%
pH	2	0.2
Residue	0.1	0.5
Break Index	0.7	3
Viscosity	3.1	5.5

In general it was found that variation in the testing was greater than variation observed in the process. All errors were less than 5.5%, which was deemed an acceptable level of variation to identify deliberate changes in formulation properties greater than 5.5%. Variances inherent in the adhesion, cohesion and settlement tests that were not assessed in the study included:

- The subjective evaluation of stone coating for adhesion.
- Uncontrolled cure conditions for cohesion testing and operator dependency.
- Difficulty decanting consistent amounts of thick emulsion during settlement testing.

Large variances in these tests reduced the ability to clearly detect changes in output emulsion properties from deliberate changes in formulation components. Only factors which greatly affected each of the properties could be detected with the large variances inherent in the tests. Discussions with the companies consultant, Glynn Holleran confirmed that most of the tests were only good as indicators of trends rather than accurate assessment methods. To gain a true understanding of emulsion properties rheological characteristics need to be measured. This however requires expensive testing equipment which is outside the budget and scope of the company. Despite these

weaknesses an understanding of how each property was controlled was gained with the tests. Measures to reduce variability in testing would correspondingly increase the accuracy of design work.

4.3.4 Raw Materials Screening

The second step in the initial design phase was to screen raw materials to identify the most appropriate materials for use in further development.

Bitumen

As a project constraint the bitumen type was fixed; Safaniya bitumen with a relatively high penetration value of 180-200 dmm was used. Penetration values are used to grade bitumen in New Zealand. A needle with a constant forces applied to it is allowed to penetrate a 100g sample at 25°C for 5 seconds to give a penetration depth, ie 180-200 dmm. Softer bitumens have higher penetration values. The bitumen used was the most common type used by Higgins as it suits the majority of local climatic conditions and the softer grade made emulsification easier.

Solvent

Kerosene supplied by Shell was the choice of solvent as it was cheaper than other solvents such as turpentine and provided similar wetting ability.

Salt

Gaudong flaked calcium chloride was the salt selected as it provided greater adhesive properties than other salts such as sodium chloride.

Emulsifier

An emulsion's behaviour, particularly viscosity, is generally governed by the choice of emulsifier (James, 1997). The choice of emulsifier is therefore one of the most critical decisions for an emulsion and a detailed description of the emulsifier screening is given.

Initially over twenty potential cationic emulsifiers were collected from suppliers, literature and current laboratory stocks (Refer Appendix 4.1). Two stages of screening were carried out to reduce this number. The first was based on technical data and the second was based on physical testing of produced emulsions.

Criteria for the literature screening included:

- Physical form: Solid emulsifiers were eliminated as they undesirably increase viscosity (James, 1997).
- Supply: Emulsifiers needed to be available in New Zealand.

Application of the screening criteria reduced the list of emulsifiers from twenty to eight:

- | | |
|------------------------------|------------------------|
| • CECA, Dinoram SL | • Witco, AA 60 |
| • CECA, Emulsamine 640 | • Witco, AA 57 |
| • Akzo Nobel, Redicote EM-44 | • Witco, DXP 5357-100 |
| • Akzo Nobel, Redicote N39L | • QuimiKao, Asfier 123 |

The second means of screening was to make a 75% emulsion using each of these emulsifiers. The physical properties of the emulsions were then tested; viscosity was the critical parameter assessed. Dinoram SL produced by CECA is a medium setting cationic fatty diamine emulsifier and was the emulsifier selected after emulsifier screening. Emulsifier screening results are described in Chapter 7.

Acid

Industrial strength hydrochloric acid (32.5% w/w) was selected as it was the preferred acid recommended by suppliers for the emulsifier identified during emulsifier screening. The acid was also routinely supplied for the production of other emulsions.

4.3.5 Processing Variables

The third step in the initial design phase was to determine key processing variables. Having the correct processing conditions is just as important as having the correct raw materials as emulsion properties are dependant on both.

4.3.5.1 Colloid Mill

Higgins have a custom built laboratory scale Charlotte colloid mill which enables the production of developmental formulations, seen in Figure 4-3. Control of processing conditions such as mill gap, phase temperatures and flow-rates are similar to the production facility located in Napier.

Figure 4-3. Laboratory Scale Colloid Mill.



Mill gap. Preliminary investigations into mill gap found a fine clearance of 0.15 mm (mill setting of 10 holes) was best to create a finer more stable emulsion.

Mill pressure. The laboratory mill was operated at atmospheric pressure with a slight pressure of 2 bar on the inlet line to even out pressure fluctuations in the mill and avoid back-flow of emulsion through the mill. This was similar to the larger mill in Napier.

4.3.5.2 Bitumen Phase Temperature

With high bitumen content emulsions the heat contribution from the bitumen phase is greater than lower content emulsions. This has the effect of increasing exit temperature and undesirably affecting viscosity (Booth et al. 1992). Therefore the temperature of the bitumen was lowered from standard temperatures of 135°C-140°C to 130°C. At 130°C the bitumen still enters the mill at a low enough viscosity to give a good dispersion. The softer grade of 180/200 bitumen used also made the lowering of the bitumen temperature less critical.

4.3.5.3 Soap Phase Temperature

A soap phase temperature was used that would give an exit temperature of around 86°C which was reported by Booth et al. (1992) to be an optimum for 80% emulsions. The final soap phase temperature used was 30-33°C. Preparation of the concentrate was at 45-50°C to aid emulsifier dispersion.

4.3.5.4 Cutter Addition

Cutter was more reliably and accurately pre-blended into the bitumen phase in the laboratory due to inconsistent performance of the pump used for co-milling. Cutter is more easily co-milled in the production facility in Napier. Differences in the processing methods needs to be investigated to establish the effect of the difference.

4.3.5.5 Flow Rates

Final emulsion was produced at a rate of 1.2 litres per minute on the laboratory mill with approximately 4 litres produced per trial emulsion.

4.3.6 Detailed Design Method

The initial design phase screened raw materials, identified appropriate settings for processing variables and established the required testing regime to evaluate prototype emulsions. This prepared development for detailed design using experimental design techniques.

4.3.6.1 Review of Experimental Design

Experimental design is a scientific approach to design which consists of making purposeful changes to inputs or factors of a process to observe the corresponding changes in outputs or responses (Barnes, 1996a). This techniques was applied to the high bitumen content emulsion; purposeful changes were made to the input components such as emulsifier, acid and kerosene and corresponding changes in output variables such as viscosity, break index and adhesion were observed. By understanding the relationship between the input components and the observed outputs the formulation was controlled to give an optimum product.

A number of different types of experimental design techniques exist, these include: mixture designs, response surfaces, central composite designs, and factorial designs. Of these factorial experimental designs were the most appropriate. Mixture designs only explore changes in concentration levels and are unable to explore changes in process variables; factorial experimental designs give the flexibility to investigate both. Response surface designs are generally used to fine tune a complex process to find an optimum, making them inappropriate for the task at hand. Central composite designs are similar to factorial experimental designs but require an increased number of runs.

Factorial experimental designs, as mentioned previously, were used by Uaphithak (1991), Kerr (1995b) and Moskowitz (1983) in the design of similar processed products.

Experimental designs have a number of advantages over traditional approaches of experimenting with one variable at a time. Experimental designs examine interactions between multiple variables as well as the effect of changing single variables. This allowed for example, the combined effect of changes in emulsifier and acid on the final emulsion pH to be investigated. Experimental designs are also more efficient than experimenting with one variable at a time, in that they yield more information for a given number of experiments. Experimental designs also provide statistical information to quantitatively assess the significance of results. Experimental design is a systematic approach, therefore an optimum product is systematically arrived at rather than an average performing product being stumbled upon. Through experimental design a more complete understanding of how the formulation variables can be manipulated to achieve the desired output responses. This understanding is valuable when changes are to be made to a formulation.

4.3.6.2 Design Of Experiment

A number of points need to be considered when designing a factorial experimental design. These include (Barnes 1996b):

- Number of significant variables to be investigated.
- Number of levels to be investigated. ie High, mid and low component levels.
- Number of response variables to be tested.

Significant variables to be investigated in an experimental design can be identified using quantitative techniques such as Plackett-Burman screening experiments. These types of screening experiments are more useful where little is known of the process (Stowe & Mayer, 1966). Through discussions with those familiar with bitumen emulsification and study of literature an understanding of the most significant variables controlling an emulsion were identified. The key variables to be investigated were:

- Cutter content
 - Acid content (pH)
- Emulsifier content
 - Salt content

Processing variables would be held constant at levels established in section 4.3.3.

The design would investigate components at two levels: high and low. Percentage levels for the four factors were also set based on product literature and discussions with those experienced in bitumen emulsion formulation. Table 4-2 sets out the four key factors selected for experimentation and the ranges over which they were investigated. The factors were coded A, B, C and D for ease of handling during design work.

Table 4-2 Key Variables and Percentage Levels Selected For Experimental Design

Code	Factor	Low	High
A	Emulsifier	0.20%	0.30%
B	Soap pH	2.5	2.0
C	Calcium Chloride	0.025%	0.10%
D	Kerosene	0.75%	2.25%

Note: All percentages are based on total emulsion weight.

Two replicate centre-point runs with components set at their mid levels were added to allow an estimation of experimental error and establish statistical significance to results. A design matrix seen in Table 4-3 set out the various combinations of high and low levels for each of the experimental runs for a four-factor, two level experiment. A high level is indicated by a '+', low levels by a '-' and mid values by a '0'. Yates coding was used to identify each run; a run coded 'b' had factor B at a high level indicated by a '+' in the B column, all other factors were at their low levels indicated by '-'s.

The design matrix was then converted to a treatment combination table describing the specific component levels for each run. This was done by substituting the component percentage levels from Table 4-2 for the +'s and -'s in the design matrix. Table 4-4 sets

out the treatment combinations for the experimental design. The runs were carried out in random order to reduce experimental error; apart from the centre-point runs, which later augmented the design. The centre-point runs would ideally be included and randomised into the design at the start of the experiment. The treatment combination table provided a complete set of experiments to explore the high bitumen content emulsion formulation. The results of the experimental design are outlined in Chapter 7.

**Table 4-3. Full Factorial Experimental Design Matrix for
Four Factors at Two Levels.**

Factors					
Run	Code	A	B	C	D
1	1	-	-	-	-
2	a	+	-	-	-
3	b	-	+	-	-
4	ab	+	+	-	-
5	c	-	-	+	-
6	ac	+	-	+	-
7	bc	-	+	+	-
8	abc	+	+	+	-
9	d	-	-	-	+
10	ad	+	-	-	+
11	bd	-	+	-	+
12	abd	+	+	-	+
13	cd	-	-	+	+
14	acd	+	-	+	+
15	bcd	-	+	+	+
16	abcd	+	+	+	+
17	centre ₁	0	0	0	0
18	centre ₂	0	0	0	0

Table 4-4. Experimental Design Treatment Combinations: Variation of Selected Variables Between High and Low Levels.

Run	Random	Code	A Emulsifier	B pH	C CaCl ₂	D Kero
1	10	1	0.2%	2.5	0.025%	0.75%
2	6	a	0.3%	2.5	0.025%	0.75%
3	16	b	0.2%	2.0	0.025%	0.75%
4	8	ab	0.3%	2.0	0.025%	0.75%
5	3	c	0.2%	2.5	0.10%	0.75%
6	9	ac	0.3%	2.5	0.10%	0.75%
7	15	bc	0.2%	2.0	0.10%	0.75%
8	13	abc	0.3%	2.0	0.10%	0.75%
9	5	d	0.2%	2.5	0.025%	2.25%
10	1	ad	0.3%	2.5	0.025%	2.25%
11	12	bd	0.2%	2.0	0.025%	2.25%
12	4	abd	0.3%	2.0	0.025%	2.25%
13	14	cd	0.2%	2.5	0.10%	2.25%
14	2	acd	0.3%	2.5	0.10%	2.25%
15	11	bcd	0.2%	2.0	0.10%	2.25%
16	7	abcd	0.3%	2.0	0.10%	2.25%
17	17	centre ₁	0.25%	2.25	0.0625%	1.50%
18	18	centre ₂	0.25%	2.25	0.0625%	1.50%

4.3.7 Conclusion

A formulation design methodology consisting of three phases was established to develop the high bitumen content emulsion. This included initial design, detailed design and optimisation. The initial design phase investigated raw material, testing and processing variables. Variance in the production process and pH, viscosity, break index and residue tests were found to have an acceptable level of less than 6%. Adhesion, settlement and cohesion tests were not established during test evaluation and had inherent variances.

Emulsifier screening identified Dinoram SL to be the most appropriate emulsifier. Hydrochloric acid, calcium chloride, kerosene and 180/200 bitumen were also identified as suitable raw materials to be used. Processing conditions were investigated to establish a bitumen phase temperature of 130°C, soap phase temperature of 33°C and a desired exit temperature of 86°C. Mill gap would be set at fine tolerance of 0.15 mm. The initial design phase provided a base on which further formulation development was carried out.

The method for conducting detailed design using a full factorial experimental design with four factors at two levels was outlined. The results of the detailed design and optimisation phases of laboratory development are outlined in Chapter 7. Before outlining these results the Project Start (Chapter 5) and Specification stages (Chapter 6) which precede the development activity are addressed.

5. PROJECT START- IDEA CAPTURE AND SCREENING AND INFORMATION COLLECTION

5.1 INTRODUCTION

The product development process developed for Higgins as described in Chapter 4 begins with the Project Start stage. In this stage ideas are captured and screened to identify the most attractive for further investigation. Once an idea has passed screening, basic information is collected on the market, technical and financial areas of the product to initially investigate the idea. A project proposal is produced which describes the projects aims and objectives more clearly, allowing better decisions to be made on project viability by management.

This section addresses tailored product development systems required for the idea collection and screening phases and the systems to undertake information collection. This section does not cover the development of project proposal documentation. Objectives for this section included:

- To develop suitable product development systems to search, capture and screen ideas for new development projects at Higgins.
- To develop a system to manage technical information within Higgins.
- To demonstrate external avenues available to Higgins for information collection.

5.2 IDEA CAPTURE

Ideas are the triggers to product development and need to be documented in some way so they are not lost and are systematically addressed. Developing appropriate systems to formally capture and screen ideas is therefore an important focus of work.

A number of large successful industrial companies were surveyed to gain a practical perspective on how they captured ideas for development. The various systems used by these companies were analysed and an appropriate method was generated for Higgins. Telephone interviews were conducted with technical staff and managers from the following companies:

- Milburn Cement.
- Humes Concrete.
- One company requested to remain anonymous.
- PDL Industries.
- Fulton Hogan.

Below is a summary of the findings:

- New product ideas from these companies generally came from market needs determined through focus groups, customer feedback, sales representatives and technical people.
- Ideas were often discussed in an informal way with technical staff.
- Ideas were then documented in a formal way. ie Market opportunity form.
- Formal idea generation sessions were not conducted by any of the companies as a means of producing ideas for future projects.
- Concepts were generally not ranked by any of the companies surveyed.
- Ideas were generally subjectively evaluated by management.
- Old ideas were generally not reviewed.

The survey findings revealed an informal approach to handling ideas, this is a reflection of current practise and not best practise. Measures however existed in these companies to formalise the capture of the ideas at some stage, this consisted of a form to detail the basics of an idea.

A concept capture sheet for documenting ideas at Higgins was produced based on the research (Refer Appendix 5.1 Concept Capture Sheet). The sheet can be filled out by any person with a potential development idea. The following sections were provided in the concept capture sheet to describe the various aspects of an idea:

- Subject/Topic: Name of concept. eg High bitumen content emulsion.
- Information source: Where the idea was found? eg Name of publication, book etc.
- Basic description: What are the basic technical principles behind the idea?
- Implication for operations: What are the benefits? Why is it a good idea?
- Potential for implementation: What are the likely problems? Will extra equipment be needed? What will be the likely costs involved?
- Key words: Descriptive words able to be used to search and locate the product concept in the Higgins Information Catalogue system.

- Illustrations: Space provided for diagrams to clearly present possible ideas.
- Review comments: Once the idea is reviewed comments on the screening of the idea can be documented and later reviewed.

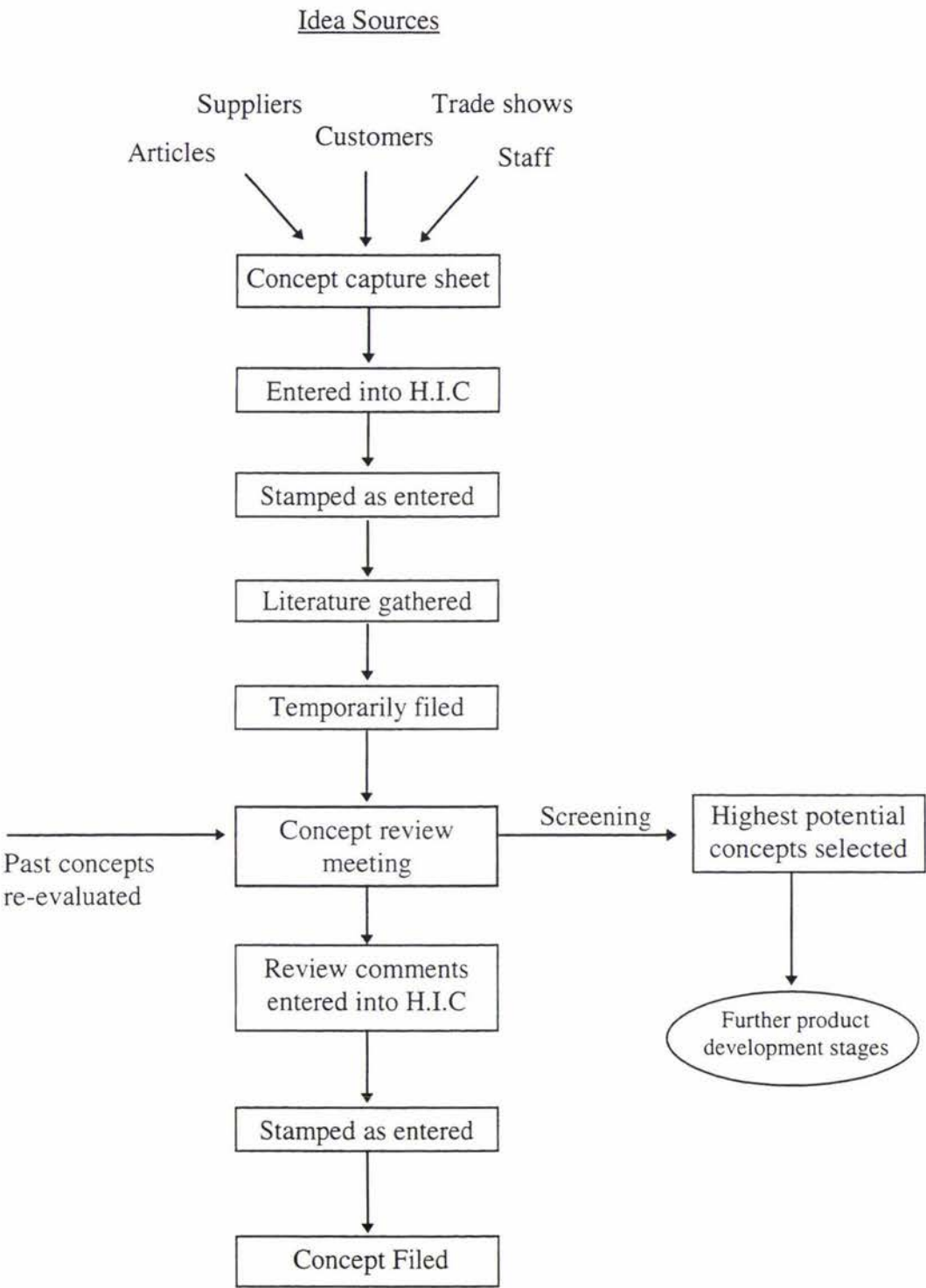
The concept capturing was integrated into the Higgins Information Catalogue system (described in section 5.4) to allow efficient handling of new product concepts. Figure 5-1 illustrates how the concept capture sheets are incorporated into the information handling system to initiate the product development process.

Ideas flow from numerous sources and are formally captured using the concept capture sheets. The information on these sheets are entered into the Higgins Information Catalogue (H.I.C) database and stamped to indicate it has been processed. The concept capture sheets along with any supporting literature are temporarily filed till a quarterly concept review meeting is held. During the concept review meeting the ideas are screened and comments on the concept's viability are recorded on the concept capture sheets. The comments are entered into the database and can be reviewed at a later stage to re-evaluate the concepts viability. The concept capture sheet along with any supporting literature is then permanently filed. Concepts which pass the initial screening enter the product development process. The concept capturing system provided a formal means to initiate the development process and was incorporated into the Higgins Information Catalogue system for efficiently handling. The process of screening these concepts will now be examined.

5.3 IDEA SCREENING

Once ideas are captured they need to be screened to eliminate unprofitable and un-viable concepts. This initial decision to proceed with an idea needs to be approached in an unbiased and systematic manner to correctly and consistently identify attractive projects. A review of literature revealed the most simple, effective and widely used tool for screening concepts was 'scoring models' (Hass, 1989; Crawford, 1993; Cooper, 1993). Checklists, decision trees and profile modeling were other methods used.

Figure 5-1 Higgins Idea Capture and Screening Process



Checklists are basically a list of criteria that ideas must have in order to be considered worthy of development. Management used the checklist as a memory jogger to assist in the assessment of ideas. Checklists were simple but provided no means to quantitatively assess and rank projects.

Decision trees were more complex and assessed the probability of success with likely financial return. Decision trees were a highly quantitative approach where all factors impacting on development were reduced to numerical costs and revenues that were then fed into a computer model (Balachandra, 1989). This can be a time consuming process to yield a decision which is dependant on the accuracy and completeness of the model and does not account for qualitative aspects of product development.

Profile modelling was a graphical method of assessing a project idea. A set of criteria was assessed on a five point scale and a profile line of the criteria scores graphically illustrated the attractiveness of the concept. This system was good for quickly highlighting strengths and weaknesses of concepts but was unable to effectively prioritise concepts.

5.3.1 Concept Scoring

Scoring models provided the ability to quantitatively rank projects without being time consuming and complex, these factors made scoring models appropriate for use at Higgins. The principles behind scoring models are relatively simple; firstly, a set of criteria is established on which all projects are screened against. Secondly, fixed weightings are assigned to criteria depending on their importance. Each criteria is then rated on a number scale; the ratings are then multiplied by the weightings to give a weighted score for each criteria. The sum of the weighted scores for all criteria is calculated to arrive at a total score for a concept. The total scores allow quick and simple comparisons to be made between the various concepts. An example adapted from Hass, 1989 and Cooper, 1993 demonstrates the principle. Firstly criteria are set with appropriate weightings.

Criterion	Weighting (%)
1. Market size and growth	20
2. Consistency with corporate objectives	15
3. Ability to match competitors strengths	20
4. Ability to manufacture	30
5. Ability to sell	15
Total	100

Each criteria is then rated on a scale from 1-5. The ratings are multiplied by the weightings to achieve a weighted score. The sum of the weighted scores gives a total score for each idea. Table 5-1 sets out the calculations.

