Bituminous Emulion:

Preparation of Its Anionic Type Using **Ash-lye**

BY

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CERTIFICATION

Thesis entitle "Bituminous Emulsion: Preparation of its Anionic type using Ash-lye" by Mr. Joshua Olajide Ojo meets the regulations governing the award of degree of Master of Science (Civil Engineering Construction Materials) of Ahmadu Bello University, Zaria, Nigeria, and is approved for its contribution to knowledge and literary presentation.

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DEDICATION

This work is dedicated
To God for he is God of
all.
To Ojo's families for their
deepest love.
To my brethren for their
help and support.
To the memory of my beloved
mother Late Mrs. Mary Olajumoke
Ojo and
To advancement of knowledge in
Construction Materials.

DECLARATION

I hereby declare that this thesis has been composed by myself and that it is a record of my own research work. It has not been accepted in any previous application for a higher degree. All quotations are distinguished by quotation marks and the sources of information are specifically acknowledged by means of references.

Joshua Olajide Ojo

Date: 10th. June. 1991

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ABSTRACT

In most parts of the world, the use of waste together with other materials in order to maximize their usefulness are being investigated for possible adaptation in construction industries. As an effective strategy for reducing cost and to enhance environmental disposal of these materials especially in developing countries where the cost of construction continues to generate concern, the use of local materials for construction works has been extensively embraced.

This thesis investigate the use of ash-lye as an emulsifier in the preparation of anionic emulsion. It involves the review of various Bituminous Emulsions, their classifications methods of preparation, various emulsifiers in use, theoretical back-ground on their performances and experimental tests on the properties of emulsion using ash-lye as an emulsifier.

A tabulated and graphical representation of results obtained were made and mathematical analysis was performed on the tested results using British Standard for comparison.

The findings indicated that Ash-lye can be used as an emulsifier in place of Sodium Octadecanoate in the preparation of anionic emulsion with 95% confidence. In using ash-lye, the charge particle, viscosity, residue on 710μm sieve, stability with coarse aggregate and storage stability both for short and long period requirements as specified in British Standard (BS 434) were met.

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ABBREVIATIONS

A	Mean of sample 1
a	Mean of sample 2
BST	Bituminous Surface Treatment
D	Standard Deviation of sample 1
d	Standard Deviation of sample 2
N	Number of Observation for sample 1
n	Number of Observation for sample 2
RRL	Road Research Laboratory
tp	Value from t-distribution table
VITA	Volunteers in Technical Assistance

CHAPTER ONE

INTRODUCTION

1.1 General Background

Most bituminous binders used in construction are viscous semi-solids at normal temperature , therefore for most operations they need to be brought to fluid state for effective and easy handling.

Similarly, in some applications such as macadam mixes, the aggregates used need to be heated as well as coated. Due to lack of adequate heating facilities, especially in remote areas, and the cost of installing modern equipment for heating there is a demand for binder that can be applied while cold.

Cut-back bitumen which would have served these purposes suffer the disadvantages of cost of production as well as limitation of application to only dry surfaces. To overcome these difficulties, bituminious emulsion was selected for the research because it will not only flow readily at atmospheric temperature but it can also be applied on damp surfaces.

Furthermore, bituminous emulsion has been effectively utilized in different fields of engineering. Engineers have capitalized its properties by using it as stabilizer for stabilizing cohesionless soil. It has been effectively used in curing of concrete especially in road construction. It also performed very well when it was used in making bricks for constructing low cost houses in India.

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1.2 Bituminous Emulsion

Bituminous Emulsion is a liquid product of bitumen in which substantial amount of bitumen is suspended in a finely divided condition in an aqueous medium by means of one or more suitable emulsifying agents.

Therefore, as said, an emulsion is a two-phase system that consists of two immiscible liquids, one being dispersed as finite globules in the other. These globules of dispersed binder are kept from joining together by the presence of a third substance called emulsifier.

It is the nature of the emulsifier that determines the type of emulsion that is produced. The four main types of emulsifiers are Anionic, Cationic, Non-Ionic and Collodial Emulsifiers.

An anionic Emulsion is an emulsion in which its emulsifier is characterised by having an anion at the interface with the bitumen which is negatively charged and in aqueous phase, is alkaline.

Cationic Emulsion is the type where the cation part of the emulsifier is soluble in bitumen leaving the aqueous phase acidic.

Non-Ionic emulsions are emulsions made from emulsifers which do not ionize in aqueous solutions.

Colloidal emulsions are those emulsions in which the emulsifer is a naturally occuring fine powder.

1.3 Objectives of the Work

The objectives of this study essentially are:-

(1) To review the main types of bituminous emulsions, the classes of emulsions, the productions and the role of the emulsifiers in their preparations.

(2) To review the uses of bituminous emulsions and to review some of the completed works where the bituminous emulsions have performed effectively.

(3) To review various tests available for bituminous emulsions as prepared by British Standard Institution.

(4) To investigate the role of Sodium Octadecanoate in anionic emulsion preparation and how ash-lye can be used as an alternative to Sodium Octadecanoate.

(5) To compare the basic properties of the anionic emulsions prepared using ash-lye as emulsifier to that of emulsions prepared by using Sodium Octadecanoate as an emulsifier, then use mathematical methods to make decision.

1.4 The Scope of the Thesis

It is within the scope of this work to concentrate only on the preparation of anionic bituminous emulsion, The work also uses the high speed mixer method of preparation as the basic method for preparation of the emulsion used for this work.

The bitumen used in the preparation was medium curing cut-back bitumen. Ash-lye was used as the only emulsifier without any additive. Only Classes A1 and A2 of anionic emulsions were used for investigation in this work.

The tests used in this work were tests recommended by British Standard Institution for emulsion.

The cost of using ash-lye in preference to other emulsifiers was not considered but it is evident that there will be a great reduction in the cost of production, since ashes are by-products of many burnt materials, e.g. specified types of wood, rice - husks, etc.

1.5 Assumptions

In this work the following assumptions were made.

(1) Bitumen referred to in this work is only refinery straight-run bitumen.

(2) If the emulsions prepared by using ash-lye pass the tests for emulsions as laid down in British Standard (BS 434), then it is believed that the emulsion will perform very well under field conditions.

(3) Only ashes from wood were considered e.g. Saw-dust or fire wood ashes.

(4) Test of hypothesis were carried out to determine whether the standard sample and the prepared sample belong to the same population.

1.6 Need for Research

The economic activity of developing countries is to a great extent dependent on an efficient transport system. A well-developed road network facilitate very rapid movement of persons and agricultural products, hence an adequate road network system becomes of prime importance.

In developing countries, unlike industrialised countries, unsurfaced roads constitute higher percentage in the national road network. The unsurfaced roads in these developing countries can even carry several hundred vehicles per day. This will allow unsurfaced roads of all types to play a vital role in the economic and social life of many of these countries, but such roads deteriorate easily.

The techniques of constructing and maintaining unsurfaced roads are thus an important part of road construction in developing countries. The interaction between initial construction standard and subsequent maintenance culture can seriously affect pavement deterioration. There is also a need to develop appropriate specification for locally - occuring building materials that are quite capable of providing satisfactory service.

There is also an urgent need to quantify the rate of deterioration of earth and gravel roads (unsurfaced road) in order to assess maintenance needs in economic terms and to plan cost-effective maintenance strategies.

Any road pavement deterioration is in the category of failure in which its failure will be disastrous to economic and social activities.

All these call for application of materials like bituminous emulsion that can be applied on the earth and gravel roads to keep them in good state for longer periods and for proper maintenance where needed. Therefore research on how to keep the cost of production and also produce a better quality of bituminous emulsion should be welcomed.

Furthermore with recent development, in the field of earth and gravel road construction, the widespread use of labour-intensive methods of construction in developing countries is becoming popular. In Asia and some African countries, labour - intensive methods have been employed on a large scale for road and many other civil construction projects . This was encouraged by the World Bank and the international Labour Organisation in the report of their recently sponsored studies. Harral et al (1977). In it, it was shown that the labour-intensive methods can in general produce the same quality of construction as equipment-intensive methods, and that they are economically justifiable in countries where the "shadow daily wage" (the wage paid but which does not reflect the true value of labour to the economy as in developing countries) for labour is less than about US$1.50 to $2.00 (₦17.00 to ₦22.00).

The spraying of bituminous emulsion can be done with hand or hand spray machines which encourages the labour-intensive method, therefore there is a need for further studies on the preparation and better performance of bituminous emulsions.

1.7 Methodology

Essentially, previous research efforts, texts and standards on bituminous emulsion were carefully studied. After these an experimental programme which involved preparations of ash-lye and emulsion were undertaken.

This experimental programme involved twelve samples of emulsions. After the preparation, the necessary anionic emulsion tests were carried out on each specimen and the results were compared with the requirements contained in British Standard (BS 434). The decisions were made based on the mathematical results.

Discussion and analysis of the test results were also based on the criteria given by British Standard Institution and conclusion and recommendations were made from the comparison and mathematical results obtained.

1.8 Organisation of the Thesis

Chapter one of the thesis introduce the major types of emulsions, the objective of the thesis, the scope of the work, the need for the research work and methodology used for the work.

Chapter two, reviews the related studies on the bituminous emulsions, types of emulsifiers, the production of emulsions, classification of emulsions and its uses.