Table 5-1 Idea Screening Using Concept Scoring

Criteria	Weighting	Rating		Weighted Scores	
		Idea 1	Idea 2	Idea 1	Idea 2
Market size	20	5	4	100	120
Corporate objectives	15	4	2	60	30
Match competitors	20	5	3	100	60
Ease of manufacture	30	3	5	90	115
Ability to sell	15	4	3	60	45
Totals	100			410	370

Rating scale: 5-Excellent, 4- Good, 3-Average, 2-Fair, 1-Poor.

Idea 1 with 410 points would be selected over Idea 2 with only 370 points. These numbers can be converted to percentages and a hurdle percentage set which a concept must exceed to be further considered. eg 60% or 300 points.

5.3.2 Determining Screening Criteria

Careful selection of screening criteria is important to ensure accurate identification of viable and profitable projects. Cooper, 1993 cites a checklist published by the Industrial Research Institute as a basis for selecting criteria from. Ten of the most appropriate criteria were selected for screening at Higgins:

- The product will fulfil a real market need.
- The likelihood of technical success is high.
- The return on investment will be high.
- The product will be unique.
- The product can be manufactured easily with current or familiar technology.
- The product is consistent with company objectives and strategy.
- The product specification defining exactly what is to be produced is clear.
- The time to complete the project will be short.
- A great deal is known about the market and customers the product will be sold to.
- The product able to be well marketed and promoted.

A balance needs to be found in the number of criteria used for screening; too few and the model is too simplistic, too many and the model becomes cumbersome and complex. The criteria selected provided a workable model which considered the main aspects for concept screening.

5.3.3 Assigning weightings

Gaining an appreciation of the relative importance of key factors for success in product development assists in assigning weightings to identify viable concepts. The factors which are important for successful product development need to be weighted more heavily to better screen concepts. Studies on new product success and failure have found genuine market need, uniqueness, and consistency with company technology as being key criteria for success (Cooper, 1993). These areas were weighted appropriately in the screening model developed for Higgins to reflect their importance and necessity for success.

Assigning weights is a very subjective process and is one of the main criticism of scoring models (Crawford, 1994). To arrive at accurate weightings the model must be trialed and revised, the fine tuning process however does not require much effort. Initial weights were placed on criteria for the Higgins scoring model based on Cooper's studies on new product success and failure. An initial trial was run with these weights, a number of known unfeasible concepts were scored using the model to determine

whether the model would eliminate these. The initial trial showed some adjustment was necessary. The initial weightings were heavily biased towards market related factors, meaning a technically unfeasible concept would still pass the screening. To amend this criteria was divided into three main areas of feasibility: market, technical and financial. The total weight was then equally divided amongst these three areas. This meant a concept had to demonstrate potential in all key areas to pass screening. The concept scoring sheet developed with the final weightings is seen in Figure 5-2.

5.3.4 Discussion

Scoring models like all other methods have their strengths and weaknesses. One of the difficulties with scoring models is the subjective nature of scoring. An individual may be biased towards a concept and therefore rate it more highly or maybe making uninformed judgements. To overcome these problems a number of people can score the concepts and the results collectively analysed to provide a balancing affect.

When analysing results from screening using scoring models it is often useful to examine why concepts have been accepted or rejected. If a project is accepted largely on the basis of a single strength the risk of the project will increase if it has been over-rated. Also if a concept is rejected, the areas which made it unattractive may need to be assessed. Looking beneath the final weighted score for each concept provides insights into the likely risk areas of the project. Further trialing of the concept scoring model will streamline this more formal approach to project selection at Higgins.

5.4 INFORMATION MANAGEMENT SYSTEMS

The next phase in the product development approach developed for Higgins was to collect information on the ideas that passed screening. This is to initially investigate the idea and produce a proposal outlining the idea in more detail for further evaluation of proposed development. Systems to manage and collect information are therefore important in this phase and others.

Figure 5-2 Concept Screening Template

Concept Screening and Evaluation.

Concept Name: _____

Evaluated by: _____

Date: _____

Instructions:

For each question circle a rating (0-10) to indicate your level of agreement.

	Strongly Disagree		Strongly Agree	Weighting
1. The product will fulfil a real market need.	0	1 2 3 4 5 6 7 8 9 10		10
2. The likelihood of technical success is high.	0	1 2 3 4 5 6 7 8 9 10		20
3. The return on investment will be high.	0	1 2 3 4 5 6 7 8 9 10		25
4. The product will be unique.	0	1 2 3 4 5 6 7 8 9 10		8
5. The product can be manufactured easily with current or familiar technology.	0	1 2 3 4 5 6 7 8 9 10		10
6. The product is consistent with company objectives and strategy.	0	1 2 3 4 5 6 7 8 9 10		7
7. The time to complete the project will be short.	0	1 2 3 4 5 6 7 8 9 10		4
8. The product specification defining exactly what is to be produced is clear.	0	1 2 3 4 5 6 7 8 9 10		5
9. A great deal is known about the market and customers the product will be sold to.	0	1 2 3 4 5 6 7 8 9 10		6
10. The product is able to be well marketed and promoted.	0	1 2 3 4 5 6 7 8 9 10		5

Patterson, (1993) likens the product development process to an information assembly line. The product developer takes raw and rough information, adds value to it in various stages until that information describes the production, support and use of a quality new product. Information collection is particularly important in the early stages prior to development where market, technical and financial information is gathered to establish feasibility, set design specifications and make decisions on development direction. Good information collection and management systems are therefore important in any product development approach.

Much technical information on various fields had been gathered by the company. However it needed to be organised in a meaningful way to better utilise it in product development. Systems and procedures also needed to be developed to better manage the flow of new information into the company for product development and consolidate this into a technical library.

The first step required to organise the technical library was to catalogue its contents. Brief details of the information contained in the library were documented to enable the organising of the material into meaningful categories. The second step was to create a means of storing and accessing the catalogued contents of the library; this was done using a computer database. The database developed was called the Higgins Information Catalogue or (H.I.C). The system was divided into two main section: data entry and information searches, which allowed data to be stored and extracted. A screen shot of the main menu can be seen in Appendix 5.2 Higgins Information Catalogue. Material put into the system was classed as either 'Reference material' or a 'New product concept' (discussed in section 5.2). Material was screened for relevance; material judged as useful technical information would be entered as 'Reference material' into the database. Once the material passed screening it was systematically classified using the following key fields:

- Category- Classification of information content. ie concrete, bitumen, polymers.
- Material type- Description of material medium or format. ie video, report, magazine.
- Key words- Four key words were selected to describe the material.
- Author and Title.

The system was designed to be flexible allowing new categories and material types to be added with full editing capabilities. Book lists could also be generated to allow the contents of the database to be queried.

Having catalogued the current contents of the library and developed a tool to efficiently store and access this information, procedures were needed to manage the ongoing flow of incoming information. Firstly an assessment of the deficiencies of the situation were made and the following recommendations were suggested:

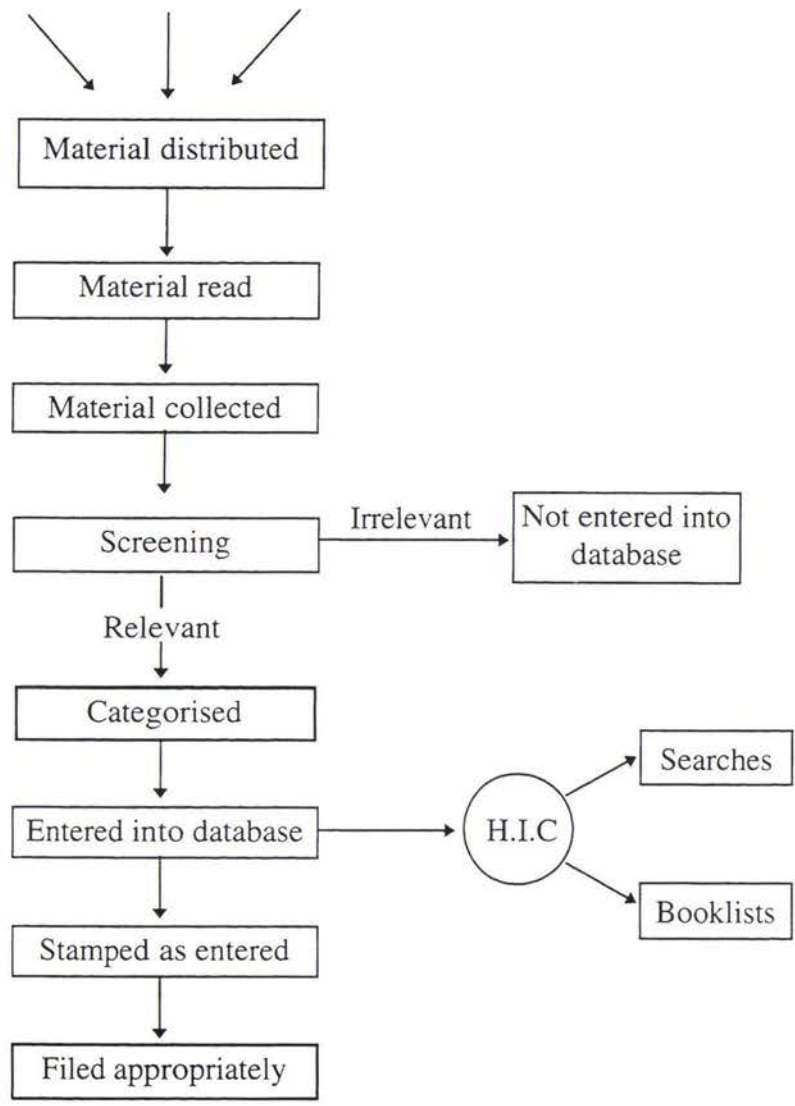
- Responsibility for the maintenance of the library be assigned to a single person.
- Regular and planned maintenance to be carried out on the library system.
- Examine the relevance of some periodicals.
- Adopt the Higgins Information Catalogue database.
- Link the physical storage of material to categories in the H.I.C for easy access.
- Inform appropriate parties of new information.
- Implement a system to record items taken from the library.

Taking these recommendations into account an overall approach to information management was then developed shown in Figure 5-3. In-coming material is distributed, read and then collected into one location. At the end of the month the material is then screened as to its relevance. Only material that is of technical benefit to product development is entered into the database. After screening, the material is categorised, entered into the database and stamped to indicate it has been processed. The material is then physically filed according to the category entered into the database.

Technical information was organised in a more meaningful way to facilitate better research based product development. The system designed provided a means to continually manage the flow of information into the technical centre with an appropriately designed cataloguing tool. This was to allow greater benefit to be obtained from the information being held by the company for development projects.

Figure 5-3 Flow Diagram of Information Management System at Higgins

Information from various sources



5.5 INFORMATION COLLECTION AVENUES

Having increased the ability to access and use technical information within the company an effort was made to increase the ability to use external means to assist in research based product development. The ability to research and gather information increases the quality of product development as the difficulties of previous researchers can be discovered and possible solutions sought. A wide range of avenues exist to obtain research information; this research project demonstrated some of the information gathering avenues and techniques appropriate to Higgins. These included the internet, libraries, databases, patent information and consultants.

5.5.1 Internet

The internet is a modern medium of information collection and provides access to a large international body of knowledge. A preliminary search on the internet was conducted to locate possible sites containing up-to-date information on high bitumen content emulsions. A number of sites were located and various people e-mailed for information and suggestions. Contacts were made with the Civil Engineering Department at Leeds University, papers were sourced from Belgium and patent information from Australia.

The internet was also useful in finding competitor information and keeping abreast of the latest international innovations and developments. The top internet sites relevant to Higgins were located and catalogued with regular checks and searches recommended to remain current with industry trends. Links with international research bodies through the internet proved to be a valuable avenue to assist information gathering in the product development approach.

5.5.2 Libraries

University and commercial libraries were a more traditional source of information used in the project. Universities are knowledge centres with their libraries containing much useful information and research. This project provided access to a vast amount of information through the Massey, Auckland and Canterbury university libraries through the inter-loan systems networking these libraries. Both Canterbury and Auckland have specialised civil engineering libraries which were particularly useful.

Commercial libraries also exist to service industrial research needs. Telis was one such organisation used to provide technical information on the industry. Telis have access to a large volume of civil engineering information and were used to conduct searches for information related to high bitumen content emulsions. Updates on new standards, papers and books are also distributed monthly to keep staff current. The recommendation to join this organisation has increased the information gathering ability for research and development at Higgins.

5.5.3 Information Databases

Large international databases containing much information relevant to Higgins were used in the research work and could be accessed through the libraries or the internet. The following databases relevant to Higgins were offered through the Massey library.

- Micro Internationally networked database.
- NewZIndex New Zealand daily newspapers.
- ABI Inform Business oriented database.
- Current contents Multi-disciplinary database accessing over 7,000 journals.
- Uncover Multi-disciplinary database accessing over 16,000 journals.
- First search Multi-disciplinary database networked to 10 international databases.

These databases can be used to gather both market and technical information for product development in the civil engineering field. An assessment of access to these types of databases through the internet was made for Higgins. It was found to be more convenient and time efficient however to access the databases through the commercial industrial library and contract this information searching out to trained personnel.

5.5.4 Patents

Patents were an invaluable source of unpublished and new information for product development used in the project. Below are some facts and benefits of using patent information (Johnston, 1979).

- They are the single biggest source of technical information. It is estimated that 85% of all technical knowledge is only available in the form of patents.
- Information must be comprehensive in order to obtain the patent.
- The information must be commercially utilisable.

- The patent must contain new data.
- The information provides a starting point for further developments.
- Information gained from patents can reduce development time thus reducing costs.
- Patents are filed before publishing data in journals, meaning earlier access to information.
- Assists in identifying competitors development direction.

The New Zealand Patent Office provided New Zealand, Australian, French and American patents with valuable information on high bitumen content emulsions. Advice was sought from a patent attorney on this information to clarify the scope of the legal claims detailed in the patents to avoid breaching patent law.

5.5.5 Consultants

The product developer must be able to effectively draw knowledge from consultants and experts in various fields and combine that knowledge together in a meaningful way. Higgins' external consultant, Glynn Holleran was a useful source of information for the project. As an expert in bitumen applications he provided advice as to possible pitfalls and solutions to the successful production of the product. Suppliers and production personnel also provided information of value to the product's development.

Developing appropriate systems and avenues to increase the research capability of the company facilitates more informed and effective product development.

5.6 CONCLUSION

The Project Start stage of product development at Higgins consists of Idea capture and Screening. Initial information collection is also carried out. Tailored product development systems were created for this stage at Higgins.

Idea capture was formalised with a concept capture sheet which allowed ideas to be locked into the Higgins product development process. While a scoring model was the most appropriate and simple system to quantitatively screen and rank ideas for selection of development projects.

Organisation of the company's technical library was carried out and a cataloguing tool developed to enable better access to consolidated product development information. New avenues of conducting research were demonstrated to the company through the project and included the internet, patents, various libraries and databases. These avenues would assist in the collection of information in initial investigations.

The development of these systems to manage the collection, capture and screening of information and ideas provided suitable systems for the first stage of the Higgins product development approach.

6. PRE-DEVELOPMENT- PRODUCT DESIGN SPECIFICATIONS

6.1 INTRODUCTION

The Project Start stage described in Chapter 5 established whether a screened idea having being initially investigated was worthy of further detailed investigation. Detailed investigation is carried out in the Pre-development stage in the Higgins product development approach outlined in Chapter 4. The Pre-development stage consists of two distinct Phases: Feasibility and Specification. Development of appropriate product development systems for the Feasibility phase are not covered in this study as the project was considered feasible prior to the projects initiation. Once the product concept has demonstrated feasibility the development of product design specifications can begin.

The aim of the work during the Pre-development stage in this study was two-fold:

- Illustrate a suitable method to develop product design specifications at Higgins.
- Develop a template design specifications for the high bitumen content emulsion.

6.2 PRODUCT DESIGN SPECIFICATIONS

The writing of product design specifications is a key planning activity carried out prior to development work to provide direction and focus. The purpose of the product design specification is to set out in precise measurable terms the attributes of the product to be developed. Many types of specifications exist to describe various aspects of a product including: quality control specifications, production specifications and design specifications. Of particular interest at the Pre-development stage are design specifications describing the intent of proposed development. Design specifications can be broken into two categories: a marketing specification and a technical specification. Studies have shown that companies who generate both marketing and technical specifications outlining not only the product's physical characteristics but also details of marketing factors, achieve a higher rate of product success. Less successful companies concentrate only on product function and price (Cross et al., 1992). The marketing specification is more concerned with the many qualitative factors related to the product,

such as the needs of the market, intended users, user requirements and marketing strategy. The marketing specification addresses issues relating to why the product is being developed and for whom. The technical specification is more of a quantitative description of the product's physical characteristics, such as required viscosity, binder content and cohesive strength. It describes in technical terms what will be developed. Generating both types of design specifications begins with market and customer research.

6.3 CUSTOMER RESEARCH

Customers are consulted prior to drafting design specifications in order that the product developed is desirable to them. Customer research gathers the opinions and requirements of the parties who will eventually use and specify the product; these requirements can then be designed into the product through the design specification.

Higgins' customers are typically local bodies, government bodies, civil engineering consultants and large businesses. A large proportion of Higgins' work comes from only a few large sources. Six of these key customers were contacted for interviews to discuss issues related to the development of the high bitumen content emulsion. Issues discussed included:

- Perception of general emulsion technology.
- Problems with other emulsion products.
- Market need for a high bitumen content emulsion.
- Product requirements.
- Information required in specifying a high bitumen content emulsion.
- Bench-marking of Higgins' performance against other competitors.

A customer research brief outlining the aims, objectives and deliverables of the research work was generated in order to guide the information gathering. A questionnaire was then drafted and used in the telephone interviews with key customers. A sample questionnaire with customer responses can be seen in Appendix 6.1. Primary data gathered from the customers was also supplemented by industry specifications to describe more fully the product requirements in the design specifications. The

information gathered from customers highlighted areas that development would need to address in order to be successful. A summary of the findings has been divided into three sections: key product areas, key performance requirements and general issues.

6.3.1 Key Product Areas.

Four broad product areas were given to customers for ranking to establish a priority area for development. The aggregate rankings for these areas was as follows:

- | | |
|----------------|----------------|
| 1. Performance | 3. Safety |
| 2. Cost | 4. Environment |

Good technical performance would therefore needed to be the focus of development.

6.3.2 Key Performance Requirements.

Probing customers further on performance characteristics, the following attributes were identified as being important:

- Even application, with no streaking on spraying.
- High enough viscosity so emulsion would not run off when applied.
- Fast cure to allow the quick resumption of traffic with no damage to new surface.
- High adhesion for good stone retention
- Performance equal to or better than cut-backs in terms of life and wear.

6.3.3 General Issues

Customers reported a number of bad past experiences with emulsion products, making them wary of using these products in the future. Part of the problem was said to have been due to a lack of experience and understanding by contractors of how emulsions are to be handled and applied. Training in this area was suggested. Demonstrated product performance using a test site would assist in overcoming the lack of confidence in emulsion products. Customers also wanted greater control of traffic by contractors on newly laid surfaces to allow greater curing. Customers were also surveyed on the need for the product. The response was positive, with international trends towards the technology being cited as a likely indication of future growth in the area.

6.4 MARKETING SPECIFICATION

Customer requirements gathered through the customer research were encapsulated formally in the marketing specification. The marketing specification contained the following elements:

- Product concept statement.
- Technical description of product.
- Intended application.
- Market environment (Opportunities and threats analysis).
- Product Strategy.
- Customer Benefits.
- Weaknesses and potential problems.
- Estimate of market size.
- Costs.

A template marketing specification was generated for the high bitumen content emulsion and can be seen in Appendix 6.2 Marketing Specification. A description of each of the elements in the marketing specification provides a complete understanding of its contents. Excerpts from the marketing specification are included to illustrate the points made.

6.4.1 Product Concept Statement

A product concept statement is a device to communicate the benefits, strengths and reasons for being (Moore, 1993). It includes the essence of the product, the benefits offered and encapsulates in a brief manner the direction of development.

There are two types of concept statements:

i) Core ideas.

Core idea concept statements are short, factual descriptions of what the product can do for the customer.

ii) Concept Positioning statement.

Positioning statements are generally longer, listing secondary benefits and comparing the product with other competitive products within the company's range. They are more promotional and designed to sell the idea; such product concept statements are usually used in customer testing.

Concept statements for industrial products tend to be more factual and provide an unbiased comparison of the new products and likely performance against other competitive products. A core ideas product concept statement was generated for the project in the template marketing specification and is seen below.

'A high bitumen content emulsion with a residual binder of greater than 75% meeting the TNZ M1 & P17 specifications. The product is to be used primarily in spray chip sealing as an alternative to hot cutback bitumen for primary roads under standard conditions. The major benefits to the customer include a safer, more environmentally friendly alternative.'

6.4.2 Technical Description of Product

This statement discriminates the product from others in a technical sense. This statement is meaningful to a civil engineer or technical personnel providing an understanding of the differentiating characteristics and technology. An example of the technical description generated for the developed product can be seen below.

'A rapid breaking, rapid curing emulsion with a residual binder of 75% or greater.'

6.4.3 Intended Application

This is a description of how and where the product is to be correctly used. An example of the high bitumen content emulsions intended application is seen below.

'Chip sealing and resealing according to TNZ/M1 and P17 Specifications.'

6.4.4 Market Environment

This section outlines the current situation of the market, the demand and the driving forces behind development; this provides an understanding of why development should take place and puts development in context. Market threats and opportunities to development are also given to provide information on market attractiveness. A description of the competitors and any known intentions provides insights into the environment the product will be launched into. Below are a few of the key market factors that influenced the project.

- *Competitors- Technics have international links with major oil companies which have developed this type of technology. These links provide a preferential option for Technics to purchase the right to produce the product and introduce it to the New*

Zealand market. To remain competitive Higgins needs to develop this type of product.

- *Industry specifications- New specifications have been developed which have a section for high bitumen content emulsions. Customers can begin specifying this type of product and Higgins will need a product which fulfils these specifications.*
- *Safety Issues- Employers are coming under increasing pressure to comply with Health and safety requirements. Lower spray application temperatures provided by emulsions offer a safer workplace.*
- *Environmental concern- Lower levels of kerosene make emulsions a more environmentally sound sealing technique over hot cut-back sealing.*

6.4.5 Product Strategy

This section outlines what will be achieved by having the product in the company's range. Possible product strategies include head to head competition, market growth or widen applications. A description of where the product is placed in the company's product range is given along with the specific benefits the product will be promoted on. The product strategy can be broken into two statements: a positioning statement and a strategy statement (Shekar, 1996). The positioning statement outlines where in the company's range the product will sit. The strategy statement outlines why it is in the range. Below is an example from the project.

Product Positioning:

The product will extend the company's cationic rapid set (CRS-2) range of emulsions.