Chapter three progresses on these early chapters by covering the theoretical back-ground of the work, the role of emulsifier in anionic emulsion, the type and properties of ash-lye, the behaviour of ash-lye in cut-back bituminous emulsion, the emulsion testing and their purposes. Chapter four discusses the materials used in the experimental design, preparation of specimens, testing of specimens, analysis of data and discussion of the results. Then chapter five gives, findings, conclusions and recommendations.

CHAPTER TWO

LITERATURE REVIEW

2.1 Bituminous Emulsion Geneology

An emulsion has been described as a two-phase system (RRL (1962)) which consists of two immiscible liquids the one being dispersed as finite globules in the others. The dispersed, or internal, phase is the liquid that is broken up into globules, and the surrounding liquid is known as the continuous, or external, phase. Special designations have been devised to indicate which is the dispersed and which is the continuous phase. Oil-in-water emulsions have the oil as the dispersed phase and water as the continuous phase. The reverse occurs when the emulsion is of the water-in-oil type. Almost without exception, road emulsions are of the oil-in-water type, the dispersed oil phase consisting of bitumen mixed together before emulsification.

The straight-run bitumen which is the main constituent is a valuable by-product obtained when petroleum is processed to obtain gasoline, kerosene, fuel oil, motor oil, and other petroleum products. There are three types of petroleum products found in the earth's crust which are:

(1) Bituminous - base crude oils

(2) Paraffin - base crude oils

(3) Mixed-base crude oils

Straight-run bitumen is easily obtained from bituminous base crude oils by a distillation process.

A material which resembles bituminous material may be obtained from paraffin-base crude oils by destructive distillation process involving chemical changes. These product materials should not be classified as bitumen. Straight-run bitumen may also be obtained from mixed-base crude oils, but the process is complicated. (Derucher and Heins (1981)).

All distillation of bituminous base petroleum is fractional, and during the distillation of petroleum several fractions are separated from the petroleum which include: Gasoline (light distillate), kerosene (medium distillate) Diesel oil (Heavy distillate), lubricating oil (very heavy distillate) and straight-run bitumen (residue).

Figure 2.1 shows a flowchart of the refining process necessary for the production of straight-run bitumen and bituminous emulsion.

When the process is controlled to prevent overheating and eventual chemical changes, the bitumen that remains as a residue is a straight-run bitumen. In the first operation the petroleum is pumped through the tube heater, in which the petroleum is placed under pressure and heated to a temperature of about 288^{o}C. This crude product, released through the bottom of the tube heater, enters an atmospheric fractionating column (tower distillation). Upon being exposed to atmospheric pressure within the column the more

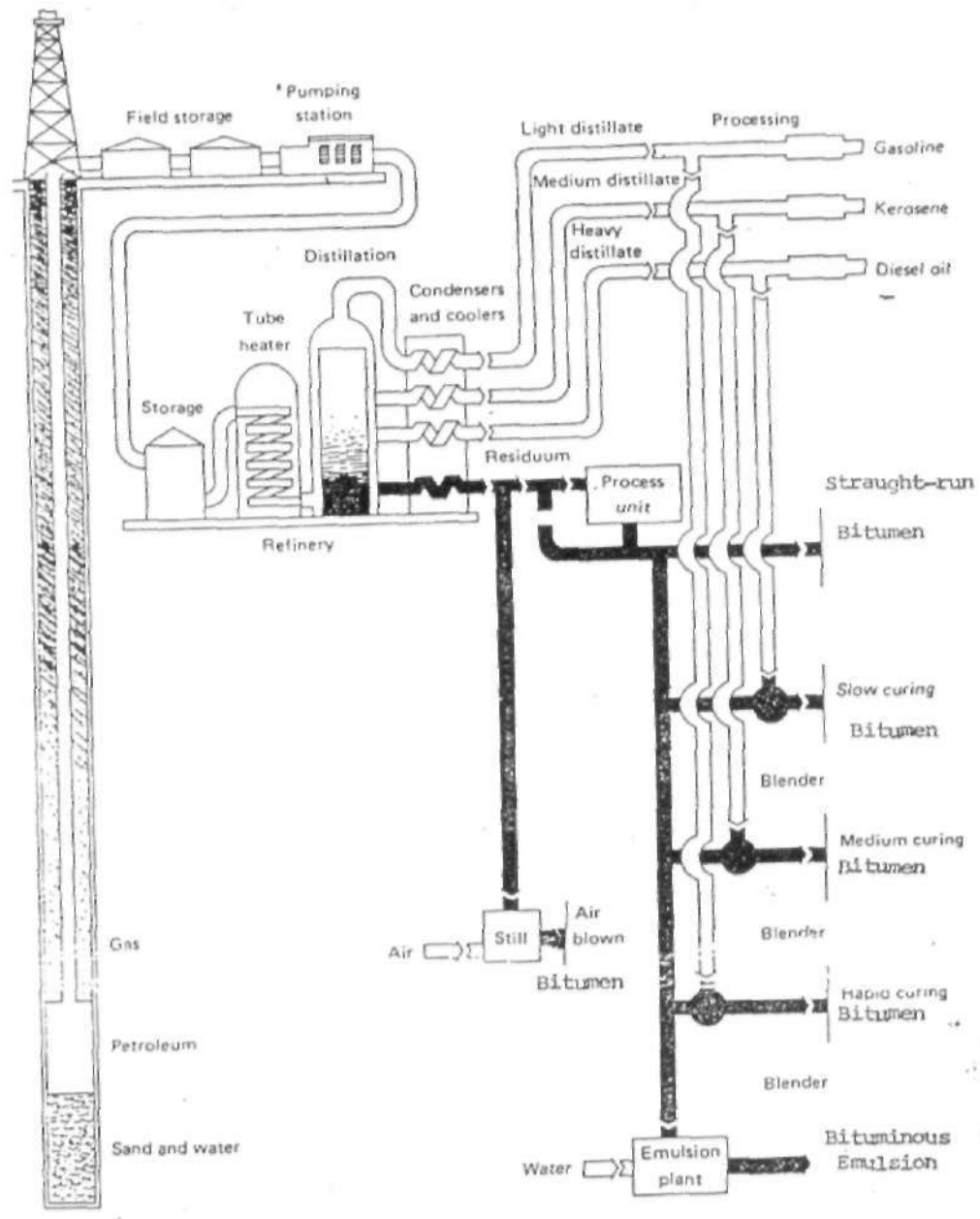


Figure 2.1 Flowchart for emulsion production process

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volatile fractions rise to the top of the column, where the vapours pass through condensers and coolers. Traps are arranged in the column at different levels where each trap is just below the boiling point of the liquid at which it is to collect. Once the vapour passes through appropriate traps, the vapours are condensed and cooled. These condensed vapours are referred to as distillates (Light, medium, and heavy). After further processing, gasoline, kerosene and diesel oil result. The product that settles down in the atmospheric fractionating column is the residue.

This product will then enter a second fractionation column. This procedure is the application of a partial vacuum in the fractionation column. The residue that results from the second fractionation column is straight-run bitumen. When volatile diluent like gasoline or kerosine diesel are added to straight-run to make liquid product, the product is referred to as cut-back bitumen. This can be rapid, medium or slow cut-back depending on whether, gasoline, kerosene or diesel are the volatile diluents.

To obtain bituminous emulsion, the straight-run bitumen or cut-back bitumen will pass through emulsion plant, where water and emulsifier will be added to it so as to form emulsion. In the production, the emulsifiers to be added has to be done in small proportions both to facilitate the formation of dispersions and to keep the globules of dispersed binder in permanent suspension.

2.2 Emulsifier

Various emulsifiers have been used for the preparation of emulsions and their results have been satisfactory. Among these emulsifiers are Sodium Octadecanote Hexadecatrimethyl-ammonium bromide, esters, ethers of fatty acids and alcohols, casein, gelatine fine powders such as clays and bentonites (RRL (1962)). Ruscheve et al (1964) reported the preparation of bituminous emulsion with various emulsifiers and some additives. Emulsifiers used include potassium hydroxide, soap, sodium hydroxide, naphtheric acid, casein and ethyl alcohol.

Also in the German Democratic Republic, Lueder et al (1973) reported synthetic product of constituent quantity as a new type of emulsifier which also serves as stabilizer.

These emulsifiers are usually classified according to the nature of their dissociation in water which in turn determine the type of emulsion. Thus emulsifiers for road emulsions are classified as Anionic, Cationic, Non-Ionic and collodial emulsifiers. Thus Anionic Emulsifiers are organic compounds which are characterised by a large organic anion forming a salt with an alkali (soap). A typical example is Sodium Octadecanoate. $(CH_3(CH_2)_{16}COONa$

Cationic emulsifiers are compounds in which it is the cation of the large organic fraction of the molecule on dissociation that is soluble in the bitumen.

An example of such emulsifier is Heradeca-trimethyl ammonium bromide ($C_{16}H_{33}(CH_3I_3NBr$).

Non-Ionic Emulsifiers are emulsifiers which do not ionise in aqueous solution. These types of emulsifiers are not widely used in the production of road emulsions. Examples of such emulsifiers are ethers or ethers of fatty acids.

Colloidal Emulsifiers are emulsifiers which form colloid with water. They naturally occuring fine powders. They are usually used for production of emulsion for industrial purposes than for road emulsions. An Example of this emulsifier is bentonite. The role of the emulsifier in the emulsion preparation is to keep the globules of dispersed binder in permanent suspension.