Product Strategy:

- i) To gain a greater number of sealing and resealing contracts by offering a high performance and cost competitive alternative to hot cutbacks.*
- ii) Offer a safer and more environmentally friendly product over hot cutbacks.*

The positioning and strategy statements take the ranked requirements of performance, cost, safety and environment from the customer research and incorporates them into the product strategy to be achieved through development.

6.4.6 Customer Benefits

In this section the desirable product characteristics the product is intended to have are listed in customer terminology. These benefits make up the product’s competitive advantage and describe why customers would buy the product over others. The intended product benefits are based on delivering to the customer the performance characteristics they felt were important, as determined through the customer research. Examples of benefits the high bitumen content emulsion offers customers over lower content emulsions include:

- *Reduced transportation costs*
- *Improved application efficiency*
- *Faster cure times*
- *Better adhesion.*

6.4.7 Weaknesses and Problems

An honest look at areas of potential difficulty and problems needs to be described so solutions can be generated or weaknesses minimised in the next development stage. The problem areas to be addressed are also based on issues customers may have raised during interviews. Examples of potential weaknesses or problems listed in the marketing specification include:

- *Run off.*
- *Streaking.*
- *Lack of customer confidence with the use of emulsions.*
- *Lack of understanding of contractors with emulsions.*

6.4.8 Estimates of market size

To determine the potential revenue of the product an estimate of market size and share needs to be estimated. This information will form the basis of a sales forecast to determine revenue and return on investment. For example the total market potential for reseals in the Higgins operating region was estimated from statistics.

Total Reseals Nth. Island 3,416.9 Km \$52,793,500

The estimated proportion of this market potential Higgins could expect can then be made.

6.4.9 Cost

An estimate of the end cost to customers and production costs are made to determine the competitiveness of the product in the market. This is important in a tendering environment which is highly price driven. A cost of 80 ¢/litre was estimated for the emulsion which is similar to current emulsions.

6.5 TECHNICAL DESIGN SPECIFICATION

The technical design specification converts user requirements and the product concept statement into measurable physical product attributes such as: viscosity, adhesion, cohesion etc.. Besides clarifying development intent the technical design specification sets out criteria which are used to screen prototypes later in the Laboratory Development stage.

The technical design specification does not specify exactly how the product is to be made and what it should contain but only how the end product should perform and function; it specifies ends and not means. The technical design specification will be able to be met by a number of concepts and does not limit creativity and innovation. For example a specification will not specify a certain emulsifier but the viscosity levels to be achieved. This allows scope to generate innovative solutions by a number of means, such as a combination of emulsifiers, new processes or different additives.

For the high bitumen content emulsion target values for the physical attributes were set which attempted to meet customer, contractor and industry requirements. An initial technical design specification was created at the Pre-development stage and is often referred to as a target technical design specification. The target technical design specification formalises what is known about the direction of development at the Pre-development stage. The review and approval of the target technical design specification by management signals the approval to enter the Laboratory Development stage. The target technical design specification can then be refined throughout development to arrive at a final product specification. The final product specification covers all areas of the product including, quality control, product composition and application.

The target technical design specification was created using a method described by Ulrich & Eppinger, (1995). The method contained the following steps:

- 1. Prepare a list of metrics.
- 2. Collect competitive bench marking information.
- 3. Set ideal and marginally acceptable target values.
- 4. Reflect on results and process.

6.5.1 Prepare list of metrics

A metric is a measurable physical attribute which describes a customer requirement. For example the customer requirement ‘Won’t run off the road’ is represented by the metric ‘viscosity’. From the customer research a list of discussed needs was developed and placed in priority order. Each need was analysed in turn to decide how it would be represented numerically. For example.

Customer Need	Metric
1. Application Efficiency	Binder level
2. Won’t Run Off	Viscosity
3. Resume traffic quickly	Cure time

In general a single metric should represent a single customer need, with all needs being covered.

6.5.2 Collect Competitive Bench-marking Information.

Before assigning values to the list of metrics, an assessment of opposition products needs to be carried out to establish competitive levels. The same testing regime to be used in development should be run on competitive products to establish appropriate targets to be achieved and exceeded. The product should better a competitor’s product in a particular area to establish a competitive advantage and differentiate the product in the market. The nature of this competitive advantage will be consistent with the product strategy in the marketing specification. Below is the product strategy for the high bitumen content emulsion.

- i) To gain a greater number of sealing and resealing contracts by offering a high performance and cost competitive alternative to hot cut backs.*
- ii) To offer a safer and more environmentally friendly alternative to hot cutbacks.*

The products competitive advantage will be in the area of performance measured by binder content, viscosity, cohesion, adhesion etc..

If competitor’s products can not be obtained for benchmark testing to set specification values, values set out in their promotional literature and papers can be used. This is not a preferable option as promotional material can sometimes be misleading with different test methods and conditions being used which influence reported values. Literature values could only be used as a guide in this study as a minimum order quantity for competitive products was large and the products could only be sourced from overseas.

6.5.3 Setting Ideal and Marginally Acceptable Target Values

Based on the competitive analysis, ideal target values were initially set. These are the best values that could be hoped for. For example a binder content of 80% would be an optimistic target for the product. Marginally acceptable limits were then set on the target values, these were values that would barely make the product commercially viable. A lower binder content limit of 75% was set for the project. Upper and lower limits may need to be considered for some variables, for example:

Customer Need	Metric	Target	Limits	
			Upper	Lower
1. Application Efficiency	Binder level	80%	85%	75%
2. Won’t Run off	Viscosity	800cP	3000cP	300cP
3. Resume traffic quickly	Cure time	2 hours	4 hours	-

These target values and limits provide a basis to screen and evaluate prototypes later in the Laboratory Development stage.

6.5.4 Reflect On Results

The final step in the process was to review the list of metrics and determine if any needs are missing or values are incorrect. Several iterations were required to obtain a specification that was accurate and achievable.

Using the described process the following list of needs, metrics and values was created and formed the basis of the technical design specification.

* At the outset the test was still to be developed so no data had been built up on appropriate values.

Customer Need	Metric	Target	Limits	
			Upper	Lower
1. Application Efficiency	Binder level	75%	75.5%	74.5%
2. Won't Run off	Viscosity	800cP	3000cP	300cP
3. Won't strip	Adhesion	80%	100%	75%
4. Resume traffic quickly	Cohesion	*		
5. Good wetting ability	Cutter level	2%	4%	0.5%
6. Even surface finish	Viscosity	800 cP	3000 cP	300 cP
7. Remain open	Break rate	200%	250%	150%
8. Storable	Settlement	<1 %	1%	-
9. Tolerate cool conditions	Cohesion	*		
10.Safe	Spray temp.	85 °C	90 °C	70°C
11.Environmental	Cutter level	2%	4%	0.5%

The target technical design specification was produced based on the above customer needs/metric list and the draft industry specifications for high bitumen content emulsions. The target design specification developed at the Pre-development stage can be seen in Appendix 6.3.

The target design specification generated for the high bitumen content emulsion was refined as laboratory development progressed to describe more clearly the nature of the product. Performance guidelines put out by the Asphalt Emulsion Manufacturers Association (AEMA, 1996) were used as an appropriate format to communicate the final product specification. Although the product was not developed through to a fully commercialised product the final product specification (Appendix 6.4) illustrated to the company the required information in an appropriate format. This final product specification fully describes the product, its benefits, technical performance, application methods and limitations.

6.6 MANAGEMENT ISSUES

To maximise the benefits of producing product design specifications a number of important management points should be noted. The document should be formal and

signed, developed with input from a number of functions and require as few changes as possible (Andreasen & Hein, 1985; Rosenau, 1990; Cross et al., 1992.)

Formalising the design specification clarifies in writing what is to be developed at the beginning of the project. By signing the document the parties concerned indicate they endorse the intent and direction of development.

Group consultation is important in creating design specifications as agreement from a wide range of concerned parties reduces the necessity of re-development due to oversights. If the requirements of parties such as production and contracting personnel are considered in the planning stage, these requirements can be designed into the product rather than added later on (Andreasen & Hein, 1985).

More successful products have been found to require fewer modifications to the design specification than less successful products (Rosenau, 1990). This demonstrates good ground work has established realistic targets with an understanding of how these targets are to be achieved. When a specification has to be greatly changed half-way through development, the goals the development team have been working towards shift, often resulting in delays. A more fixed design specification becomes more critical when a large team is involved and the ramifications of small changes increase.

6.7 DISCUSSION

When setting the target technical design specification for the high bitumen content emulsion a number of challenges were faced. These included a lack of established tests, uncorrelated data and qualitative assessment of attributes.

Some of the tests necessary for setting specification and evaluating the product were not currently used by the company; the cohesion test for example. No prior experience or data was available when attempting to set appropriate cohesion levels. In addition the cohesion test was new to the industry and little published data was available from other researchers. Any results would also be highly specific to the type of bitumen and formulations used. Thus setting a metric value for cohesion was done throughout the development work as data was gathered. The data gathered throughout the project work

would make setting of specifications clearer in future.

As the Higgins technical centre is fairly new, other tests existed but still little experience or understanding of the test had been developed. For example an acceptable break index was set with little understanding of acceptable field performance to be expected from the set level. This information would also be gathered through the work. Some metrics in the specification are qualitative in nature and are unable to be accurately tested. Example of the more qualitative factors in the product's design included the subjective assessment of stone coating in the adhesion test and judging the acceptability of the final appearance of the laid surface, wetting ability.

Setting of specification targets was carried out despite the difficulties which made the specifications somewhat inaccurate at the beginning of design work. The levels that were set however provided some direction to the work and forced the consideration of many important variables.

6.8 CONCLUSION

Product design specifications are drafted during the Pre-development stage of product development at Higgins to clearly communicate the direction and focus of the planned Laboratory Development stage. Design specifications can be broken into two distinct documents; a marketing specification and a technical specification. Both are based on customer research to establish customer requirements which are then designed into the product. The marketing specification outlines why a product is to be developed, gives a description of the market environment, product positioning and desired product attributes customers require. The technical specification takes the customer requirements and converts these into quantifiable metrics which accurately describe the physical nature and performance of the product. An appropriate method was demonstrated to the company as to the creation of technical design specifications. The method consisted the following four steps: create a list of metrics, assess competitor products, assign target values and review. The target technical specification is refined to a final product specification. Examples of both a target technical design specification and a final product specification were generated for the company.

Specific challenges related to the high bitumen content emulsion specifications were discussed and it was found that well understood and established tests would produce higher quality specifications in future.

7. LABORATORY DEVELOPMENT

7.1 INTRODUCTION

Three distinct phases in Laboratory Development were identified in Chapter 4: initial design, detailed design and optimisation. Initial design identified raw materials, processing conditions and testing to be used. Detailed design was carried out using experimental design techniques to explore various formulation possibilities in order to systematically identify a likely product formulation area. Through optimisation, the formulation was refined and readied for scale-up. This chapter outlines the results from the three design phases in the Laboratory Development stage. The main objectives achieved through the design work included:

- To illustrate a systematic design method appropriate to processed products at Higgins.
- To understand how changes in formulation components affects final emulsion properties.
- To identify a preferred formulation.
- To optimise the formulation for scale-up.

7.2 INITIAL DESIGN

During the initial design phase, each of the eight emulsifiers were used to make an emulsion with a bitumen content of 75%. The emulsions were then tested to determine a preferred emulsifier. Table 7-1 summarises the results.

Table 7-1. Emulsifier Screening: Emulsion Properties

	Dinoram SL	EM44	N-39L	Emuls 640	Asfier 123	AA-60	AA57	DXP 5357100
Residue (%)	75.21	75.64	75.37	75.30	74.64	75.78	74.94	75.11
pH (Units)	2.34	2.57	2.03	2.50	2.48	2.40	3.84	2.86
Viscosity (cP)	760	770	920	3360	4040	4720	620	470
Break Index (%)	200	221	213	142	118	130	147	588

*Viscosity @ 70 °C, Spindle #1, 5rpm.

Residue and pH were not used as screening criteria. Residue was monitored to assess consistency of residual binder at approximately 75%. pH was allowed to vary according to suppliers' recommendations.

The DXP 5357100 sample proved unstable as shown by a high break index and was therefore eliminated. The break index for AA-57 was also outside the lower limit of 150% set in the technical design specification. The low break index is due to AA-57 being a slow breaking emulsifier; this type of emulsifier being more appropriate for bitumen contents in the order of 80%, where break is more rapid. Supply for AA-57 was also not established, with only a small amount available for laboratory evaluation. Emulsions made with Emulsamine 640, Asfier 123 and AA60 had extremely high viscosities in the order of 3000⁺ cP and were eliminated as they would be difficult to spray and handle.

The remaining emulsifiers were: Dinoram SL, EM-44 and N39L. Dinoram SL was selected as it gave the lowest viscosity and had the lowest break index within the range set in the technical design specification. Dinoram SL had been previously used by the company in other emulsions and had showed good results; supply was also already established.

7.3 DETAILED DESIGN RESULTS AND ANALYSIS

Emulsifier, kerosene, calcium chloride and pH levels were identified in the initial design phase as key variables to be investigated in the experimental design outlined in Chapter 4. Results from the experimental design were analysed using a computer package called 'Minitab' (version 11.0). The effect of varying single components from low to high levels were determined as well as the interactions between two, three and four components. The following outputs were produced to determine how the formulation system was controlled:

1. Factorial Fit Analysis. To identify the size and significance of effects.
2. Main effects plots. To illustrate the nature of significant single variables.
3. Interaction plots. To illustrate the significant interactions between two variables.
4. Cube plot. To illustrate the significant interactions between three variables.

Table 7-2 summarises the results from the experimental runs. Full results are held in Appendix 7.1.

Table 7-2. Summary of Test Results from Experimental Design.

Code	Residue (%)	pH (Units)	Viscosity* (cP)	Settle-ment (%)	Break Index (%)	Adhesion (%)	Cohesion Value (kgcm)
l	76.76	3.15	820	0.63	270	80	6.2
a	76.97	2.96	680	0.93	257	70	5.8
b	77.52	2.50	960	0.56	256	65	9.9
ab	76.27	2.36	760	0.13	253	50	7.6
c	76.55	3.06	630	-0.23	235	68	6.3
ac	76.40	3.29	740	-0.48	238	70	6.7
bc	76.57	2.52	730	-0.98	227	60	7.9
abc	76.27	2.30	820	-0.17	204	30	9.0
d	76.66	3.04	630	-0.04	192	80	7.4
ad	76.21	3.10	805	-0.55	197	80	5.7
bd	76.08	2.30	720	-8.65	175	60	8.9
abd	75.68	2.48	790	0.19	198	60	7.5
cd	76.46	3.17	1000	-0.07	160	60	8.6
acd	76.04	3.08	850	-3.18	194	80	6.5
bcd	76.09	2.63	920	-0.58	165	80	10.2
abcd	76.03	2.53	800	-1.13	192	55	9.0
CP1	76.40	2.83	750	0.99	200	75	6.3
CP2	76.28	2.81	750	-0.36	205	75	7.5

*Viscosity at 5 rpm, 70°C, spindle #1.

Key: a: Emulsifier b: Soap pH c: Calcium chloride d: Kerosene CP: Centre point

7.3.1 Emulsion Composition Properties

Residue and final emulsion pH are separated from the main body of results in Table 7-2 as they were not output properties to be optimised.

7.3.1.1 Residue

Residual bitumen content was kept as a constant and was tested to verify emulsions were produced at a residual binder of 75-76%. The mean residue for the experimental runs was 76.41% with a standard deviation of 0.44%. Emulsions were produced consistently and comparison between runs can be made, eliminating variance in binder content as a variable.

7.3.1.2 pH of Final Emulsion

Final emulsion pH was largely determined by soap solution pH which was controlled as an input variable in the experimental design. Final pH was recorded as a check of production quality and for setting specifications.

7.3.2 Determining Significant Effects

A factorial fit analysis was run on the experimental results to determine the effect and significance of varying the four variables over the various runs. The effect values described the size and direction of changes in emulsion properties which resulted from changes to input variables. For example, if soap pH had an effect value of +16% on adhesion, adhesion would increase by 16% as soap pH moved from high pH to low pH. To be considered significant, effects had a statistical p value of less than 0.05. This meant the probability of an observed change in a property which did not result from a deliberate change in the component levels would be less than 0.05. If no variables were significant at the 0.05 level the first most significant variable was identified as having the greatest effect. Table 7-3 contains all effect and p values for all variables. Significant variables are highlighted in grey.

7.3.2.1 Significant Effects on Final pH

Although final pH was not a property to be optimised, it was analysed to determine its relationship to input components. Soap pH, calcium chloride and a three-factor interaction between emulsifier, calcium chloride and kerosene were significant controlling variables (Table 7-3). The effect of soap pH was over 7 times larger than the other two effects; seen by the effect values in Table 7-3. This means final emulsion pH was predominantly controlled by soap pH and changes in the other components only slightly affected final pH. The slope of the soap pH line in the main effects plot (Figure 7-1) reveals the nature of the relationship between soap pH and emulsion pH; an increase in soap pH levels results in an increase in final pH. Increasing calcium chloride levels also slightly increased final pH. A soap pH of 2.0 was found to yield a final pH just below 2.5 and a soap pH of 2.5 returned a final pH of 3.1. The centre-point was half-way between these values at 2.80, indicating a strong linear relationship.

Table 7-3. Significance and Effects of Variables Controlling Emulsion Properties

Final pH			Viscosity*		Settlement		Break Index		Adhesion		Cohesion Value	
Code	Effect	P	Effect	P	Effect	P	Effect	P	Effect	P	Effect	P
a	-0.03	0.230	-21	0.397	-0.06	0.887	7	0.330	-7	0.251	-0.9	0.189
b	0.65	0.001	-43	0.155	0.25	0.592	9	0.225	16	0.071	-2.1	0.049
c	0.09	0.049	41	0.170	-0.70	0.221	-23	0.048	-5	0.359	0.6	0.312
d	0.02	0.353	47	0.136	-0.38	0.439	-58	0.008	8	0.224	0.5	0.375
ab	0.04	0.209	19	0.421	-0.11	0.802	1	0.908	10	0.149	0.0	0.991
ac	-0.01	0.627	3	0.886	0.00	0.994	4	0.554	-1	0.854	0.5	0.403
ad	0.05	0.144	14	0.534	-0.17	0.710	16	0.097	6	0.317	-0.6	0.312
bc	0.00	0.955	31	0.253	0.19	0.689	1	0.924	-3	0.595	0.1	0.846
bd	-0.04	0.173	57	0.098	-0.07	0.870	-6	0.382	-5	0.406	0.3	0.647
cd	0.04	0.209	116	0.027	0.32	0.505	10	0.191	4	0.461	0.5	0.375
abc	-0.06	0.058	-22	0.375	-0.08	0.866	8	0.280	9	0.181	-0.4	0.487
abd	0.08	0.084	-1	0.977	-0.03	0.944	-3	0.579	1	0.854	-0.3	0.589
acd	-0.10	0.040	-132	0.021	-0.17	0.714	5	0.475	0	0.951	-0.5	0.375
bcd	-0.07	0.074	21	0.397	0.29	0.541	-5	0.417	-6	0.317	-0.3	0.603
abcd	-0.05	0.144	-12	0.601	0.37	0.453	-1	0.809	2	0.674	0.2	0.663

*Viscosity at 5 rpm, 70°C, spindle #1.

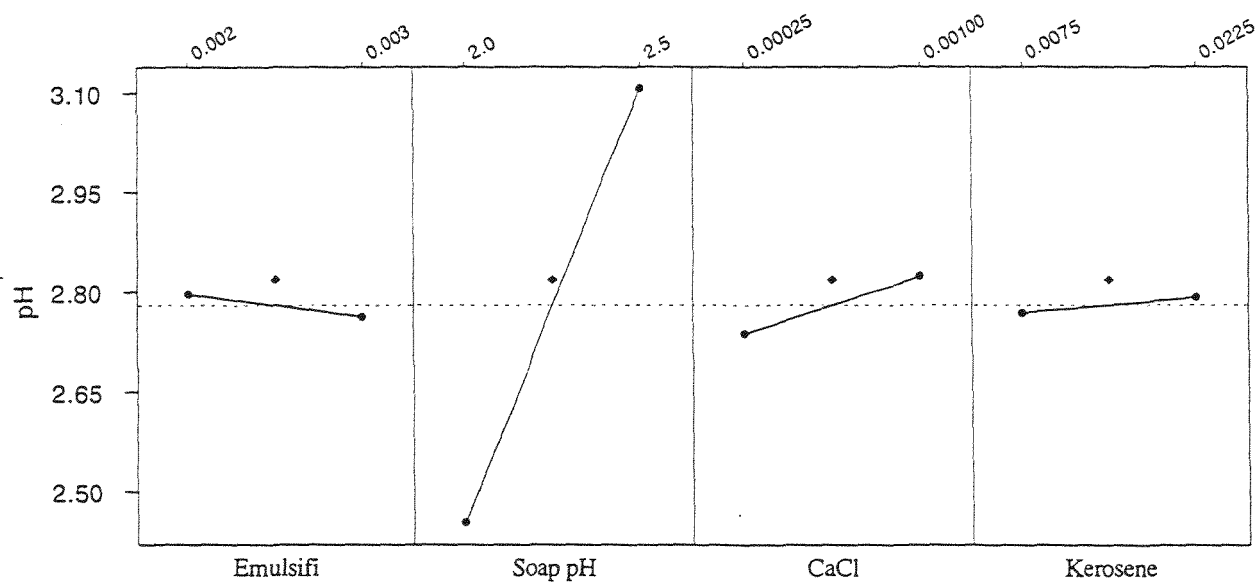
Key: a: Emulsifier b: Soap pH c: Calcium chloride d: Kerosene

■ Significant variable

Figure 7-1 Main Effects Plot for pH

• Centerpoint

Main Effects Plot For pH



7.4 PHYSICAL EMULSION PROPERTIES

Viscosity, settlement, break index, adhesion and cohesion were emulsion properties to be optimised. The significant effects highlighted in Table 7-3 controlling each property are discussed in turn to provide an understanding of how each can be optimised.

7.4.1 Viscosity

Viscosity was measured at three different shear rates (5,10 & 20 r.p.m) according to the method described in Chapter 4. A shear rate of 5 rpm was chosen for analysis as industry specifications use this value to specify viscosity and is therefore an appropriate benchmark.

No single component controlled viscosity, rather it was found to be controlled by a three-way interaction between emulsifier, calcium chloride and kerosene levels. The interaction had an effect value of -132 cP and a significance of 0.021. A cube plot was produced to understand the three-way effect, Figure 7-2. It was found that lower viscosities were achieved when only one of the three interacting variables were at their high levels. Therefore low viscosity could be achieved in three ways:

- Emulsifier at 0.3%, calcium chloride at 0.025%, kerosene at 0.75%.

Viscosity = 680cP (Figure 7-2)

- Emulsifier at 0.2%, calcium chloride at 0.1%, kerosene at 0.75%.