With an emulsifier present, an absorbed film of the emulsifier is formed around each globule in the emulsion, this film acting as a protective coating which affords a considerable resistance to coalescence of the globules.

2.3 Preparation of Emulsions

2.3.1 Materials

Almost all grades of bitumen can be emulsified, from the hard penetration grades (10/20 penetration) to softer grades of straight-run bitumen (400/500 pen). Hader grades of bitumen are more difficult to emulsify and the emulsions made of 190/210 pen or 280/320 pen bitumen find the greatest use in road making especially for surface-dressing and grouting work. RRL (1962). This then makes the British Standard

Institution BS 434(1973) to recommend bitumen that comply with BS 3690 which is also within the grade range of 70 to 300 penetration to be used in the production of emulsions. For pre-mixed materials which are required to stand in Stockpiles for some time before use, emulsions made from cut-back bitumens are commonly used.

2.3.2. Methods of Preparation

There are two empirical methods used for the preparation of road emulsions. They are:

(i) The colloid mill method

(ii) The high-speed mixer method.

The main difference between the methods is that with the colloid mill the emulsion is produced continuously whereas with the high-speed mixer a number of separate batches are produced. Power requirements also differ, more power being required for comparable output in the case of colloid mill.

2.3.2.1 Colloid Mills

The type of colloid mill generally used consists essentially of a high-speed rotor which revolves in a stator, the clearance between the rotor and the stator being approximately 0.5m. The rotor is driven at a speed to produce a velocity at the edge of the rotor of about 50m/s.

The main emulsifying effect depends upon the hydraulic shearing forces exerted by the rotating surfaces upon the particles of bitumen in the presence of an aqueous emulsifying liquid.

A hot solution of the emulsifier in water, and the heated bitumen, are fed separately at a constant rate into the machine in the appropriate constant proportions so that an emulsion of uniform binder content is continuously produced. The product of the emulsion prepared by this method has been observed to have a mean particle size of approximately 1 to 2 microns. The divergence from the means depending upon the apparatus used and the condition of emulsification. RRL(1962).

To maintain a uniform product a careful control of constituent condition is essential. Consideration must also be given to the following points when designing plant for the production of road emulsions; the colloid mill must be driven so that it will run at a constant speed, and the bitumen and emulsifier solution must be maintained at constant temperatures so that the viscosity of the liquids passing through the machine will be constant.

2.3.2.2 High-Speed Mixer Method

This method is a batch process and it involves running appropriate amount of water at just below boiling point in a mixer, the diameter of which is at least equal to the total depth of liquid it is supposed to mix. The mixer is fitted with a high-speed propeller-type stirrer mounted off-centre to avoid the production of a vortex.

Emulsifier is then added to the water in the mixer, and bitumen at about 100°C is slowly run in with continuous stirring after which any necessary stabilizer may be added. Particles obtained by this method may be less than 1 micron in diameter whilst some may be more than 10 microns in diameter. RR(1962).

In their specification, Krmmev et al (1959) recommend that if a paddle mixer is used for mixing, a high concentration will be obtained at a low rate of mixing (80 to 100 rev./min). So also the emulsion produced with this type can be easily transported and then diluted on the site easily.

Other methods that have been developed in USSR are:

(1) Acoustic method in which the production plant is employing ultrasonic vibration for the preparation of bitumen emulsions. This ultrasonic method was said to be simpler and safer and even more economical than other techniques. (Bogelsarov et al (1962)).

(2) Resonance Method. Voladimirov (1963) shows that satisfactory results were obtained with experimental production equipment developed in USSR. For the preparation of bitumen emulsions by this method, a 400 litres tank and pipe system were first heated to 60 or 70°C and 100 to 180 litres of water at 50-60°C was circulated as emulsifier was being introduced. The water was then pumped from a nozzle in a special dispersion unit in the lower half of the tank on to the end of a fixed cantilever steel strip until resonance was obtained.

Bitumen at 140 to 145°C was then added at 30 to 40 litres per minute. A continuous dispersion system was then developed.

After emulsification by any of these methods, the material is pumped into storage tanks where it is allowed to cool. It is advantageous in overcoming 'skinning' difficulties if the final product can be cooled as rapidly as possible and storage tanks are frequently fitted with stirring mechanism and cooling coils for this purpose.

With the colloid mill it is possible to preclude the entry of air which again tends to reduce the formation of skin in the finished emulsion.