Viscosity = 630cP (Figure 7-2)

- Emulsifier at 0.2%, calcium chloride at 0.025%, kerosene at 2.25%

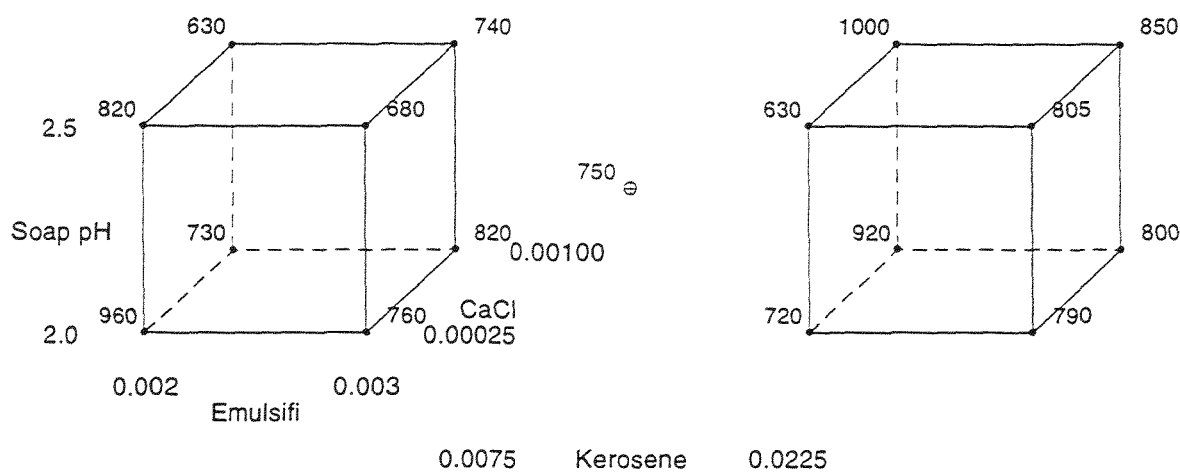
Viscosity = 630cP (Figure 7-2)

When two or more of these interacting components were at their high levels viscosity increased. This interaction was particularly strong in the case of kerosene and calcium chloride, which had an effect of +116 cP and significance of 0.027. If both kerosene and calcium chloride were increased from their low to high levels, viscosity moved from the lowest level observed (630 cP) to the highest level observed (1000 cP).

Figure 7-2 Cube Plot for Viscosity Interaction.

- ⊕ Centerpoint
- Factorial Point

Cube Plot- Means for Viscosity at 5 rpm.



7.4.2 Settlement

All factors for settlement had p values greater than 0.05. The higher p values are probably due to the level of variability inherent in the test, which reduced the ability to clearly detect significant changes in settlement. The settlement test is usually used with lower bitumen content emulsions which flow easily from the glass cylinders. High bitumen content emulsions have greater viscosity and do not flow easily out of the cylinders when sampling the top and bottom 50 mls for residual testing. The viscous nature of the emulsion is the likely cause of variability in the test.

Calcium chloride was identified as the most significant variable affecting settlement. Increasing calcium chloride levels had an effect of +0.70% and a p value of 0.224. Increasing the calcium chloride level from 0.025% to 0.1% caused greater settlement as seen by the slope of the calcium chloride line in the main effects plot, Figure 7-3. At both high and low levels of calcium chloride the average settlement was less than the specified 1% level. Settlement is a less critical factor to be optimised for high viscosity emulsions as particle motion and settlement rate are reduced (Whiteoak, 1990). Lower settlement was achieved at a calcium chloride level of 0.025%.

7.4.3 Break Index

Two factors were significant in influencing the break index: kerosene and calcium chloride. Increasing kerosene levels had an effect value of -58% on break index; over twice the effect of increasing calcium chloride levels which had an effect value of -23%. These effects are seen in the main effects plot, Figure 7-4. To achieve a lower break index, kerosene and calcium chloride were better at higher levels of 2.25% and 0.1% respectively. This can be seen by the results in Table 7-2; run 'cd' with calcium chloride and kerosene at high levels had the lowest break index of 160%. Runs without these two components at their high levels had a high break index, such as run 'a' with a break index of 257%.

Figure 7-3 Main Effects Plot for Settlement.

• Centerpoint

Main Effects Plot For Settlement

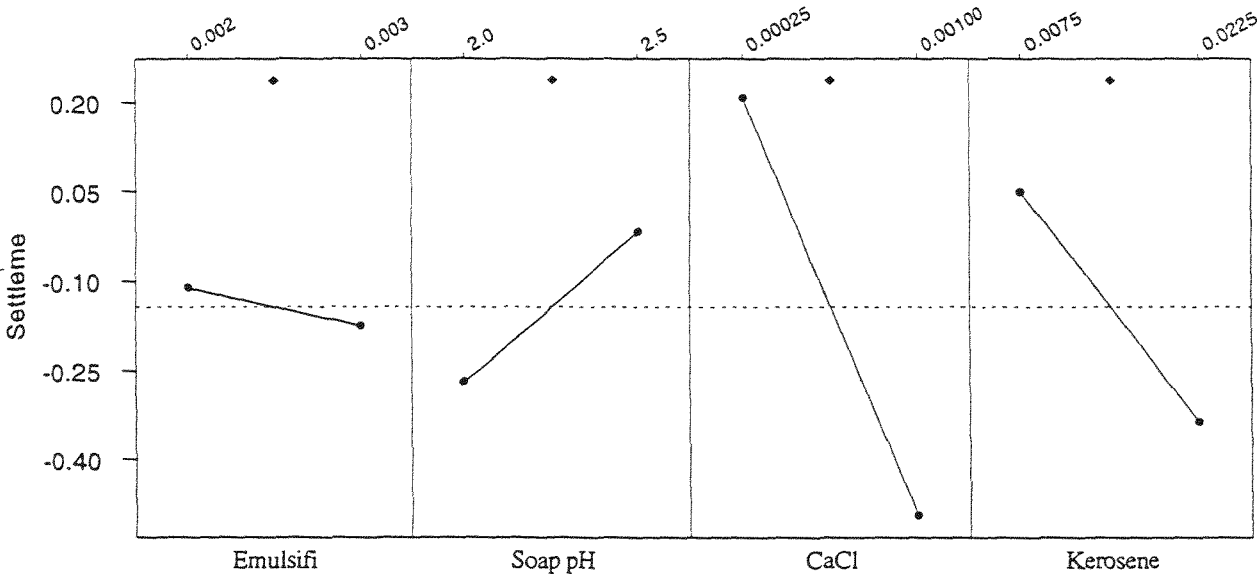
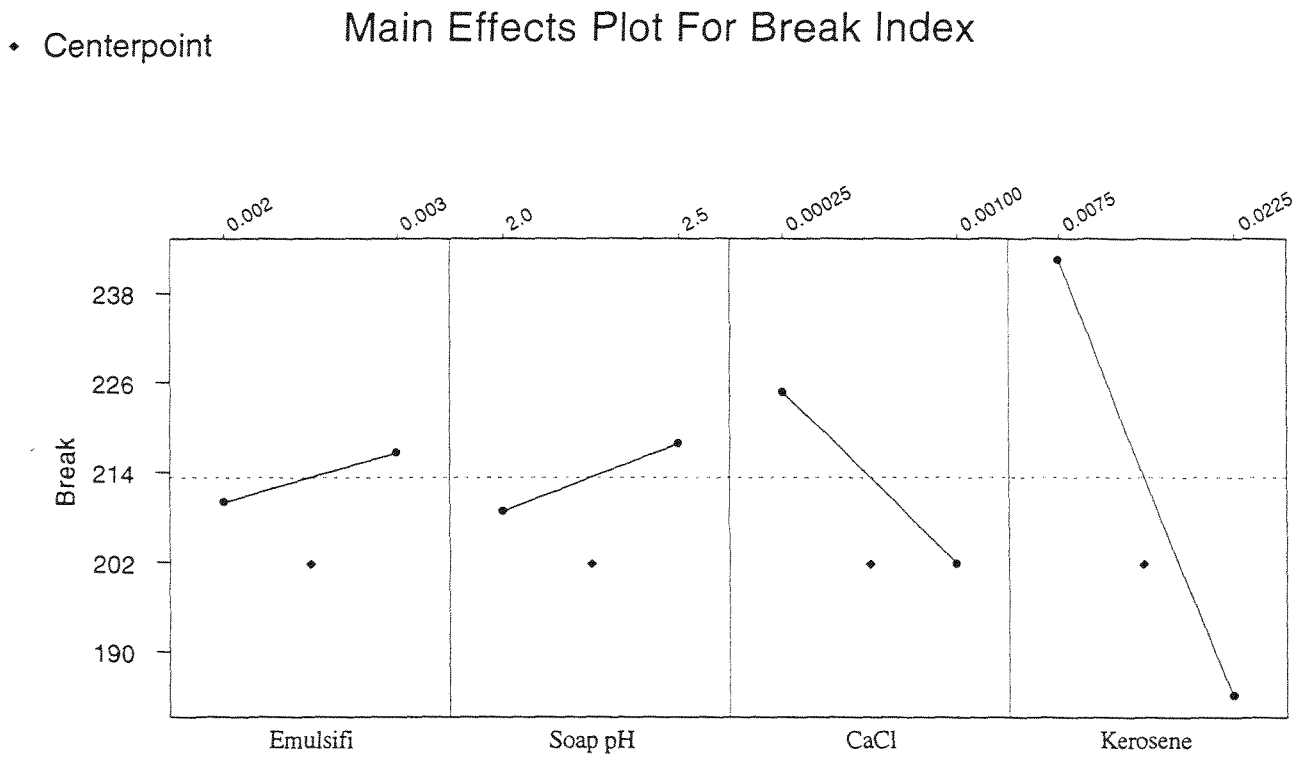


Figure 7-4 Main Effects Plot for Break Index.



7.4.4 Adhesion

Soap pH was the most significant effect for adhesion. Soap pH had an effect value of +16% and a p value of 0.071. The p value is slightly over the desired 0.05 level. The subjective nature of evaluating stone coating in the adhesion test is one likely factor increasing the uncertainty. The level of uncertainty is less than 0.1 still indicating an effect of significance. Examining the effect of soap pH on adhesion in the main effects plot, Figure 7-6 revealed greater adhesion was achieved at a soap pH of 2.5 compared to 2.0. This effect can be seen from the results in Table 7-2 also. For example run ‘b’ with a soap pH of 2.0 had lower adhesion at 65%, whereas run ‘d’ with a soap pH of 2.5 had an adhesion of 80%.

7.4.5 Cohesion

Cohesion was measured at intervals of 1, 2 and 3 hours. A single standardised value that considered the relationship between all three points was used for analysis. To obtain a standardised value a straight line was fitted to the three values with the intercept being used as the basis for analysis; the method used is illustrated in Figure 7-5. Individual cohesion figures recorded at the hourly intervals are held in Appendix 7.1. The cohesion values obtained using the standardising procedure are recorded in Table 7-2.

Figure 7-5. Graphical Method to Determine Cohesion Values

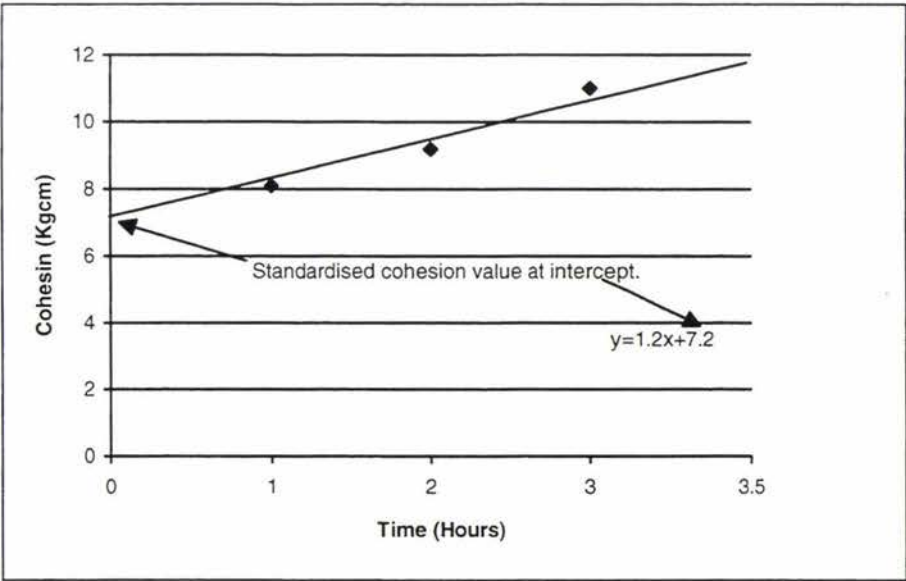
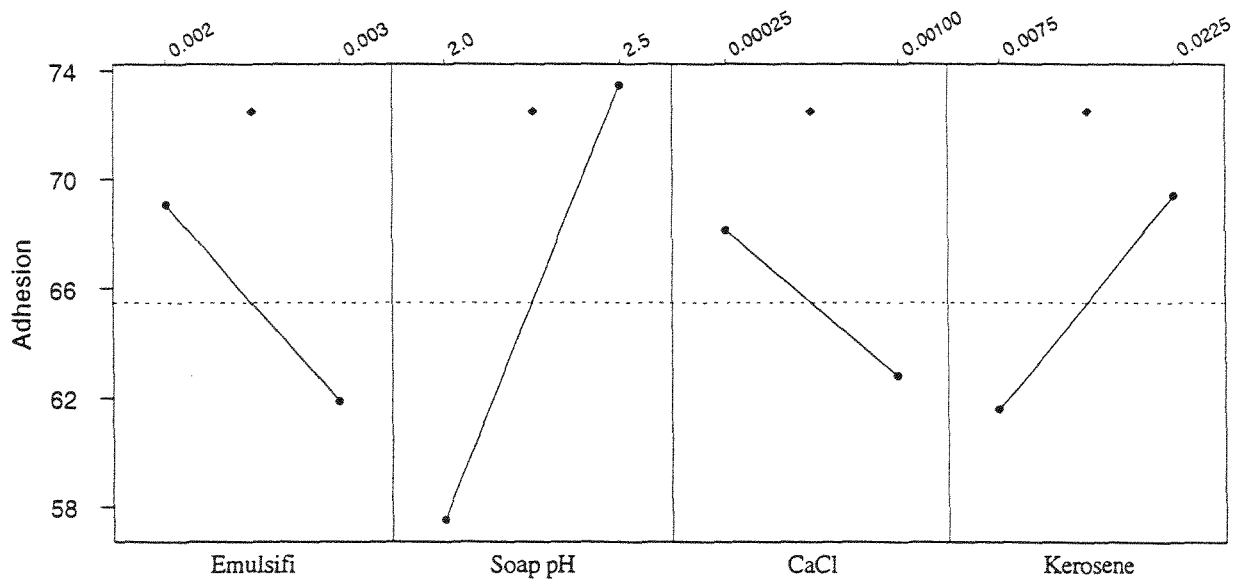


Figure 7-6 Main Effects Plot for Adhesion.

• Centerpoint

Main Effects Plot For Adhesion



Analysis of the cohesion values revealed soap pH as the significant controlling variable. Soap pH had an effect value of -2.1 with a p value of 0.049. Lower pH values increased cohesion as seen by the slope of the soap pH line in the main effects plot, Figure 7-7. Greater cohesion was achieved with a soap pH of 2.0. This relationship can also be seen in the results from Table 7-2; for example run 'a' with soap pH of 2.5 had a cohesion value of 5.8 kgcm whereas run 'b' with soap pH at 2.0 had a cohesion value of 9.9 kgcm.

7.4.6 Determining an Optimum Formulation Area

Table 7-4 summarises the significant effects controlling each of the five properties discussed and Figure 7-8 displays how these emulsion properties relate to field performance. Information relating to the control of the emulsion properties was used to identify a formulation area which had the most preferred set of properties. The identified formulation area could then be further investigated in the optimisation phase.

Table 7-4. Summary of Significant Effects Controlling Emulsion Properties

Response Property	Significant Effects	Effect	p Value
Viscosity	Emulsifier/Calcium chloride/Kerosene (* ACD)	-132 cP	0.021
	Calcium chloride/Kerosene (CD)	+116 cP	0.027
Settlement	Calcium chloride (C)	-0.70 %	0.221
Break Index	Kerosene (D)	-58 %	0.008
	Calcium chloride (C)	-23 %	0.048
Adhesion	Soap pH (B)	+16 %	0.071
Cohesion	Soap pH (B)	-2.1 kgcm	0.049

Note: (Design coding in brackets)
*ACD represents an interaction between components A, C and D; not single components themselves.

Figure 7-7 Main Effects Plot For Cohesion.

• Centerpoint

Main Effects Plot For Cohesion

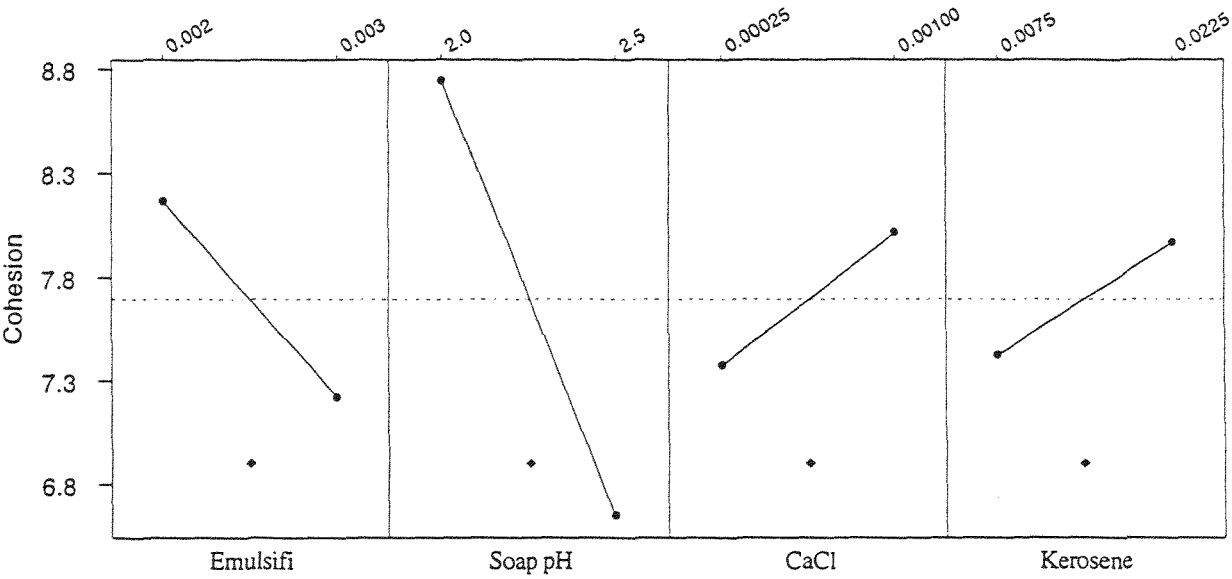
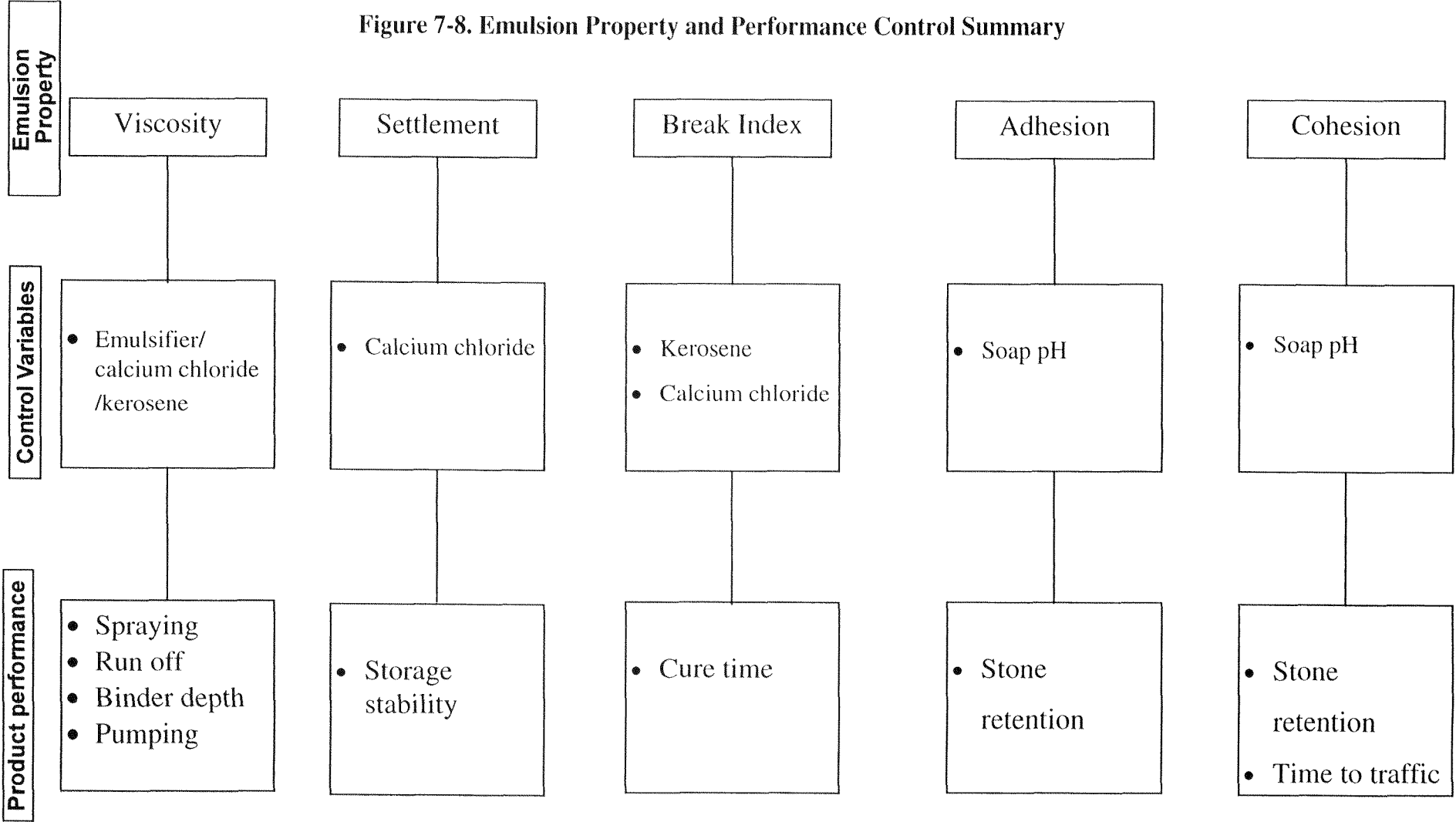


Figure 7-8. Emulsion Property and Performance Control Summary



Determining the optimum formulation area used the following rationale to consider both the means to control individual properties and the effect on other properties.

Viscosity was controlled by the interaction between emulsifier, kerosene and calcium chloride. Low viscosity could be achieved in three ways as discussed in section 7.4.1. The most preferable of these three areas was where emulsifier was at 0.2%, calcium chloride at 0.025% and kerosene at 2.25%. With components at these levels settlement and break index would also be optimised.

Table 7-4 shows settlement was controlled by calcium chloride levels. Increasing calcium chloride levels would increase settlement and increase viscosity, particularly if kerosene was present at its higher level. Calcium chloride was therefore better kept at 0.025% to minimise viscosity and settlement.

Table 7-4 shows break index is controlled by kerosene and calcium chloride levels. To achieve a minimum break index, both kerosene and calcium chloride were to be at their higher levels. However increasing both of these components would dramatically increase viscosity due to the strong interaction between these two components. By keeping kerosene at its higher level of 2.25% and calcium chloride at its lower level of 0.025%, break index could be lowered to around 200% while achieving minimum viscosity. The break index was higher at 235% and 257% in the other two areas at which viscosity was low. Viscosity, settlement and break index were therefore better in the formulation area where emulsifier was at 0.2%, calcium chloride at 0.025% and kerosene at 2.25%.

Table 7-4 shows soap pH was the significant variable controlling both adhesion and cohesion, however the relationship was in opposing directions. Increased pH favoured adhesion but reduced cohesion; a trade-off therefore had to be made. This was done in favour of higher adhesion as more chips seals fail due to poor adhesion rather than cohesion (Holleran, 1995). Adhesion is also a stronger bonding force compared to cohesion which would give greater stone retention. High initial cohesion is also more important under poor weather conditions where cure is slower and cohesive strength does not build up as rapidly; under more standard sealing conditions initial cohesive

strength is less important (Holleran, 1989). This meant a soap pH of 2.5 was more favourable to give higher adhesion in the order of 80%. With emulsifier, calcium chloride and kerosene set at levels to optimise other properties, a soap pH of 2.5 resulted in cohesion value of 7.4 kgcm. Cohesion values of this magnitude were in the middle of the range of cohesion values observed during the experimental runs.

In order for viscosity, break index and settlement to be minimised, emulsifier was to be at 0.2%, calcium chloride at 0.025%and kerosene at its high level of 2.25%. Soap pH was better at 2.5 to maximise adhesion. Components at these levels corresponded to the run coded ‘d’ in Table 7-2. This run identified one point in the experimental design which was used to mark out an optimum formulation area..

Three other runs had a break index less than 250%, adhesion greater than 75% and settlement under 1%, meeting the criteria of the technical design specification. These were runs ‘ad’, ‘bcd’ and the centre-point runs. An excerpt of results from Table 7-2 for these runs can be seen in Table 7-5. Run ‘bcd’ had high viscosity and run ‘ad’ had high viscosity and low cohesion. Of these three runs the centre-point runs had the lowest viscosity of 750cP, compared to 805 cP and 920 cP for the other runs. Cohesion was similar to the other optimal run ‘d’. A large variability in settlement was again evident.

Table 7-5 Excerpt of Results from Table 7-2: Optimum Experimental Runs

Code	Residue (%)	pH (Units)	Viscosity (cP)	Settle-ment (%)	Break Index (%)	Adhesion (%)	Cohesion Value (kgcm)
d	76.66	3.04	630	-0.04	192	80	7.4
ad	76.21	3.10	805	-0.55	197	80	5.7
bcd	76.09	2.63	920	-0.58	165	80	10.2
CP ₁	76.40	2.83	750	0.99	200	75	6.3
CP ₂	76.28	2.81	750	-0.36	205	75	7.5

The centre-point runs were selected to establish the second point to mark out an optimum formulation area.

7.5 OPTIMISATION

To gain more information about the behaviour of the emulsion in the identified optimum area another set of experiments were designed in which smaller changes to components were made in order to locate an optimum.

The preferred formulation area lay between the centre-point run and the corner-point coded 'd' in the experimental design. The centre-point was coded 0,0,0,0 and run 'd' was coded -1,-1,-1,+1 in the design matrix. Three optimising runs were designed to explore the area. Using the design matrix notation the following optimising runs were planned.

Table 7-6. Component Levels For Optimisation Experimental Design

Run #	Code	Emulsifier	Soap pH	CaCl ₂	Kerosene
1.	0,0,0,0 (Centre point)	0.25%	2.25	0.0625%	1.50
2.	0,0,0,+1	0.25%	2.25	0.0625%	2.25%
3.	-1/2,-1/2,-1/2,+1	0.225%	2.38	0.0438%	2.25%
4.	-1,-1,-1,0	0.20%	2.50	0.0250%	1.50%
5.	-1,-1,-1,+1 ('d')	0.20%	2.50	0.0250%	2.25%

7.5.1 Results

Table 7-7 contains the results of the optimising runs.

Table 7-7. Emulsion Properties from Optimising Experimental Design

Run #	Code	Visc [*] (cP)	Settlmt. (%)	Break (%)	Adhesn. (%)	Cohsn. (kgcm)	pH (units)	Resd. (%)
1.	0000	750	0.32	202	75	6.3	2.82	76.34
2.	000+1	740	-0.10	170	80	6.8	2.89	75.67
3.	-1/2 -1/2 -1/2 +1	650	0.48	190	90	7.2	2.84	75.95
4.	-1-1-1 0	620	0.84	219	95	6.6	2.76	76.59
5.	-1-1-1+1	630	-0.04	192	80	7.4	3.04	76.66

*Viscosity @5 rpm, 70°C, spindle 1.

Runs 3 and 4 were identified as optimum runs within the area. Run 4 had minimum viscosity at 620 cP and maximum adhesion of 95% which were the most important factors to optimise; break index and settlement were within the appropriate specification limits of 1% and $200 \pm 50\%$ respectively. Cohesion was at a value in the middle of values observed for all runs. The area marked by run 4 was considered to be the final optimised formulation and had components at the following levels.

Component Levels For Final Optimised Formulation.

Emulsifier	0.2%
Soap pH	2.5
Calcium chloride	0.025%
Kerosene	1.5%
Bitumen	75%
Water	to 100%

7.5.2 Additional Investigations

Investigations were conducted with the final formulation to consider the following issues:

- Adhesion with various aggregates.
- Susceptibility of the product with changes in bitumen and soap temperatures.
- Effect of mechanical action.

7.5.2.1 Adhesion with Various Aggregates

Adhesion tests were carried out using aggregates from a number of quarries to determine variation in adhesion with aggregate type. The following results were observed:

Table 7-8 Adhesion of Optimum Formulation with Various Aggregates

Aggregate Source	Adhesion
Toatoaroa	95%
Fraser	85%
Bulls	75%
Child metal	45%
Te Matai	30%

Bulls, Fraser and Toatoaroa chip had acceptable adhesion while Te Matai and Child chip which had similar river sources were comparatively low. Tests were conducted to determine the cause of the problem and it was found that chip mineralogy and composition were the most likely source of the poor adhesion. The formulation was optimised using Toatoaroa chip, other aggregates can be tested prior to sealing to determine their compatibility.

Pre-coating the chip with emulsion was suggested to solve any suspected adhesion problems.

7.5.2.2 Temperature Susceptibility

The susceptibility of the emulsion to changes in production temperatures were investigated to determine temperature ranges in which acceptable emulsion could be produced. Bitumen temperatures of 130 °C, 140°C, 145 °C, 150 °C and 155 °C were investigated while the soap temperature was held constant at 36°C. Changes in soap temperature were also investigated; bitumen temperature was held at 140°C, while soap temperatures of 33°C, 36°C, 40°C and 45°C were investigated.

Adhesion and break index remained unchanged with temperature increases; settlement showed no clear trend while viscosity and cohesion generally increased. It was found that bitumen temperature could range between 130-140°C and soap temperature could range between 33-40°C and still produce product meeting the design specification. It was found the most preferable bitumen temperature and soap temperatures were 130°C and 33°C respectively to achieve a preferred exit temperature of 86°C. The maximum permissible exit temperature was found to be 91-92°C; beyond this emulsion properties were found to degrade. The product was considered robust to changes in production temperatures within the identified limits.

7.5.2.3 Effect of mechanical action

Excessive mechanical action and shearing from agitation and pumping can cause emulsified bitumen particles to come into close proximity and coalesce. Two tests for mechanical action were carried out. Firstly a stirrer was used to simulate agitation and secondly a pump was used to simulate shearing. Break index was used to judge the emulsion's stability before and after applying mechanical action. No change was observed in break index after 10 and 30 minutes of mechanical action.

7.5.3 Discussion and Recommendations

The design work used a definite process and a distinct design technique to arrive at the optimised formulation. A discussion of issues relating to the process and techniques used provides a basis for future improvement.

Product testing was found to be one of the most critical areas in the design process, as conclusions from design work are based on the results of the tests. It was found to be important to identify and establish the required tests early, to gain an understanding of the limitations and accuracy of the tests. Greater accuracy and reduced variability of testing would increase the ability to detect the effect of formulation changes on emulsion properties, providing clearer results in experimental design. Measures to reduce variability in some of the tests may need to be investigated, such as: controlling cure conditions for cohesion testing, more accurate assessment of adhesion coating and the appropriateness of settlement testing for high viscosity emulsions.

Establishing the correct screening criteria during initial design was also found to be important to identify preferred raw materials. When screening the emulsifiers basically two tests were used; viscosity and break index (other tests were not fully established). During later design work it became apparent that the adhesion of Dinoram SL was poor with local chip, if adhesion had been able to be used as a screening criteria this problem would have been evident earlier, therefore it is important to establish key screening criteria early. A balance must also be found between the number of tests conducted and the speed of screening. Identifying the necessary key screening criteria will allow rapid identification of preferred concepts and gaining the most important information.

Reducing the number of different batches of raw materials, particularly bitumen would also improve design work. Due to limited storage capacity several unavoidable changes in raw material batches were encountered throughout the design work. These changes may have contributed to variation in experiments and made comparisons between runs more difficult. Ambient conditions were also changing during the course of the work, which likely influenced cure conditions.

Any uncontrollable variables need to either be eliminated in future or recorded so allowances can be made for the variation.

By increasing the accuracy of testing and reducing the variation in raw materials greater benefit can be obtained from experimental designs as accurate regression equations can be generated. Mathematical regression equations can be used to describe in a succinct manner the relationship between input component levels and out responses in properties

from experimental designs. These equations can be used to more easily to identify an optimum formulation through the use of liner programming optimisation techniques.

The full factorial experimental design used in the study required over two months full-time work to execute, this would make the design method unfeasible for use by Higgins under normal circumstances. Instead of conducting full factorial designs, fractional factorial designs could be used. In these designs higher order interactions are aliased with single components, reducing the number of runs investigated. Less information is gained from these designs, however the more important main effects and lower order interactions are investigated which are generally found to be the significant controlling variables; this was consistent with the results from the experimental design conducted. It was found that the significant controlling variables were mainly single factors or two factor interactions; there was only one significant three-way interaction and no significant four-way interactions. As lower order interactions generally control a system, half factorial designs could be run in which fewer runs would be necessary to yield the more valuable information on the main effects and lower order interactions. A full factorial was run in this study to gain a thorough understanding of all interactions within the chosen formulation area.

7.6 CONCLUSION

Dinoram SL was identified as the preferred emulsifier in raw material screening during initial design. Detailed design used a full factorial experimental design with four factors at two levels to gain an understanding of how the formulation system was controlled and to identify a preferred formulation area. Viscosity was controlled by a three-way interaction between emulsifier, calcium chloride and kerosene levels. Settlement was controlled by calcium chloride levels; break index by kerosene and calcium chloride levels. Adhesion and cohesion were both controlled by soap pH levels. Minimum viscosity was achieved when kerosene was at 2.25% with emulsifier and calcium chloride at 0.2% and 0.025% respectively. Settlement and break index were also minimised with kerosene and calcium chloride at these levels. Adhesion and cohesion were both controlled by soap pH levels, however the relationship between these two properties was in opposing directions and a trade off in favour of greater adhesion was made to give a preferred soap pH of 2.5.

Components at the levels described above identified one point which was used to mark out an optimum area. The centre-point runs with components at there mid-levels gave similar optimum properties and was the second point used to identified an optimum area. Optimisation explored the identified area with smaller changes in component levels to locate a final optimum formulation. The final optimised formulation had the following composition and processing parameters.

<i>Formulation Components</i>		<i>Processing Variables</i>	
Dinoram SL	0.2%	Bitumen temperature	
130°C			
Soap pH	2.5	Soap temperature	33°C
Calcium chloride	0.025%	Mill gap	0.085mm
Bitumen	75%		
Kerosene	1.5%		
Water	to 100%		

The optimum formulation was ready to move to Mid and Full-scale Development stages.

8. MID-SCALE AND FULL-SCALE DEVELOPMENT

8.1 INTRODUCTION

Scale-up took the optimised formulation developed during the Laboratory Development stage and attempted to demonstrate performance on actual production and application equipment. Scale-up had three distinct stages: small-scale, mid-scale and full-scale development. These stages would progressively indicate the validity of laboratory work.

8.2 SMALL SCALE EVALUATION

The scale-up process began by determining the performance of the optimised formulation developed in Chapter 7 using small 20 x 20 cm test patches laid in the laboratory yard. These patches were used to assess field performance variables unable to be evaluated in the laboratory. These variables included: run off, break time, wetting ability, effect of rolling, traffic and weather.

8.2.1 Method

A four litre batch of emulsion was made using the optimised formulation which had components at the following levels: Dinoram SL 0.2%, calcium chloride 0.025%, Soap pH 2.5 and kerosene at 1.5%. Bitumen and soap temperatures were at 130°C and 33°C respectively.

An emulsion application rate of 2.66 L/m² was used to give a residual binder application rate of 2.0 L/ m²; reflecting the application rate to be used in later scale-up. The emulsion was applied by hand with a plastic transfer pipette at 67.5°C. Pavement temperature was 30°C; air temperature was 20°C with light wind and slight cloud cover. Grade 3 chips from Fraser's quarry in Napier were then spread over the emulsion; 550g of aggregate was used to give a total and uniform coverage. The same type of aggregate would also be used in later scale-up. The 400 cm² area was then rolled 3 times in perpendicular directions with a 20 kg roller to form a tight matrix. The patches were inspected after 1, 2, 3 and 24 hours to assess the cure, adhesion and likely field performance.

8.2.2 Small-Scale Trial Results

Assessment of the patches was limited to observations and comments, as one of the difficulties facing the industry is the lack of simple quantitative tests to evaluate the field performance of chip seals.

- There was no sign of run off.
- Chips were applied two minutes after emulsion application with good wetting observed.
- Inspection of the patch after 1, 2 and 3 hours revealed sufficient strength had developed after 3 hours to drive on. A car was used to test this by braking and accelerating on the patch; no damage was observed.
- Adhesion of the chip was excellent and was evaluated by pulling chips from the binder and inspecting them for bitumen coating; a lack of coating indicating poor adhesion. All chips assessed were well coated.
- Inspection of the patch after 24 hours revealed a cured and sound matrix resistant to scuffing.
- The patch was still holding well, four months later.

Although these tests lacked a quantitative means of evaluation they provided evidence to further validate laboratory work and proceed with scale-up.

8.3 MID-SCALE DEVELOPMENT

The Mid-scale Development stage was planned to follow the standard product development process outlined in Chapter 4. This stage was to prove the emulsion's potential on a medium scale to avoid costly errors by moving directly from small scale to large scale development. However alterations to the laboratory mill and development of specialised application equipment was necessary for this to take place. Implementation of this equipment would have taken longer to achieve than the course of study would allow, therefore planning for the implementation of this facility was carried out instead of the actual mid-scale trials and is discussed later in Section 8.5.2.

8.4 FULL-SCALE FIELD TRIALS

Planning of the Full-scale field trial was a key activity to gain the most benefit from the work. A template field trial plan was generated to assist the organisation, execution and recording of field trials for emulsion spray sealing at Higgins. The trials were divided

into two distinct sections: production and application. Production consisted of preparation of soap solution and milling while application consisted of spraying, chip application and rolling. The required activities, resources, testing and contingency plans for each section were laid out in the template plan. The field trial plan can be seen in Appendix 8.1 while data recording templates developed can be seen in Appendix 8.2.

Two full-scale field trials were conducted in Napier with emulsion being produced on the larger mill at Belspray Asphalts Limited. Belspray is a subsidiary company owned by Higgins which produces emulsion for Higgins. The first trial involved emulsion production and application. The second trial consisted of emulsion production only. Emulsion production of both trials is firstly addressed, followed by the results and discussion of emulsion application.

8.4.1 Emulsion Production

The emulsion was produced according to the process described in Chapter 3, Section 3.4, by preparing a soap solution which is milled with bitumen and kerosene through a colloid mill in appropriate proportions.

8.4.1.1 Soap Preparation Procedure

A similar procedure used in the laboratory to prepare the soap solution was used in Napier. A total of 500 litres of dilute soap solution was made for both trials. The concentrate solution was made with 180 litres of water at 45 °C, calcium chloride was added with the tank under constant agitation. Two thirds of the hydrochloric acid was then added, followed by the emulsifier. The remaining 320 litres of water was added to achieve a dilute soap temperature of 34°C. pH of the dilute soap solution was adjusted with the remaining acid to a pH of 2.45, which was within the same accuracy used in laboratory preparation.

8.4.1.2 Milling

The soap solution, bitumen and kerosene were separately pumped to the mill and simultaneously milled. In the laboratory setting, kerosene was pre-blended into the bitumen as opposed to being co-milled at Belspray. The difference in processing was due to the differing set-ups and scale between laboratory and production facilities. Bitumen was unable to be easily and consistently pre-blended at Belspray particularly

for smaller trial quantities, making co-milling a more viable option. In the laboratory setting, kerosene is more reliably added by pre-blending into the bitumen phase prior to milling due to difficulties with the pump used for co-milling. The pump is also unable to accurately deliver kerosene at the relatively low flowrates. The effect of this difference in processing needs to be investigated to determine its affect on emulsion properties; the differences should be rectified if the effect is found to be significant.

Bitumen content was initially set at 74% on the mill's gauges. After production of 550 litres of emulsion the mill was stopped while a sample was tested to assess bitumen content before further production. A residual bitumen content of 71.76% was recorded. Bitumen content was then set at 77% on the mill's gauges and another sample taken after 900 litres of production. A residual of 73.26% was recorded at that point; still under the target of 75%. A total of 1,051 litres of emulsion was produced. The production of lower residual emulsion was due to the uncalibrated and non-linear nature of the mill's gauges. Bitumen temperature was also not calibrated, with bitumen temperature set at 123°C as gauges read 3-4°C lower than the actual temperature. From information gained in trial 1, correct mill settings were deduced. A sample was taken after 900 litres of emulsion production in trial 2 and a residual of 75.07% was achieved.

During emulsion production trials, valuable information on the required mill settings had been determined and the scale-up of the soap solution could be accurately prepared to reflect laboratory preparation.

8.4.2 Comparisons Between Laboratory and Full-scale Production

Comparisons were made between the emulsion produced on the full-scale mill and emulsion made on the laboratory mill to establish likely trends in scale-up. Soap solution was sampled from the first trial conducted at Belspray Asphalts in Napier and bought back to the Palmerston North laboratory. The soap solution was used to produce a similar emulsion on the laboratory mill. The emulsion produced on the Belspray mill was compared against the emulsion produced on the laboratory mill and the optimum laboratory emulsion. Table 8-1 shows the comparison between the three emulsions.

Table 8-1. Comparison Between Laboratory and Full-scale Emulsion Properties

Mill	Soap Solution	Residue (%)	pH (Units)	Viscosity* (cP)	Break (%)	Adhesion (%)	Cohesion Value (kgcm)	Cohesion @ 3 hrs. (kgcm)
Belspray	Belspray	73.26 %	3.33	1000 cP	99 %	95 %	6.6	17.2
Lab.	Belspray	74.90 %	3.17	940 cP	180%	95%	4.1	12.8
Lab.	Lab.	76.59 %	2.76	620 cP	219%	95%	6.6	11.6

*Viscosity @ 5 rpm, 70°C, spindle 1.

The low residual emulsion produced on the Belspray mill meant significant differences in residue existed between the emulsions made on the two mills and direct comparisons were unable to be made. A further difference which made comparisons more difficult was the difference in the method of kerosene addition. These differences meant only likely trends could be established when moving from the laboratory mill to the mill at Belspray in Napier.

The pH values were similar between the first two emulsions, this was expected as they were produced with the same soap solution. The final pH of these emulsions were close to the expected pH of 3.10-3.20 set in the technical specification which was based on the experimental runs in the laboratory. The pH of the optimum laboratory formulation at 2.76 was lower than expected as other emulsions produced with a soap pH of 2.5 yielded a final pH in the region of 3.1-3.2. From the design work an expected decrease in adhesion and an increase in cohesion would be expected with lower pH, this was not observed in the results in Table 8-1. A different batch of the same grade bitumen was used to produce the optimum laboratory emulsion and is a possible reason for the difference. Differences in bitumen composition and acidity were unable to be tested and its effects are unknown.

The most significant difference between emulsions produced on the different mills was the break index. The break index of emulsion produced on the Belspray mill was almost half that of laboratory formulations: 99% compared to 180%. During experimental design work it was found that kerosene was the most significant variable influencing break index. A possible reason for the reduction in break index of Belspray emulsions is the co-milling of kerosene rather than pre-blending it as in the laboratory. This difference in processing may possibly reduce the break index of Belspray emulsions.

Investigations into this are required to establish the significance of the difference. Rectifying the differences in processing will allow more accurate comparisons to be made between mills.

Viscosity increased when moving from the laboratory mill to the larger Belspray mill. The emulsion from Belspray had a viscosity of 1000cP and a residual of 73.26%, with increases in residual to 75% further increases in viscosity can be expected. The production mill has higher flow rates which increase viscosity, particularly with higher content emulsions (Whiteoak, 1993). The larger mill also has a higher shear rate producing a finer particle size which will also increase viscosity (Durand and Poirier, 1996). The effect of co-milling versus pre-blending kerosene may also influence viscosity.

Adhesion was the same for both emulsions at 95%. Emulsion produced on the Belspray mill yielded higher cohesion. The cohesion value for Belspray emulsion was 6.6 kgcm compared to 4.1 kgcm. Cohesion after three hours for Belspray emulsion was also significantly higher than any other emulsion produced in the laboratory; 17.2 kgcm compared to 11.6 kgcm and 12.8 kgcm for laboratory emulsions. Given the cure conditions were similar and the exact same soap solution was used in the first two emulsions in Table 8-1, the increase in cohesion between laboratory and Belspray emulsion can most likely to be attributed to the larger mill and differences in kerosene addition. A possible theory as to the marked difference in cohesion is that when kerosene is pre-blended the kerosene is able to become more intimately mixed with the bitumen at a temperature around 130°C, reducing the cohesive strength between bitumen constituents. When kerosene is co-milled it is added at ambient temperature just prior to milling and does not have time to interact with the bitumen, remaining largely as discrete droplets in the aqueous phase and not reducing the cohesive strength to the same extent.

Initial results indicated the following trends when moving from the laboratory mill to the larger Belspray mill:

- Viscosity increases
- Break index decreases
- Adhesion remains unchanged
- Cohesion increases

Future work will need to be carried out to provide more of an accurate assessment of correlations between laboratory and Belspray emulsion production.

8.4.3 Emulsion Application

Once produced, the emulsion was pumped into a spray tanker ready for application. Emulsion application was carried out in the first trial only and was applied on an industrial access way adjacent to the production facility. The produced emulsion was sprayed in three sections at both a high and low application rates to gauge performance at different application rates.