2.3.3 Stabilizers

For normal road emulsion the percentage of emulsifier used is about one-half to one percent by weight calculated on the finished emulsions. This amount affords a reasonable protection against coagulation of the binder particles, but for some uses it is necessary for added protection to be given and the addition of further agents is required. Emulsions are classified into groups, according to their resistance to coagulation of the finished emulsion. The groups are 'labile emulsions', which contain a minimum of emulsifier 'semi-stable emulsions', which are more highly stabilized and 'fully stable emulsions' that contain even higher proportion of stabilizer.

These stabilizers can either be added during the manufacture of the emulsion or can be added at a later stage. Examples of commonly used stabilizers are Casein and potassium soap.

2.4 Classification of Bituminous Emulsion

Generally, road emulsions are classified according to their order of stability or bre king. In British Standard BS 434(1973) only anionic and cationic emulsions were classified.

For anionic road emulsions the clasification are:

(1) Class A1: Labile Emulsion. This is an emulsion characterised by rapid breakdown on application and normally unsuitable for mixing with aggregate and it is used cold.

(2) Class A2: Semi-stable emulsion: This is an emulsion with sufficient stability to permit mixing with certain grades of aggregates before breakdown occurs It is also used cold.

(3) Class A3: Stable Emulsion: This is an emulsion with sufficient mechanical and chemical stability for all purposes involving mixing with aggregates including those containing large proportions of fines or chemically active materials such as cement or hydrate lime. It is also used cold.

(4) Class A4: Slurry seal: This is an emulsion specially formulated for the slurry seal process with the addition of water, sufficiently stable to form a free - flowing slurry with the aggregate specified and capable of sustaining this condition throughout the laying procedure adopted. Setting time of the mix may be within a few minutes or extended as desired. Special proprietary emulsions may be included in this class.

(a) Class A4, slow setting slurry seal: This is slurry suitable for mixing in simple mixers for hand laying, in bulk transit concrete mixers or mobile mixing machines.

(b) Class A4, rapid setting slurry seal: This is slurry suitable for handling in special mobile mixing only.

For cationic road emulsions the classification are:

(1) Class K1, Rapid acting emulsion: - This is an emulsion characterised by rapid deposition of binder on contact with road surfaces and aggregates followed by early resistance to rain. Unsuitable for mixing with aggregates. For high binder content grades used hot.

(2) Class K2, Medium acting Emulsion: - This is an emulsion in which the rate of deposition of binder is sufficiently delayed to permit mixing with certain clean coarse aggregates, before breaking to form a continuous adhesive film without stripping: It is used cold.

(3) Class K3, Slow acting Emulsion: This is an emulsion in which the rate of deposition of binder is sufficiently delayed to permit mixing with certain fine aggregates before breaking to form a continuous adhesive film without stripping. It is used cold. This class also includes Cationic Slurry Seal emulsion which are suitable for handling in special mobile mixing machine.

2.5 Uses and Application of Bituminous Emulsion

The use of bituminous emulsions are numerous partly because they exist in different classes and partly because they can be applied while cold. The uses of emulsions in road construction and maintenance are based on their rate of breaking (setting).

Class 1, Labile Emulsions and Rapid Setting Emulsions

They are used for surface dressing, grouting, tack coating, concrete curing, more specialised uses are as a slip coat to prevent bonding between successive layers of concrete, and as a protective mulch when seeding verges and embankments with grasses. Greatly improved germination is claimed as the film of binder retards loss of water from soil by evaporation.

For Class 2, Semi-Stable Emulsions and Medium Setting Emulsions

They are used mainly for retreading, pre-mixing, seal coating, crack treatment and track coating. In using these emulsions for pre-mixing one should guard against having too high proportion of fine material because it can cause premature breaking of the emulsion.

For Class 3, Full-Stable Emulsions and Slow Setting Emulsions

These emulsions are used for the following:- Pre-mixing, tack coat, mist-spraying. These emulsions can also be used for making slurry seal with fine sand or quarry fines. Soil stabilisation is also carried out with these emulsions. Slurry seal can be used for treating airfield and road surfaces by applying a slurry composed of a fine

aggregate and bitumen emulsion which seals cracks and to provide even riding surface or a base for further treatment like surface dressing, thin carpet coat or friction courses. Rubber can be added to bituminous emulsion in other to produce an emulsion that will be less sensitive to temperature changes after breaking. Bituminous emulsions are also used in the agricultural sector for paving barns, mulches, and as disinfectants.

In building, as Damproofing, water proofing, covering insulating fabrics, asbestos felt, laminating roofing, acoustical blocks, architectural decoration, bricks, and blocks building.

In Hydraulics and erosion control; for canal, dam or reservior lining embankment protection, dam groutings, levee protection and sand-dune stabilization.

In industries it is used for making Aluminum foil, clutch facing panel board, battery boxes, electrical insulating compound, black grease, embalming, painting inks, impregnated, treated materials, paints, and varnishes.

In his work, chaffin W. (1974) called for substitution of emulsion for cut-backs bitumen and investigated various way in which bituminous emulsion may be useful. He also admitted that emulsion is an ideal material to rubberlize since latex is also an emulsion, if the emulsion and rubber carry the same charge. In his report Keinert, H. (1971) gave detail of the practical tests carried out since 1966 with bituminous emulsions. It was found that surfaces made with this type of emulsion were still perfect after several years.

It was also established that acid resistant containers should be used for manufacture of cation - active emulsions and that long heat-treatment should be applied in order to attain perfect emulsification and the emulsifying agent should also be stabilized, for emulsions to be durable. This is in line with RRL recommendation (RRL (1962)).

In his own report on the use of bituminous emulsions in South Africa, Jenkin (1972) gave detail on how surface treatments and stabilization of road bases were achieved. He also gave specifications for the main grades of anionic and cationic emulsions and slurries produced in South Africa which can be used for both new construction as well as maintenance in order to achieve best results.

Langumer (1985) in his own finding on how to overcome cracks, points out that when a layer of emulsion treated materials is inserted between a hydraulic mix sub-base this bituminous surface layer prevents setting and shrinkage cracks. Such structures make it possible to benefit from the advantages of hydraulic mixes, exhibiting little deformation and prevent unavoidable setting and thermal shrinkage cracks. When Luszanski (1973) discussed how to reduce the difficulties and cost of repair and maintenance of coated roads in Poland, he recommended the use of bituminous emulsions in order to facilitate repair operations while still lowering the cost. Emulsions do not require pre-heating and they bind aggregates very quickly. Emulsions and slurry seals can be easily be applied for maintenance operations. Writing on maintenance of surfacings Pujadas (1972) gave an example of the use of slurry

in Bacelonia, where excellent results were obtained. "A slurry was used which contained granitic aggregates and portland cement as additional filler." He reported that only a thin layer of slurry seal was applied and it did not reduce the height of the foot-path above the carriage-way. Further in their work on how to prepare rapid-curing bituminous slurry for the use of road repairs Dybalok et al (1971) wrote that it can be prepared by first Prewetting fine - grained aggregates with a water - solution of anionic surfactant and then mixing the wetted aggregate with emulsion of the bitumen in an aqueous solution of a cationic emulsifier.

Thus, a fine limestone aggregate mixed with 2 percent weight of Portland cement was wetted with 8 percent weight of water (weight are base on aggregate) containing 0.25 percent of sodium dioctylsulfosuccinate. A 16 percent bituminous emulsion containing 60 bitumen, 40 water and pentamethytrimethylene diammonium dichloride of 0.8 percent was blended into the wetted aggregate to form a slurry. It was then discovered during testing that a layer of slurry, applied to a surface cured in 45 minutes, whereas a slurry made without the use of anionic surfactant required 180 minutes to cure.

Concerning the effective construction of bituminous surface treatment (BST) Faro and Yukon (1984) said the technique commonly accepted and understood by most provincial and territorial highways department is not just squirting of bituminous emulsion to a surface and covering it with some gravel, but the BST construction

involving many approaches, applications and techniques. The basic technique that can be generally adopted is the use of either rapid setting emulsions and chip aggregates or high float emulsions and well-graded aggregates. The right approach is to analyse the expected traffic volumes, the condition of sub-base, the quality and quantity of aggregate available, the type of emulsions available and the cost of installing should also be undertaken.

Uppal (1971), in his study on how to make use of stabilized soil to build houses in India, reported that the hand moulded bricks made by using bituminous emulsion was not only economical but also effective. It prevents the penetration of water to the wall surface therefore the wall needs no plaster or any other protective coating.

CHAPTER THREE

THEORETICAL BACKGROUND

3.1 Constituents of Bitumen

Bitumen which constitues a major components in emulsions is made up of complex chemical elements. The Chemical nature of the crude oil from which bitumen is derived shows a very wide difference reflecting the different geological conditions of their formation.

The separation of bitumen into fractions by means of solvent e.g. carbon disulphide has played a large part in research on its constitution. Most bitumen used in road making have insoluble 'carboids' which are not more than 0.1%. This very small fraction has an effect on the properties of bitumen and it serves as an index of the degree of 'cracking' or breaking.

There are other systems for bitumen analysis but most of them divide the bitumen soluble in carbon disulphide into three main fractions which are:

(1) Carbenes: - Fraction insoluble in carbon tetrachloride;

(2) Asphaltene:- Fraction insoluble in light aliphatic hydrocarbon solvent such as petroleum ether.