Section 1: 2.66 L/m²

Section 2: 2.20 L/m²

Section 3: 2.66 L/m²

Fraser’s grade 3 chip was then spread over the emulsion and rolled 6 times with a pneumatic tyred roller.

8.4.3.1 Emulsion Application Results: Trial 1.

Emulsion spraying encountered excessive run-off at both high and low application rates and can be seen in Figure 8-1. Run-off is caused by low emulsion viscosity. However when the emulsion was sampled during production it did not have a low viscosity. A sample of emulsion was taken from the spray-bar and compared against the sample taken from the mill after 900 litres of production. The results can be seen in Table 8-2.

Table 8-2 Comparison Between Produced and Applied Emulsion: Field Trial 1

Sample	Residue	Viscosity 5 rpm (cP)	Emulsion Temp. (°C)
900L	73.26 %	1,000	70
Sprayer	64.81 %	60	35

Residual tests revealed a drop of 8.45% had occurred from production to application. This drop caused viscosity to decrease from 1000 cP to 60 cP and resulted in the excessive run-off. Emulsion sampled from the spray-bar had cooled and could not be tested at the standard temperature of 70°C, if tested at 70°C the viscosity would be slower still.

Figure 8-1. Excessive Run-off During Field Trial 1.



The decrease in bitumen content was initially suspected to have resulted from dilution by one or more of the following ways:

- Water in the spray tanker prior to emulsion addition.
- Wash water entering from the mill's cleaning cycle.
- Water in pipe-work.

An accurate assessment of the field performance of the final pavement with respect to variables such as adhesion and cohesion could not be carried out as the drop in residual would dramatically affect these results.

8.4.3.2 Emulsion Application Results: Trial 2.

The second trial was conducted to eliminate possible causes of suspected dilution encountered during the first trial. The spray tanker was checked for water, soap solution was monitored more closely and other possible entry points for water were eliminated. Production was not stopped at the 500 litre mark during production in the second trial to avoid wash water from the mills cleaning cycle entering the system during stoppages. A sample was taken after 900 litres of production and from the spray-bar, these samples were tested before proceeding with application.

It was apparent that the same drop in residual and viscosity had occurred between production and exiting the spray-bar. The sample taken after 900 litres of emulsion production had a residual of 75.07%; the sample taken from the spray bar had a significantly lower residual of 67.17%. The drop in residual again resulted in a large drop in viscosity and therefore spraying and chip application were abandoned.

Additional water had been eliminated as the cause for the drop in residual, therefore the decrease had resulted by some other means. The following discussion puts forward possible theories for the observed reduction in residual.

8.5 DISCUSSION

8.5.1 Emulsion Stability

The drop in residual from the trials is most likely to be due to emulsion instability. The emulsion was produced at a bitumen content between 73-75% as confirmed by samples exiting the mill and cross checked by total volumes of bitumen and soap solution used. The emulsion was produced at 86°C and pumped into the sprayer which was at ambient temperature, subjecting the emulsion to thermal shock and rapid cooling. The emulsion was also subjected to shearing through the action of pumping and circulation. It is suggested that this treatment caused bitumen particles to come into closer proximity to each other resulting in coalescence of the emulsified bitumen. Once a proportion of the bitumen coalesced out of the emulsion the system would have become more stable at a bitumen content of 65-67%.

Sieve tests were conducted in which emulsion was passed through a 850 µm sieve to determine the number of large bitumen particles; large coalesced particles are a sign of instability. These tests provided evidence that the emulsion was possibly not stable during application. The sieve test carried out on emulsion sampled from the mill, which had not been subjected to shearing or rapid cooling had only a few large bitumen particles retained on the sieve; indicating a stable emulsion. The emulsion exiting the sprayer which had undergone shearing and a rapid temperature drop had an increased number of large bitumen globules retained on the sieve. This would suggest that the emulsion was stable when initially produced but it was unable to tolerate the conditions

to which it was subjected in the sprayer. Under such conditions coalescence occurred, increasing the number of large bitumen particles which settled out, which were observed in the sieve test. The reduction in bitumen held in the emulsion consequently reduced viscosity. If the emulsion was unstable bitumen would settle out leaving a bitumen sludge on the bottom of the spray tanker. The spray tanker driver commented on the fact that there was more bitumen sludge than usual left in the tanker after the first trial.

However some doubt still exists over this theory. At some point during spraying, emulsion would have become very thick as the coalesced bitumen sludge would have begun to be sprayed. This was also not observed; emulsion remained thin during the entire spraying trial with run off a continued problem. The spray tanker's dipstick was checked for evidence of bitumen sludge on the bottom of the tank after the second trial as evidence of separation and instability. No significant sludge was observed on the bottom or sides of the tanker.

The effect of rapid cooling on emulsion was investigated in the laboratory to determine if this would induce coalescence. An emulsion was made using the laboratory mill with soap solution from trial 1. The emulsion was rapidly cooled in a water bath to simulate a rapid temperature drop. Sieve tests were then carried out on both rapidly cooled and standard emulsion samples. Only a few large particles were retained with both samples. The emulsion did not coalesce with rapid cooling when tested in the laboratory.

The emulsion produced on the laboratory mill using the soap solution from trial 1 was also tested in the laboratory for its ability to handle pumping and shearing. During the field trial the emulsion was circulated in the tanker for less than 3 minutes and the phenomenon occurred. In the laboratory the emulsion was circulated for 30 minutes using a similar type of gear pump with no significant changes observed in the emulsion's properties.

A further possibility for the observed instability is the type of bitumen used by Higgins. This type of bitumen may not be able to be increased to a bitumen content of 75% due to its relatively high asphaltene and salt contents which make emulsification more difficult.

Competitors that have high bitumen content emulsions use imported Venezuelan bitumen, which make more stable higher bitumen content emulsions. Higgins does not have the facilities to import this type of bitumen. Modifiers may need to be investigated that can be added to the currently used bitumen to make it easier to emulsify.

Reformulation of the product was also discussed with the company's consultant to identify possible means to eliminate the suspected instability problem. The following suggestions were given:

- Change the emulsifier.
- Adjust the soap pH from 2.5 to 2.0 to drive a greater number of emulsifier molecules into the bitumen phase thus increasing stability.
- Increase calcium chloride to 0.1% to promote more structure in aqueous phase.
- Increase the emulsifier from 0.2% to 0.4% to provide more stability via the emulsifier.

A further field trial conducted by Higgins' product development technologist on a polymer modified 75% emulsion took these formulation suggestions into consideration. The trial used a different type of emulsifier, Akzo Nobel N39L at 0.4% with a soap pH of 2.0 and calcium chloride at 0.1%. Similar processing conditions to previous trials were used. The same drop in viscosity and residual occurred between production and application, indicating the instability was not resolved with the suggested reformulation.

This tends to suggest the formulation is not necessarily where the entire problem lies but in the treatment of the emulsion in the sprayer or a combination of both.

8.5.2 Mid-scale equipment

These problems highlight the need for mid-scale equipment to explore these types of application issues more fully and accurately on a smaller scale before entering full-scale trials. The results of mid-scale trials can be used to predict more accurately final product properties and performance.

The following benefits would be gained by implementing a mid-scale sprayer (Bearsley, 1998):

1. Estimate the product's tolerance to pumping, circulation and temperature changes .
2. Assess likelihood of run-off.
3. Establish correlations between laboratory results and field trial results.

4. Determine application parameters such as spray pressure, spray temperature and bar height for less cost.
5. Determine effects of field conditions such as: rolling, time to chipping, pavement temperature, humidity and susceptibility to early rain.
6. Evaluate possible chip loss.
7. Provide approximations of emulsion break time.
8. Estimate the time to traffic.
9. Determine if the product can actually be applied.
10. Sprayer can also be used in the development of other emulsion products and small spray sealing jobs.
11. Sprayer unit would be useful to evaluate new and alternative types of spray nozzles.

There are many obvious benefits to implementing mid-scale trial equipment providing a less risky and intermediate step to scale-up. The implementation of mid-scale trial equipment was planned and will assist in better assessment of development work. To conduct mid-scale trials for emulsion spray sealing at Higgins using the laboratory mill two issues needed to be addressed.

- Increased storage capacity of final emulsion from 30 litres to 300-500 litres.
- Application of emulsion quantities in the order of 300-500 litres.

Spray tankers currently used by Higgins can only effectively heat and hold a minimum of 1000 litres of emulsion due to the positioning of heating coils. This made the use of standard tankers unfeasible for mid-scale quantities. To effectively apply quantities of 300-500 litres a purpose built mini-sprayer would be needed. It was suggested a heated and insulated tank be mounted on a trailer along with a standard spray-bar and pumps to accurately simulate field application. The quantity of emulsion would be made in batches using the current laboratory mill setup and pumped to the mini-sprayer for storage and application.

8.6 RECOMMENDATIONS

The following recommendations have been made in an attempt to solve the problems encountered during the full scale trials:

- Implement the mid-scale sprayer unit or laboratory mounted spray-bar to enable a more accurate and cost effective means to test the stability to shearing during pumping and circulation.
- Investigate alternate pump, spray-nozzle and spray-bar designs which minimise shearing.
- Conduct an in-depth investigation into the ability of bitumen used by Higgins to be used in high bitumen content emulsions.
- Investigate a different emulsifier, Redicote 611 as suggested by consultant, Glynn Holleran. This emulsifier was used in high bitumen content emulsions in Australia and has a greater affinity to the bitumen phase compare to Dinoram SL which may provide more stability to the emulsion.
- Investigate the effect of co-milling versus pre-blending in the laboratory to establish the significance of the difference in processing.
- Implement a more reliable and accurate pump for co-milling on the laboratory mill to eliminate the differences in processing of kerosene thereby allowing better comparisons between laboratory production and full-scale production to be made.
- Conduct preliminary field trials earlier in future to establish viability. In this project a likely formulation was identified several months before field trials were conducted. This formulation although not fully optimised could have been tested in a preliminary field trial in the production facility yard to assess the ability for the product to be applied; application remained an unknown area for a majority of the project. The instability during application would have been discovered at an earlier stage.

8.7 CONCLUSION

Scale-up ideally requires three key stages at Higgins: small, mid and full-scale development. Each stage is vital in the validation process. Small-scale testing provided an indication of performance variables unable to be evaluated in the laboratory and confirmed the readiness for further scale-up. Mid-scale development was unable to be carried out due to a lack of the necessary facilities. Planning for the implementation of a mini-sprayer unit to both store and apply mid-scale quantities of emulsion was carried out and is a necessary stage in development for the product. Two full-scale field trials were conducted. During emulsion production, the soap preparation procedure was

found to accurately scale-up from the laboratory setting and the required mill settings were determined for correct bitumen and kerosene contents. In both trials emulsion was produced at a bitumen content between 73-75%. Likely trends in moving from small to full scale production were established: viscosity increased, break index decreased, adhesion remains unchanged and cohesion increased. Rectifying processing differences in kerosene addition will allow a more accurate comparison between laboratory and full scale production in future.

During application the emulsion's residual dropped to 65-67% when exiting the spray-bar. Dilution with water was eliminated as a possible cause for the reduction in residual and emulsion instability was identified as the likely problem. It is suspected the emulsion is unable to tolerate the shearing and temperature decrease it encounters when it enters the spray tanker. Reformulation was carried out in an attempt to stabilise the emulsion, with no improvement. Rapid cooling and shearing were investigated in the laboratory but testing was unable to simulate the phenomena or provide answers.

The type of bitumen used by Higgins which contains a relatively high percentage of asphaltenes and salts was a further area identified from which instability may result. It is recommended to further investigate the suitability of the bitumen used by Higgins to be emulsified in high bitumen content emulsions. It is also recommended that a means to better test the emulsion's stability to shearing and other application variables be established, such as a laboratory spraying rig or the planned mid-scale sprayer. This will allow problems to be investigated and solved on an appropriate level and more successful product development to be carried out in future. The mid scale equipment will be valuable for investigating critical applications variables at an earlier stage; these variables remained unknown in this study. Further work at the laboratory and mid-scale level will attempt to identify the source of the problems to see the product successfully scaled up in future.

9. DISCUSSION AND CONCLUSIONS

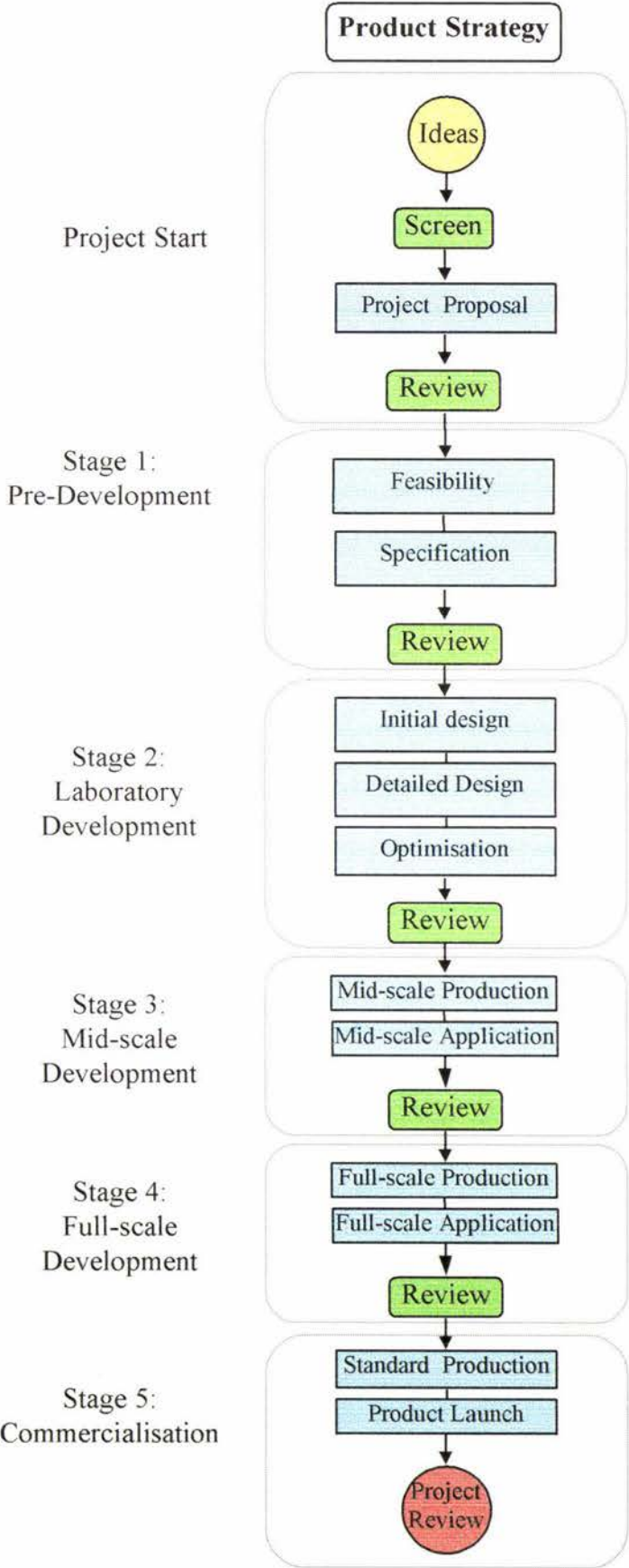
9.1 INTRODUCTION

The research conducted was focused towards developing a tailored product development process with supporting systems for use in a specific company environment. It utilised the development of a high bitumen content emulsion as vehicle to practically demonstrate these systems to the company. An appropriately designed, six-stage product development process was produced based on a product development process developed by Cooper, (1993). The developed process was used to guide the high bitumen content emulsion project and could be used by future research and development projects at Higgins. The project covered four of the six stages in-depth: Project start, Specification, Laboratory Development and Full-scale Development. Template systems were generated for the stages covered to increase the ability to carry out efficient and systematic product development at Higgins.

9.2 PRODUCT DEVELOPMENT PROCESS

A systematic approach to product development provides greater consistency in developing new products to satisfy customer needs and generate wealth for the company. It is also important that the product development process suit the company environment so the process is efficient and workable. These were the driving factors behind developing a tailored product development process for Higgins. The product development process developed for Higgins as seen in Figure 9-1, took work done by Cooper, 1993 and made appropriate changes based on the environment at Higgins. A product strategy step was added to recognise the impact product development has on strategic company direction. Terminology used by Cooper to describe the stages and smaller phases in the process was changed to terminology with which the company was familiar. Principles relating to the division of the design activity from the BS-7000, 1989 were incorporated into the Laboratory Development Stage to give three distinct phases to design and obtain more control over this critical area.

Figure 9-1 Product Development Process Developed for Higgins



Product development at Higgins is largely a technical activity at present and much of the marketing aspects and focus presented by the various processes reviewed were not appropriate to be incorporated into Higgins model at this time. Planned infrastructure development in this area may see the basic marketing requirements outlined in the developed approach expanded upon. The industrial and processed nature of Higgins products meant a greater need existed to emphasise the scale-up of products for production. A Mid-scale Development stage was added to reflect this specific need.

The product development process established a basic framework to guide the stages of product development projects at Higgins. This however lacked the necessary detail to implement the process. Work by Earle, (1997) and Rosenau, (1990) described a product development process by the required activities, outputs and decisions made at each stage. These principles were applied to the Higgins environment and assisted in describing in greater detail the developed process. Having set out the required activities and outputs to be generated systems and templates tailored to Higgins were developed to practically carry out the activities and generate the outputs.

Templates developed during the work included:

- Project brief
- Idea capture sheet
- Idea screening model
- Information consolidation system
- Information gathering systems
- Marketing specification
- Technical design specification
- Laboratory development plans
- Field trial plan
- Field trial data recording sheets

The product development process guided the broad stages and phase of product development. Details of the requirements of each stage were generated with template systems created to practically implement and support the product development process. Future work is required to produce complete product development template documentation at Higgins, particularly for the later Commercialisation stage not covered during the study.

9.3 PROJECT START STAGE

The Project Start stage included idea capture, screening and outlining ideas that passed screening in a project proposal. Ideas initiate product development, with formal systems needed to document and capture the ideas to avoid loss of valuable project ideas. A concept capture sheet was developed for Higgins to document new ideas, providing a formal means to outline an idea for eventual screening. All ideas are not turned into development projects; ideas need to be screened to identify viable concepts before further resources are committed. Weighted scoring models were found to be an appropriate means to screen ideas at Higgins. Having the correct criteria and weightings for these screening models was found to be important with improvements being made by trialing the model.

In order to produce a proposal which outlines a screened idea, information needs to be collected on the opportunity. The work demonstrated to the company new avenues it could use to collect information to conduct research based product development. Systems, procedures and tools were also developed to manage the flow of information within the technical centre in order to better utilise information in product development.

9.4 PRE-DEVELOPMENT STAGE

The Pre-Development Stage in the Higgins product development process consisted of Feasibility and Specification phases. A detailed investigation into the financial, technical and market aspects of a product establishes project viability. As this project was already considered viable detailed systems were not developed for the feasibility phase and further work is required to develop these systems. Feasibility is followed by the Specification phase. Before embarking on design work a clear understanding of the characteristics and attributes a product must have, needs to be established to focus work. The desired product characteristics are formally set out in product design specifications. To outline both market and technical aspects of a product, product design specifications were divided into marketing and technical design specifications. Examples of both types of design specifications were generated during the work and were refined as greater information became available. It is desirable that specifications be as fixed as possible at the Pre-development stage to maintain a consistent focus during further

development. A final product specification detailing the performance and testing criteria to be achieved by the end product was developed and is a useful template for other Higgins products.

9.5 LABORATORY DEVELOPMENT STAGE

The Laboratory Development stage was divided into three phases, incorporating the BS 7000, (1989) approach to design into Cooper's base product development process. The three phases were: Initial design, Detailed design and Optimisation. During initial design, significant variables impacting upon the product were identified, the number of possible raw materials were reduced, a testing regime was established and an initial design was identified; initial design prepared the way for detailed design. Factorial experimental design was identified as an appropriate technique to carry out detailed design for processed products. Detailed design used a full factorial experimental design with four-factors at two levels to explore the formulation and identify an optimum area. Experimental design was found to be a systematic means of gaining a complete understanding of how both components and process variables can be altered to control the final product. Variances inherent in testing methods reduced the ability to clearly detect purposeful changes in settlement and adhesion tests. Reduced variability of tests and fractional experimental designs requiring less runs would make this design technique more suitable for standard use at Higgins. Using fractional experimental designs would reduce the time and cost of development while gaining the most significant information about a formulation.

In the Optimisation phase the optimum area identified during detailed design was further examined to identify a final laboratory formulation.

9.6 MID-SCALE AND FULL-SCALE DEVELOPMENT STAGES

Having produced an optimum product in the laboratory which met the technical design specification, the product was planned to be incrementally scaled-up. This was to demonstrate an ability to produce and apply the developed product on standard full-scale equipment. Three phases were planned; small, mid and full-scale development. Small-

scale testing conducted in the laboratory yard provided insights into likely field performance and further confirmed the readiness for scale-up. Facilities for mid-scale development were not yet implemented therefore mid-scale development was unable to take place. Planning for the commissioning of new equipment to store and apply mid-scale quantities of emulsion was carried and will be valuable for future work. Mid-scale development is a critical stage where product application and field performance variables unable to be accurately evaluated in the laboratory can be assessed.

Thorough planning and data collection were found to be key in obtaining the greatest benefit from the two full-scale field trials conducted. Template systems to plan and record field trials were produced for Higgins. Two full-scale field trials were conducted. Scale-up of emulsifier solution was found to accurately reflect laboratory preparation and correct mill settings were established with emulsion produced at 73-75%. Comparisons between the laboratory and production mills were made identifying likely trends in future scale-up: viscosity increased, break index decreased, adhesion remains unchanged and cohesion increased.

Problems were however encountered during emulsion application. The emulsion became unstable and bitumen came out of the emulsion reducing the bitumen content to 65-65%. The instability is possibly caused from the shearing during circulation in the spray tanker and the thermal shock of entering the cooler spray tanker. Investigations were carried out in the laboratory into the effect of mechanical action, shearing, rapid cooling and reformulation but did not provide conclusive results. It is recommended to implement planned mid-scale application equipment and investigate the problem at a more appropriate scale.

9.7 RECOMMENDATIONS

Recommendations are made in two sections, covering both the high bitumen content emulsion and the product development systems developed.

9.7.1 Bitumen Emulsion

The following recommendations provide possible direction for future work to overcome

suspected stability problems and see the product effectively scaled-up.

- The relatively high asphaltene and salt content of the bitumen used by Higgins is known to make emulsification more difficult. It is recommended to investigate further the composition of bitumen used by Higgins and its ability to be used in high bitumen content emulsions.
- Redicote 611 is an emulsifier used by Emoleum in Australia in high bitumen content emulsions and has a greater affinity to the bitumen phase producing a more stable emulsion. It is recommended to investigate this emulsifier in the formulation to increase stability.
- Commission laboratory or mid-scale equipment to effectively test the stability to pumping and shearing during application. Using this equipment the likely performance of the final chip seal of this emulsion and others can also be evaluated more cost effectively.
- In order to gain a greater consistency between laboratory and full scale production and make more accurate predictions on scale up it is recommended to investigate the difference between co-milling and pre-blending kerosene. The difference is recommended to be rectified if the difference in processing makes a significant impact on the resulting emulsion properties.

9.7.2 Product Development Systems

Future work in the following areas will prove to strengthen the tailored product development process and systems introduced to Higgins.

- Develop template documentation for all key activities and outputs for the various stages of the developed product development process. This will assist in the practical implementation of systematic and reproducible product development at Higgins.
- Consolidate the product development process and templates into a product development manual to provide a documented summary of the product development process at Higgins. This will ensure the process remains independent of changes in personnel.
- Introduce systems to consider financial and marketing aspects of product development to assist in eliminating unviable projects and focusing on products with greater demand.
- Increase the flow of communication between contracting, production and technical

parties by formally integrating them into the product development process. The formal integration of these parties earlier in the process will serve to avoid potential problems.

- Continually improve the systems through trial and review to produce an efficient process.

9.8 CONCLUSION

This study produced a systematic product development process tailored specifically for the needs and environment at Higgins. The process was detailed more fully by the activities and outputs to be generated in each stage then practically implemented with template product development systems. The entire product development approach is suitable for conducting product development at Higgins in future. Further work conducted on an appropriate scale is required to investigate the stability of the high bitumen content emulsion during application to see the product effectively scaled-up.

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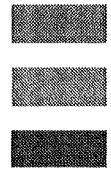
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APPENDIX 2.1 EARLE'S PRODUCT DEVELOPMENT APPROACH

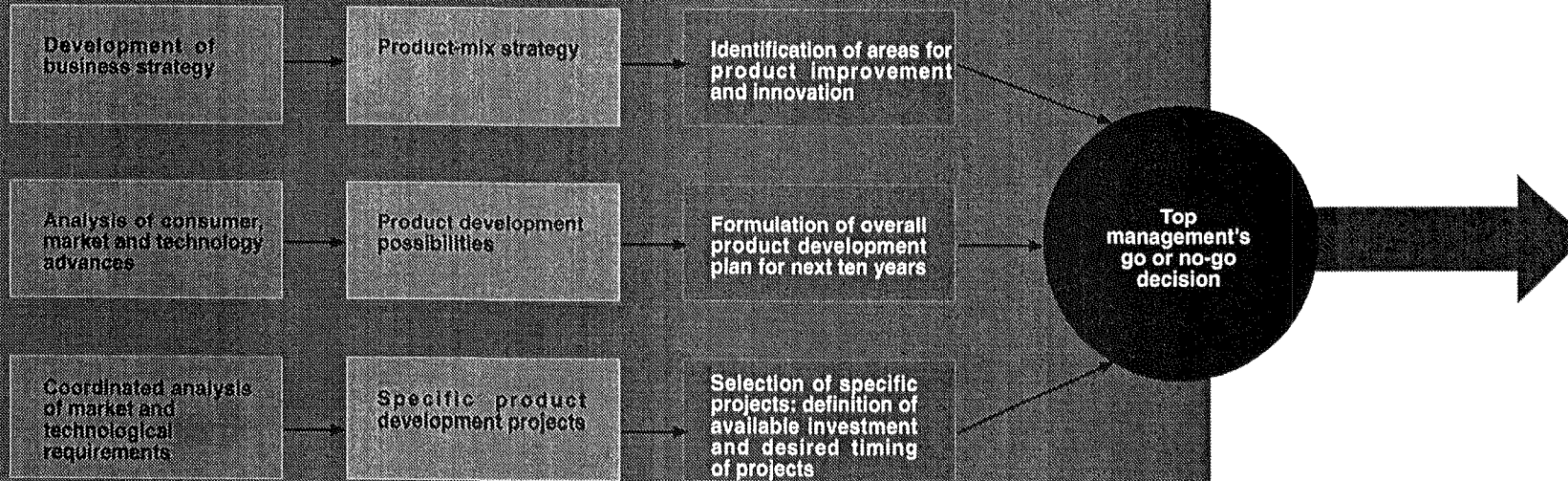
Key

Activities

Outcomes

Management
actions and
decisions

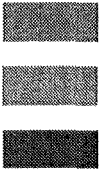
Stage 1. Product strategy and planning



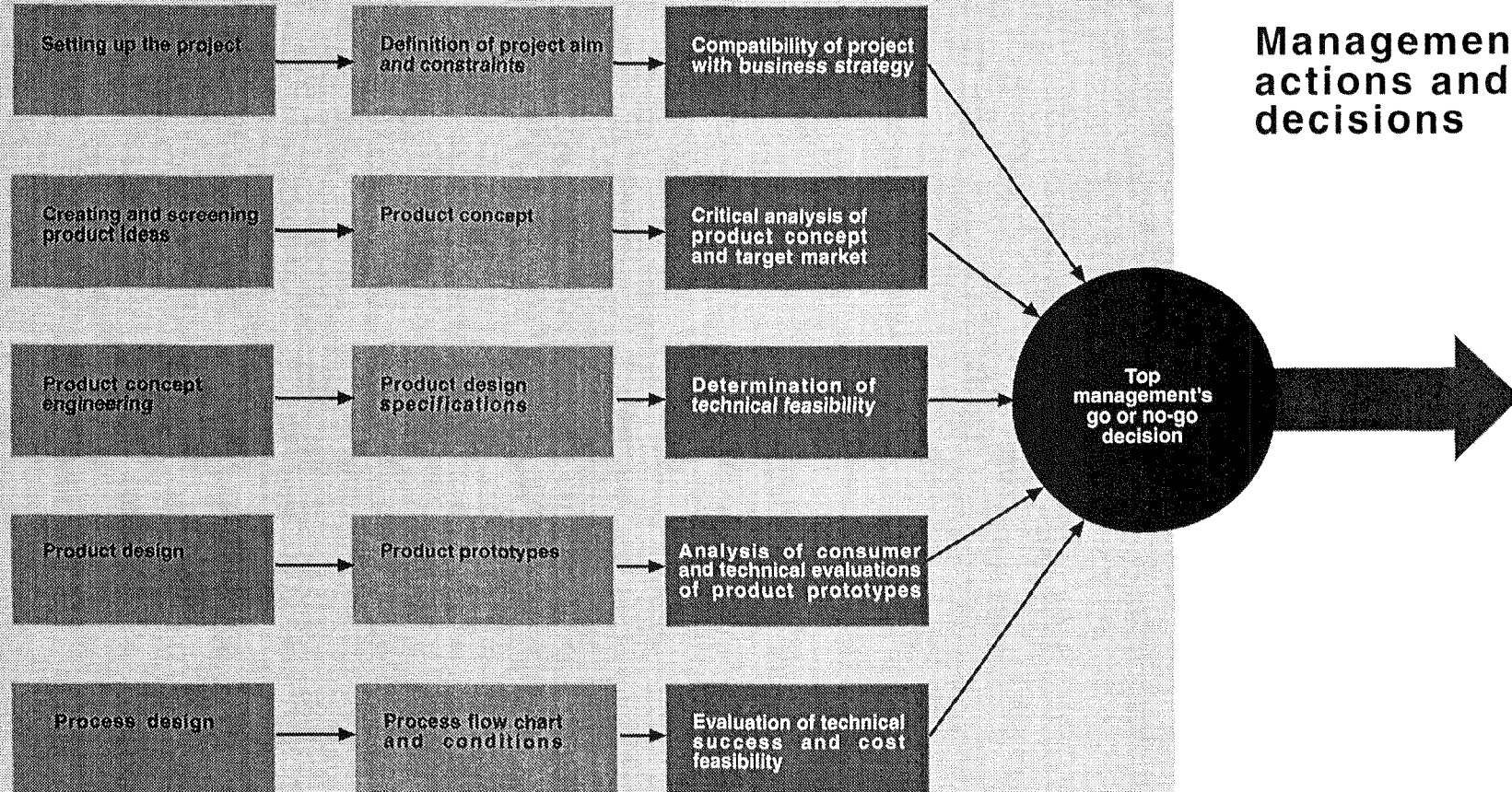
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Activities

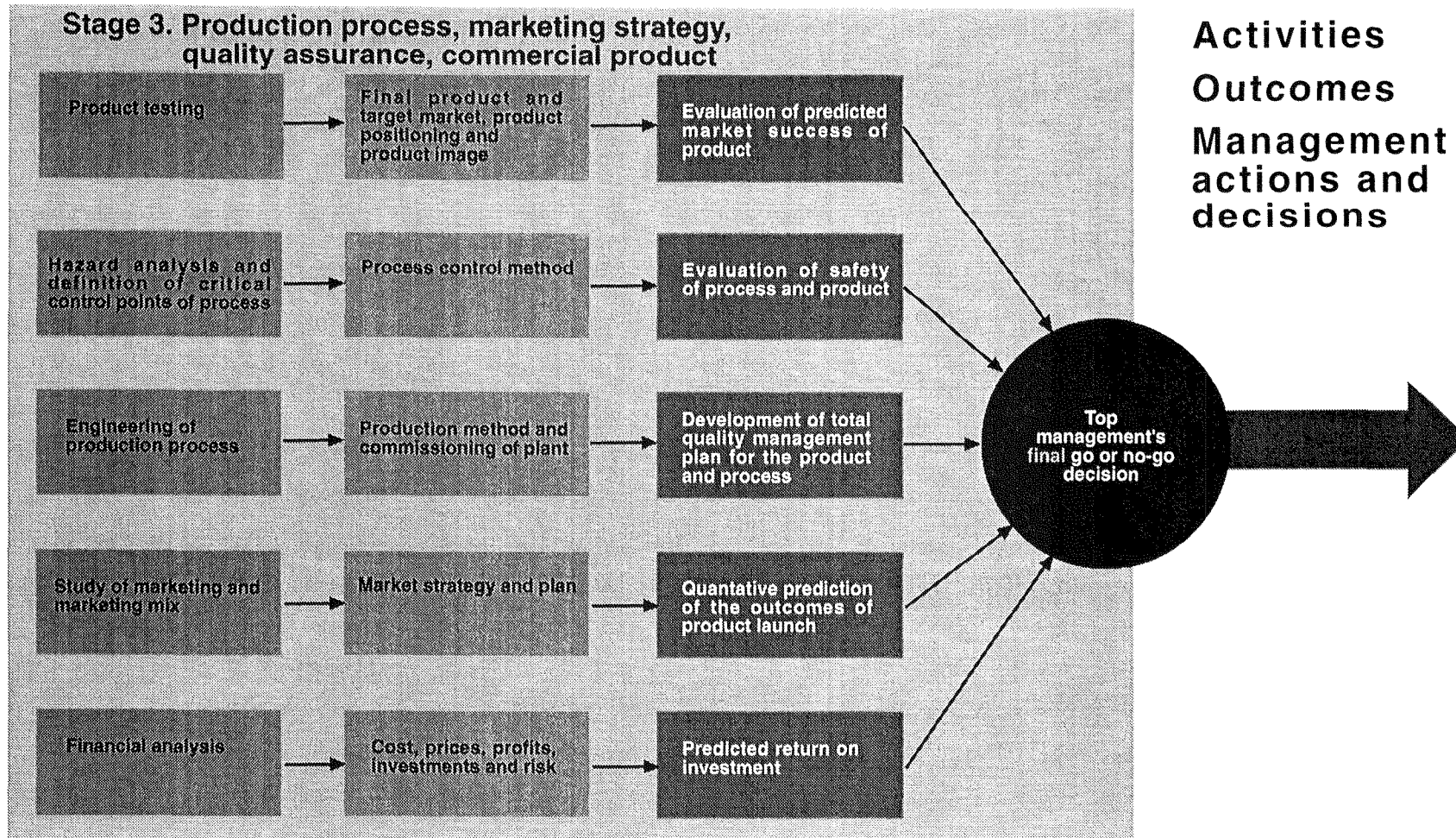
Outcomes

Management
actions and
decisions

Stage 2. Creation, design and development of product



Key



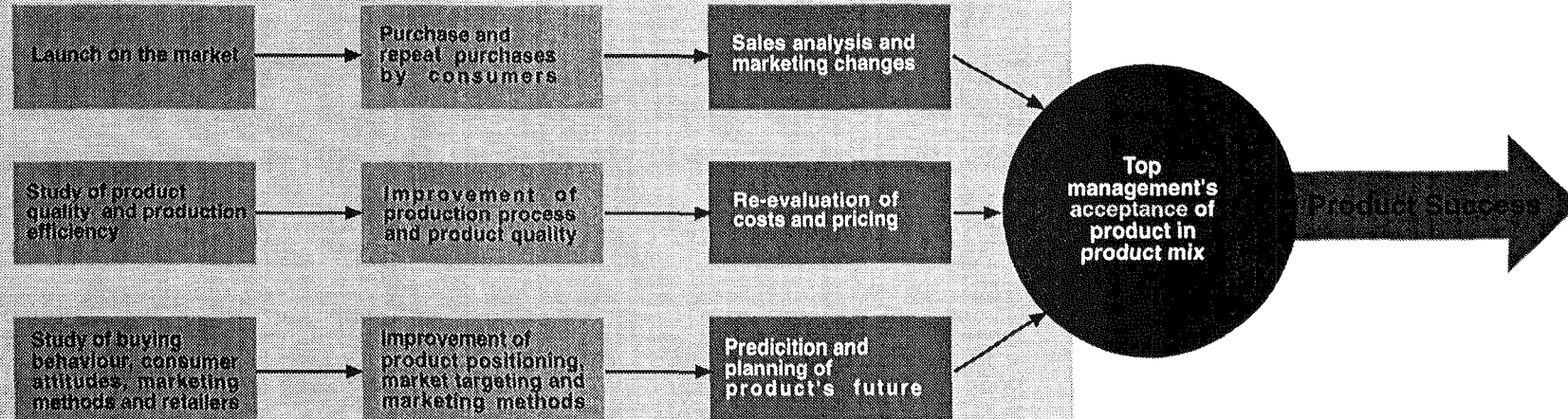
Key

Management actions and decisions

Activities

Outcomes

Stage 4. Launch and post-launch



APPENDIX 4.1 LIST OF POTENTIAL EMULSIFIERS

Emulsifiers before screening:

- Akzo Nobel, Redicote EM-24
- Akzo Nobel, Redicote EM-44
- Akzo Nobel, Redicote E-9
- Akzo Nobel, Redicote 103
- Akzo Nobel, Redicote N-39L
- QuimiKao, Asifer 123
- QuimiKao Asifer 100
- CECA, Dinoram SL
- CECA, Emulsamine 640
- CECA, Emulsamine L60
- Valley Slurry Seal, Road Chem 400
- Valley Slurry Seal, Road Chem 401
- Witco, DXP 5357100

Witco, Arosurf - AA 23, AA 27, AA 28, AA 54, AA 54-100, AA 55, AA 60, AA 75.

APPENDIX 5.1 CONCEPT CAPTURE SHEET

Subject/Topic: _____ Date: _____

Information Source: _____ Publication Date: _____

Basic Description of Technology: (*General principles*)

Implications for operations: (*Benefits, Product Need*)

Potential for implementation

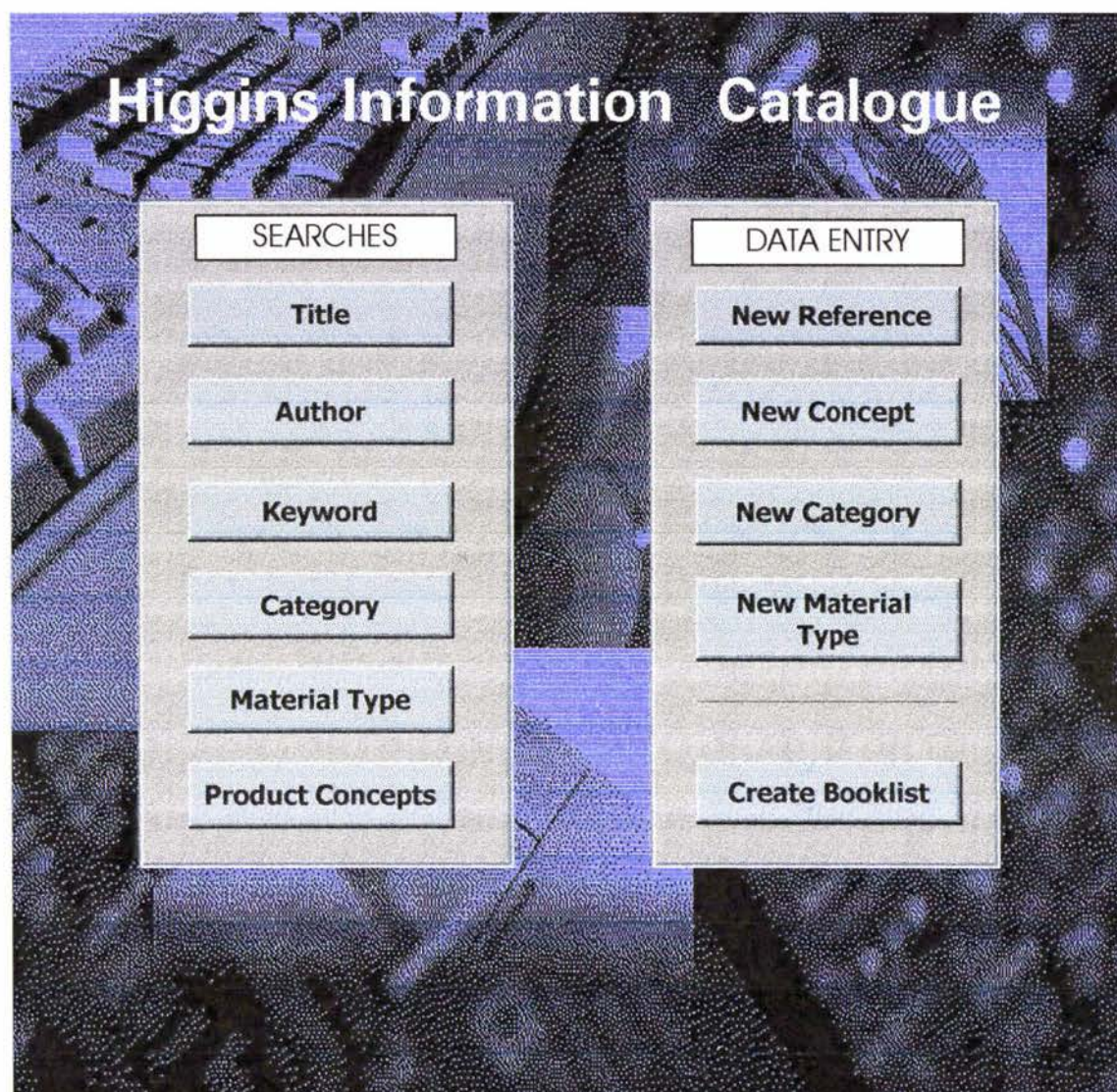
Key Words: 1. _____ 2. _____ 3. _____ 4. _____

Illustrations

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Review Comments

APPENDIX 5.2 SCREEN SHOT OF HIGGINS INFORMATION CATALOGUE: MAIN MENU



APPENDIX 6.1 CUSTOMER REQUIREMENTS SURVEY

A. Perceptions of Emulsion technology

1. What are the main advantages you see in using emulsions?

- Reduced Kerosene, less bleeding and less sticky in summer.
- More environment friendly.
- Better wetting ability as its water based.
- Safer, important for workers.
- Less fumes so drivers can see what is happening and bits aren't missed, especially on curves.

2. What are the main difficulties you perceive in using emulsions?

- Difficult to spray, Uneven distribution, streaking.
- Stones stripping off, possibly low application rates. Meniscus effect? Want one that hangs in there.
- Traffic being let on too quickly
- Not given time to set on the first day. Slow to set initially, but good once its set.
- Too runny, ends up in the gutter. Hard to keep the edges tidy.
- Hard to predict weather it will hold off rain for long enough.
- We have stopped using emulsions as the agricultural nozzles used here get blocked and cause streaking problems. Bitumix are importing trucks with Australian nozzles. Need to calibrate nozzles in the lab.
- The man on the ground doesn't know how to use emulsions especially polymers. Lack of understanding and training.
- Expensive and are all too dear, used for special conditions high stress.
- Problem with polymer modification is the stones don't lay down on rolling therefore produce a lot of noise. Rate payers have complained about noise.
- Polymer modified emulsions provide good grip and last long time but need to consider the urban context, would be good in rural areas though.

3. What specific criteria is applied to determine a change from conventional hot cutback chip sealing to an emulsion?

- Don't really know.

4. What information can Higgins provide to assist in specifying alternatives to cutbacks?

- A lot of it doesn't mean much, it is how it works that counts. I need evidence. If a guarantee can be given for the first winter or first year I'd be happy.

5. Do you have any other problems or needs associated with specifying emulsions or other alternatives to standard cutbacks?

- No.

B. High Bitumen Content Emulsions (HBCE)

1. Do you see a need for a HBCE?

- Yes, as it's more like bitumen and holds the bigger stones, grade 4 & bigger.

Less water to cart around compared to other emulsions. Still a lot of water compared to cutback. 80% bitumen compared to 95%.

2. How large do you see the need, both now and in the future?

- Growing, International trend.

3. If Higgins were to offer a HBCE would you specify it?

- Yes, But have to know that the technology is well understood proof of reliability to overcome past experiences. Test patch?

4. In the past what has prompted the need for a 70 % emulsion?

- High stress areas or late season. But even in late season been referred to cut backs, tried and true method. General lack of confidence in emulsions.

5. Were you satisfied with the 70% emulsion performance ? What were the advantages problems?

- Have stopped using emulsions, have been bitten before. It rained and ended up in the gutter, rate payers complained. Washed into channels and end up on peoples feet and then in their carpets.
- Emulsions generally have good wetting.

6. Rank the requirements of an emulsion.

Environment friendly	<u>4</u>
Safety	<u>3</u>
Performance	<u>1</u>
Cost	<u>2</u>

- The bottom line is if it works we'll use it.
- Cost always comes into it.
- Reduced kero and lower temperatures are important form a safety point of view.
- Environmental danger of kero not really proven? Is it really harmful? Environment concerns are also coming from ratepayers.

C. Bench marking

1. How does Higgins performance compare against other contractors? Ranking.

- Pretty good, one of the best spent a lot of time with them. Top at this stage

2. Where do Higgins need to improve ?

- Lack of house keeping. Letting the spray bar dribble when its resting leaving large globs. Can't they dribble it onto paper? Keep the public happy.
- Stay on site a little longer, an extra 15 minutes and run their eyes over the job.
- Controlling traffic in critical early stages to allow proper setting, slow things down a little.
- Doing the little tidying up things is important, the finishing touches like the clearing sumps and the manholes, tidying the edges.
- Problems at intersections still.

3. What are Higgins doing well?

- Know what they are doing.

APPENDIX 6.2 MARKETING SPECIFICATION

Product Concept Statement: (*Precise description of the intended development.*)

A high bitumen content emulsion with a residual binder of greater than 75% meeting the TNZ M1& P17 specifications. The product is to be used primarily in spray chip sealing as an alternative to hot cutback bitumen for primary roads under standard conditions. The major benefits to the customer include a safer, more environmentally friendly alternative.

Product Description: (*Description of core technical characteristics*)

A rapid breaking, rapid curing emulsion with a residual binder of 75% or greater.

Intended Use: (*What application is the product intended for.*)

Chip sealing and resealing according to TNZ/M1 and P17 Specifications.

Market Environment: (*Opportunities and threats in the market.*)

Recent advances in bitumen technology have allowed the development of high bitumen content emulsions, currently none of the local competitors have products of this nature. Industry specifications have already been drafted for these products in anticipation of their arrival therefore development of such products by Higgins' competitors should be assumed.

Technics have a slight edge in the market as they have a CRS-2 product with 70% bitumen, and have first option to buy rights to the Australian product containing 80% due to links with the Mobil developers. They also have a well established research and development infrastructure to develop competitive products. The other major threat in the market is Bitumix, the local council has also used their 70% emulsion product in the past.

Pressure may be exerted on other competitors from Occupational Health and Safety if this safer technology can be developed. On the other hand if competitors develop the technology first pressure will be placed on Higgins to reduce the risks associated with high temperature bitumen. Environmental benefits will also become increasingly important in the future.

Higgins will need to develop such a product in order to compete in the emulsion market. Early entry into the market will assist in gaining a reputation with emulsion technology and market share. Introducing the latest technology will also begin to establish Higgins in the market as a company with a technological edge and a reputation for offering a full range of roading solutions.

Product Strategy: (*Describes what the product will achieve for the company.*)

Product positioning: (*Where the product fits in the companies product range?*)

The product will extend the companies cationic rapid set (CRS-2) range of emulsions.

Product Strategy: (*What is achieved by having the product in the companies range?*)

i) To gain a greater number of sealing and resealing contracts by offering a high performance and cost competitive alternative to hot cut-backs.

ii) Offer a safer and more environmentally friendly product over hot cut-backs.

Customer Benefits: *(What attributes make the product desirable to the customer?)*

- Lower transportation costs compared to other emulsions
- Lower energy consumption compared to cutbacks resulting in cost savings
- Safer, lower operating temperatures compared to hot cutbacks
- Reduced danger of explosions
- More environmentally friendly, less hydrocarbons in the form of cutters.
- Less fumes compared to hot cutbacks
- Better wetting ability compared to cut backs
- Greater efficiency in application
- Reduced cure times compared to other emulsions
- Greater resistance to rain compared to other emulsions
- Tolerate cool conditions to give a longer sealing season

Potential weaknesses: *(What areas need attention during development)*

- Run off, emulsion ending up in gutter.
- Susceptible to heavy rain.
- Viscosity needs to be low enough to be pumped and sprayed evenly
- Limited storage stability
- Overcoming lack of confidence with emulsions due to bad past experiences.
- Skinning- a cheesy state formed on the surface of emulsion reducing wetting of stones & adhesion

Market size: *(Estimated volumes)*

The intended market for the product is mainly resealing. Market estimates of this were taken from Transit roading statistics (Transit, 1996b).

State Hi-way Reseals:

Nth. Island	759.8 Km	\$28,050,800
N.Z Total	1,241.7 Km	\$43,241,000

Local Roads Reseals:

Nth. Island	2,657.1 Km	\$24,742,700
N.Z Total	3,897.8 Km	\$35,807,100

Total Reseals	Nth. Island	3,416.9 Km	\$52,793,500
	N.Z Total	5,139.5 Km	\$79,048,100

The total market size for bitumen reseals is valued at \$79,048,100 this is the main target market. Higgins only operates in the North island therefore a figure of \$52,793,500 is more appropriate of the potential market. The figures do not include rehabilitations or seal extensions which are proportionally small but gives a pessimistic value of the market size for an alternative to hot cut backs.

Cost:

The cost should be similar to current emulsions at 80 cents/L.

APPENDIX 6.3 TARGET TECHNICAL DESIGN
SPECIFICATION

Industry Specifications:

Table 1: Transit New Zealand M/1 Specifications: Class CQ-75 emulsions.

Property	Method	Value
Viscosity @ 70 C	TNZ M/1 :1996 Section 2.2	300 cP min.
Viscosity @ 85 C	TNZ M/1 :1996 Section 2.2	215 cP min.
Binder content, mass %	BS 434: part 1 1984	75% min
Residue on 710 µm sieve, % by mass	TNZ M/1 :1996 Section 2.3	0.05% max
Particle charge	BS 434: part 1 1984	+ ve
Diluent content, % w/w binder	ASTM D244-83a	4% max

Laboratory Testing Performance Specifications:

Table 2: Laboratory Testing Specification Requirements.

Parameter	Method	Value	Tolerance
Residue	LP 16. 10g @ 120°C	75 %	± 0.5%
Final pH	TMNEL4	3.0	± 0.1
Break index	T 66-017	200%	± 50%
Cohesion	Frosted marble	No established values.	-
Adhesion	NF T66-018	> 75%	<100%
Viscosity @ 70 C	TNZ M/1:1996 Section 2.2	800 cP (5rpm)	± 50 cP
Storage stability	ASTM D244:45-50	1% max @ 24hrs	-

Date:_____	Version: <u> 1.2 </u> .
Signed:_____	Signed:_____
John Bryant (GTM)	Phillip Clark (Product developer)

APPENDIX 6.4 REFINED TECHNICAL SPECIFICATION

Performance Guidelines For CRS2-75 Spray Seal Emulsion

1. Scope

These guidelines have been prepared for the benefit of those engaged in chip seal construction. The guidelines highlight areas essential to achieving consistent high quality results. Information is presented on the bitumen emulsion, aggregates, application methods and construction procedures necessary for successful sealing.

2. Product Definition

CRS2-75 is rapid breaking, rapid curing, cationic emulsion with a residual binder of 75% for use in chip sealing and re-sealing.

3. Applications

CRS2-75 is to be used for the following applications:

- Single coat seals and re-seals
- Two coat seals

Applicable to low stress areas and traffic volumes of less than 3,500 vehicles per day.

4. Standards

The seal should comply with the following standards:

- Transit New Zealand M1- Specification for roading bitumen
- Transit New Zealand M6- Specification for sealing chips
- Transit New Zealand P17- Performance based specification for bituminous reseals.
- Higgins quality performance indicators- Section 8.1 Emulsion Specification

5. Product Benefits

CRS2-75 provides a number of benefits over other emulsions and standard hot cutbacks.

CRS2-75 Compared to Other Emulsions

- Less water cartage translating to lower transportation costs
- Greater application efficiency
- Reduced cure times
- Faster time to traffic
- Greater resistance to rain

CRS2-75 Compared to Hot Cutbacks

- Lower and more safer operating temperatures
- Reduced danger of explosions
- More environmentally friendly, less hydrocarbons in the form of cutters.
- Less fumes
- Better chip wetting ability
- Tolerate cooler conditions to give a longer sealing season

6. Product Limitations

- Product is not suitable for high stress conditions or areas with greater than 3,500 v.p.d.
- CRS2-75 should not be mixed with anionic emulsions. Anionic emulsions should be cleaned from equipment before using CRS2-75.
- Dilution of CRS2-75 with water may cause emulsion to break.

7. Temperature and Weather Restraints

CRS2-75 has a greater resistance to rain compared to other emulsions however a clear forecast for at least 24 hours after laying will give a consistent seal.

Rain within 24 to 48 hours after construction combined with action of traffic may cause emulsion to re-emulsify and run. In these circumstances further rolling is recommended.

Pavement temperature should not be below 10°C to avoid poor wetting and excessively long cure times.

8. Emulsion specification

The emulsion shall comply to TNZ/M1 specifications for high bitumen content emulsions.

Details of emulsion properties given in Table 1.

Table 1: Transit New Zealand M/1 Specifications: Class CQ-75 emulsions.

<i>Property</i>	<i>Method</i>	<i>Value</i>
Viscosity @ 70 C	TNZ M/1:1996 Section 2.2	300 cP min.
Viscosity @ 85 C	TNZ M/1:1996 Section 2.2	215 cP min.
Binder content, mass %	BS 434: part 1 1984	75% min
Residue on 710 µm sieve, % by mass	TNZ M/1:1996 Section 2.3	0.05% max
Particle charge	BS 434: part 1 1984	+ ve
Diluent content, % w/w binder	ASTM D244-83a	4% max

8.1. Emulsion Composition

<i>Component</i>	<i>Percentage Of Total emulsion</i>
Bitumen 180/200	75%
Diluent	1.5%
Water, emulsifier and additives	23.5%
Total	100%

8.2. Higgins Quality Performance Indicators

In addition to Transit New Zealand specifications the emulsion will meet Higgins Quality performance indicators outlined in Table 2.

Table 2: CRS2-75 Performance Indicators.

<i>Parameter</i>	<i>Method</i>	<i>Value</i>	<i>Tolerance</i>
Residue	LP 16. 10g @ 120°C	75.5%	± 0.5%
Emulsion pH	TMNEL4	3.0	± 0.1
Break Index	T 66-017	200%	± 25%
Cohesion	Frosted marble	1 hrs 9.0 kgcm 2 hrs 10.5 kgcm 3 hrs 12.0 kgcm	± 0.5 kgcm ± 0.5 kgcm ± 0.5 kgcm
Adhesion	NF T66-018	> 75%	<100%
Viscosity @ 70 C	TNZ M/1:1996 Section 2.2	800 cP (5rpm)	± 50 cP
		600 cP (10rpm)	± 20 cP
		450 cP (20rpm)	± 10 cP
Storage stability	ASTM D244:45-50	1% max @ 24hrs	-

9. Emulsion Storage and Handling

The following conditions are recommended for storage and handling of the emulsion.

- Applied within 24 hours of manufacture
- Held at a constant temperature of 85°C till application
- Gentle circulation

The following conditions should be avoided during storage and handling.

- Excessive pumping and agitation.
- Large fluctuations in temperature
- Boiling or freezing of the emulsion
- Rapid temperature increases and decreases

10. Aggregates

Grade 3 and 4 chip complying with TNZ/M6 should be used for single coat seals.

10.1. Aggregate Compatibility

Compatibility of aggregate with emulsion should be evaluated prior to sealing. Poor compatibility will result in low adhesion and increase the susceptibility to early rain. Compatibility can be evaluated using adhesion test NS T 66-018, adhesion should be greater than 75%.

10.2. Aggregate Cover

A uniform coverage of aggregate should be applied as soon as practicable after spraying to avoid inadequate chip wetting and adhesion. Aggregate coverage should meet standards set out in TNZ P17.

11. Emulsion Spraying

The following variables should be considered when spraying CRS2-75 emulsion.

- Optimum spray temperature - 85°C (Acceptable range of 80°C-90°C)
- Nozzles correctly positioned
- Nozzles are clear, undamaged and of similar size.
- Spray bar height adjusted to obtain a double overlap spray pattern.
- Correctly calibrated.
- Correct pressure maintained to produce sufficient atomisation

12. Rolling

A minimum of three passes with pneumatic tyre rollers should be used. Two rollers should be kept as close as possible to chip spreader to ensure rolling before emulsion breaks.

13. Brooming

Removal of excess chip should be carried out the morning following application, when binder is firm and has developed sufficient strength to withstand brooming. Care should be taken to avoid removal or turning over of embedded chip.

14. Traffic Control

Proper traffic control during the initial curing period is critical to seal quality. Initial strength is reduced as full aggregate interlock has not developed and water is still leaving emulsion.

During construction all traffic and equipment should be kept off the seal. After construction all traffic should be kept off the seal for at least 90 minutes to allow initial strength to be developed.

Traffic speed should be kept below 30 Km/h for a minimum of 24 hours.

The length of time the newly constructed seal must be protected from high speed traffic is dependant on conditions.

Date: 12.2.98	Version: 1.3
Signed: _____ John Bryant (GTM)	Signed: _____ Phillip Clark (Product Developer)

APPENDIX 7.1 EMULSION PROPERTIES FROM EXPERIMENTAL DESIGN EXPERIMENTS

Code	Residue	pH	Viscosity 5 rpm	Viscosity 10 rpm	Viscosity 20 rpm	Viscosity Value	Settle- ment	Break Index	Adhesn.	Cohesn. 1 hour	Cohesn. 2 hours	Cohesn. 3 hours	Cohesn. Value
1	76.76	3.15	820	585	450	1093	0.63	270	80	8.0	9.8	11.6	6.2
a	76.97	2.96	680	485	373	907	0.93	257	70	8.0	8.6	11.6	5.8
b	77.52	2.50	960	680	550	1355	0.56	256	65	10.4	11.6	11.8	9.9
ab	76.27	2.36	760	545	418	1014	0.13	253	50	9.0	10.4	9.6	7.6
c	76.55	3.06	630	443	339	847	-0.23	235	68	8.9	9.6	13.2	6.3
ac	76.40	3.29	740	515	383	1018	-0.48	238	70	9.2	9.4	13.0	6.7
bc	76.57	2.52	730	530	410	963	-0.98	227	60	8.4	9.0	9.4	7.9
abc	76.27	2.30	820	580	433	1119	-0.17	204	30	9.6	10.6	11.0	9.0
d	76.66	3.04	630	450	345	841	-0.04	192	80	9.2	11.0	12.8	7.4
ad	76.21	3.10	805	550	408	1118	-0.55	197	80	7.8	8.4	11.2	5.7
bd	76.08	2.30	720	515	390	969	-8.65	175	60	9.2	9.4	9.8	8.9
abd	75.68	2.48	790	560	418	1078	0.19	198	60	8.4	10.2	10.6	7.5
cd	76.46	3.17	1000	700	550	1429	-0.07	160	60	9.0	9.0	9.6	8.6
acd	76.04	3.08	850	595	440	1171	-3.18	194	80	8.2	9.8	11.6	6.5
bcd	76.09	2.63	920	630	450	1306	-0.58	165	80	10.3	10.3	10.4	10.2
abcd	76.03	2.53	800	570	420	1098	-1.13	192	55	10.4	12.2	13.4	9.0
CP1	76.40	2.83	750	525	388	1034	0.99	200	75	8.6	10.4	13.0	6.3
CP2	76.28	2.81	750	535	405	1011	-0.36	205	75	9.6	11.2	13.6	7.5

APPENDIX 8.1 FIELD TRIAL PLANNING TEMPLATE

The field trial will be broken into two distinct sections: production and application. Application consisting of spraying, chip application and rolling. In each section checklists of the required activities and equipment have been outlined. Contingency plans for possible unfavourable outcomes have also been generated. A Gantt chart outlining the general timing of the trials is also included.

Production

Activities

- Sample raw materials
- Sample emulsion 500 litres
- Sample final product
- Conduct break, adhesion, residue and pH tests.
- Record soap preparation procedure and readings
- Record production variables

Equipment

- Sand for break test.
- Toatoaroa & Fraser's chip - adhesion test.
- Pottles and cups for tests
- Hand Thermometer -test viscosity

Production Contingency Planning

If emulsion is too *viscous* after 500 litres.

1. Reduce bitumen content- make a useable lower content emulsion.
2. Continue regardless.
3. Abort.

If emulsion has a *too low* viscosity after 500 litres

1. Add calcium chloride which was found to increase viscosity.
2. Increase bitumen content.
3. Continue regardless.
4. Abort.

Application

Divided into three areas:

1. Emulsion spraying
2. Aggregate application
3. Rolling

Emulsion spraying

Activities

Calibrate spray bar to correct rate.

Sample emulsion from truck.

Observe spray pattern for even distribution.

Record all variables- bar height, pressure, nozzle positions.

Observe for run off and break time.

Assess spray temperature and effect of spraying (shear).

Equipment

Sample tin

Ray gun / Thermometer for spray temperature.

Building paper- calibration

Spraying contingency planning

If emulsion *runs-off*- Lower spray temperature.

If emulsion is *too viscous*- Increase spray temperature.

May need to start at lower spray temperature and increase if required.

Spray temperature will need to be set once viscosity is known.

Aggregate Application

Activities

Sample aggregate

Take emulsion temperature when chips applied

Observe chip wetting ability

Observe for any skinning /cheesy state

Record total volume of chips used

Record distance and time between emulsion spraying and chip spreading

Equipment

Stop watch

Sample bags

Ray gun thermometer

Aggregate Application Contingency Planning

If there is poor control over aggregate application rate, chips should be over applied rather than under applied.

Rolling

Activities

Observe pavement before rolling

Observe pavement after rolling

Observe for pick up

Rolling Contingency Planning

Excess pick up- Longer cure time before rolling.

Video Operator Instructions

Critical Footage at Each Stage:

1. Emulsion application

Spray pattern

Emulsion behaviour on ground

Uniformity of application

2. Chip Spreading

Chip wetting

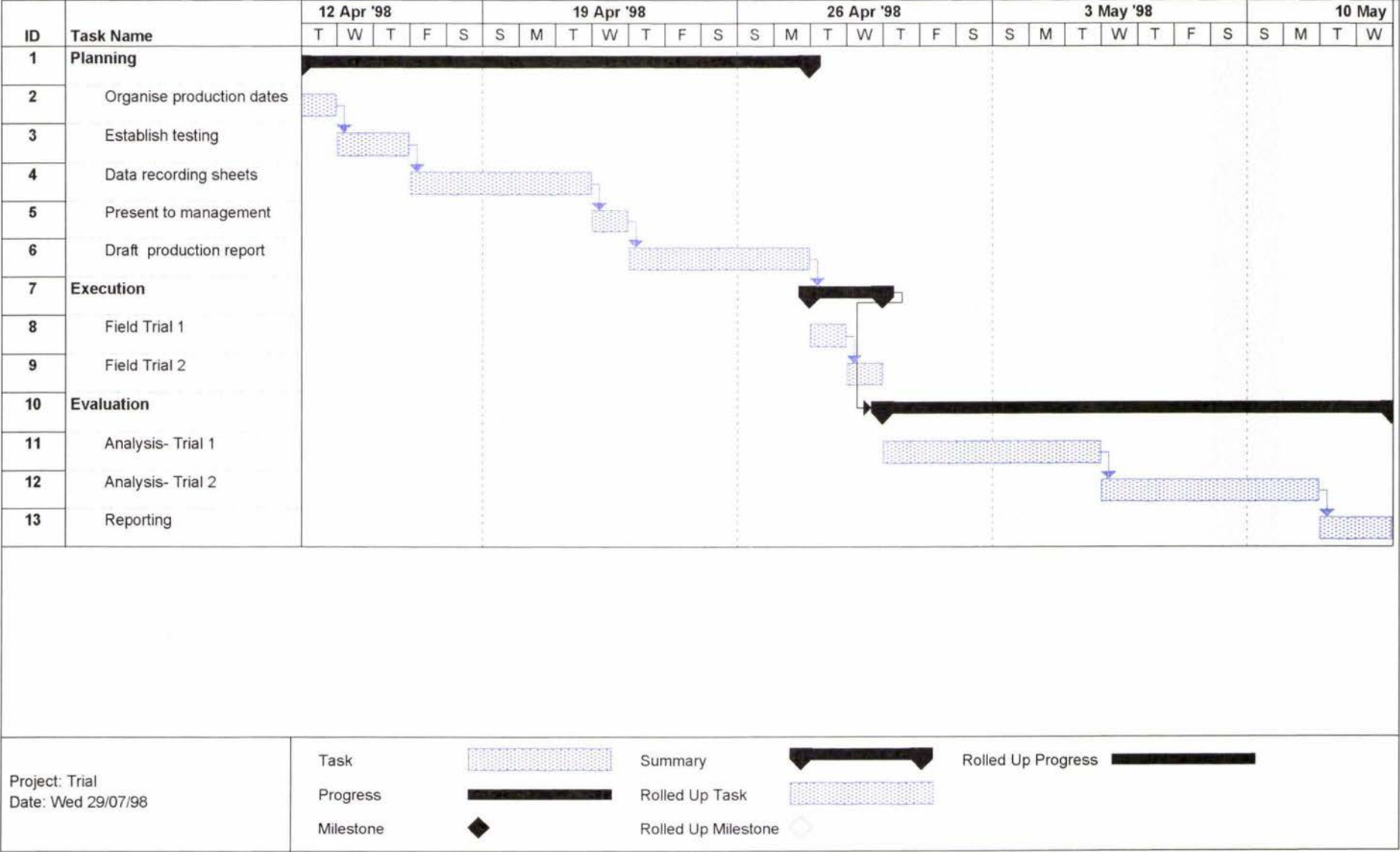
Uniformity of application

3. Rolling

Before rolling and after rolling

Stone pick up by roller

Appearance after rolling



APPENDIX 8.2 DATA RECORDING TEMPLATES
FOR FIELD TRIALS

EMULSION PRODUCTION TRIAL RECORD

Date: _____ Emulsion type: _____

Soap Solution

Type	Amount (kg)	Measurements	Value
1.		Conc. Temp	°C ± °C
2.		Conc. pH	
3.		Dilute Temp	°C ± °C
4.		Dilute pH	

Mixing time _____Min

Soap Volumes

Concentrate volume _____ Litres
Dilute volume _____ Litres
Total dilute volume used _____ Litres

Cutter

Type	Volume used
	L

Bitumen

Type	Volume used	Temperature
1.		°C ± °C

Blended Products

Type	Loaded (kg)	Used (kg)
1.		
2.		

Blending Method

Bitumen phase Soap Phase Post added

Processing Temperatures

Exit temperature _____°C ± _____°C
Mill temperature _____°C ± _____°C

Flowrates

	Setting	Ltrs Used
Soap	_____ L/min	_____
Bitumen	_____ L/min	_____
Cutters	_____ L/min	_____
Additives	_____ L/min	_____

Emulsion Tests

	500lts	Final
Residue	_____%	_____%
pH	_____	_____
Viscosity (5rpm)	_____cP	_____cP
Viscosity (10rpm)	_____cP	_____cP (Spindle #____, ____ °C,)
Viscosity (20rpm)	_____cP	_____cP

CHIP SEALING FIELD TRIAL RECORD

Binder description

Bitumen grade _____ Emulsion type _____
Cutter type & content _____ Polymer type & content _____
Adhesion agent type & content _____

Aggregate

Source: _____ Grade: _____
Conformance to TNZ M/6 Pass / Fail _____
ALD _____ mm Passing 4.75mm Sieve _____ %
AGD _____ mm Broken Faces _____ %
AGD/ALD _____
Least Dimensions within 2.5 mm of ALD _____ %

Pavement conditions (Moisture, condition, type)

Pavement Temperature _____°C

Weather

24 hours prior _____
Current _____
24 hours after _____
Air temperature _____°C

Application

Emulsion Flowrate _____ L/min Emulsion Volume Used _____ L
Bar height _____ Bar pressure _____
Emulsion Application temperature _____°C
Chip Application temperature _____°C
Spray pattern (Comments) _____

Aggregate Flowrate _____ m³/min Aggregate Volume Used _____ m³
Roller passes _____

Observations

Run Off _____
Wetting ability _____
Effect of rolling _____
Break Time _____
Adhesion _____
Cohesion _____
Time to traffic _____
Cure time _____