(3) Mallenes:- Fraction insoluble in light aliphatic hydrocarbon. Carbenes is of no use in road bitumen making. The maltenes fraction is often sub-divided into a fraction absorbed on an active power (such as fuller's earth)

or insoluble in a second solvent and referred to as 'resins' and a fraction known as 'Oils'. The use of this separation has thrown light on the relation between the constituents of a bitumen and its properties. The analysis of this type has also shown that the oily part of bitumen is similar in chemical nature to lubricating oil but they have higher molecular weight.

Asphaltene are large and high-molecular-weight hydrocarbon fractions precipitated from asphalt. Asphaltenes have a carbon-to hydrogen ratio of 0.8.

The hydrocarbons in road bitumen are principally of the following forms:

(1) Saturated aliphatic group or paraffins;

(2) Naphthenic groups or cycloparaffins;

(3) Aromatic ring compounds.

Many properties of bitumen, particularly the non-Newtonian flow properties that are often exhibited, suggest that bitumen is a colloidal system. The colloidal concept of bitumen is attributed to the presence of asphaltenes which is in association with high molecular-weight materials of lower C/H ratio. This complex is normally referred to as the 'micellar' phase the lower molecular weight maltenes forming the continuous 'intermicellar' phase. On the basis of evidence from flow properties, bitumen can be divided into two types; 'Sol' type, in which there is little interaction between micelles or a 'gel'

type in which the quantity and association of the micelles are great enough to cause a loose structure formation. The type of structure taken up by a bitumen depends largely on the chemical nature of maltene fraction. If this is much more naphtenic or paraffinic in character than asphaltenes, then its peptizing power is less and a gel type structure will develop. Most distilled road bitumen are 'sol' type.

Although it is common practice to speak of bitumen as complex mixtures of hydrocarbons, studies have shown that bitumen contains elements other than carbon and hydrogen, for example sulphur and also oxygen. And perhaps the most important of these elements are those which confer acidic properties on the molecules. It is this acidity that is of importance for the performance of the material as a road binder even though this varies considerably from different sources.

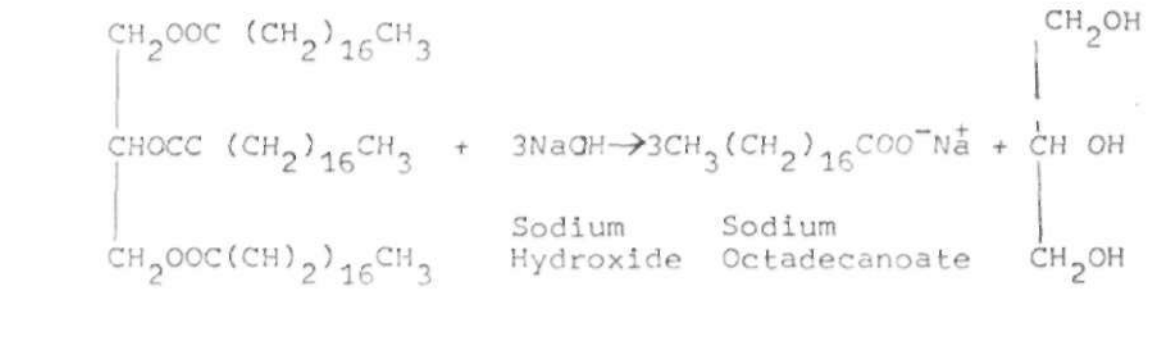
3.2 Anionic Emulsions

Anionic emulsions are emulsions produced by using anionic emulsifiers. These types of emulsifiers are characterised by large organic anion forming a salt with an alkali. In other words they are soaps. Example of such is Sodium Octadecanoate($CH_3(CH_2)_{16}COONa$).

3.3 Soaps

Soaps are Sodium or Potassium salts of long-chain carboxylic acids (Fatty acids). Soap is formed by hydrolysis of an ester by alkali. The hydrolysis is carried out by boiling harder fats (esters) such as allow, mutton fat, palm, palm kernel, coconut, cotton-seed, soya-bean, hydroganited oils etc. with an alkali such as Sodium or Potassium hydroxides. Potassium soaps are milder than Sodium soaps.

A typical reaction is



Propan-1,2,-triyl triociadecanoate

Propane-1,2,3-triol

The soap formed shows a different property from the fatty acid because it is ionic rather than molecular. Thus they are salts that are soluble in water and their non-polar end soluble in oil or greasy substances. Soap is a mixture and its solution possesses a high degree of electrical conductivity in dilute or concentrated solution. (Strong (1952)).

3.4 Emulsifier for Anionic Emulsion

When soap like sodium octadecanoate dissolves in water this dissociates into the (negative) Octadecanoate anion ($CH_3(CH_2)_{16}COO^-$)and the (positive) sodium cation (Na^+). The long-chain fatty-acid octadecanoate anion is soluble in bitumen, the carboxylic group (COO^-) which carries the negative charge being the least soluble part. In consequence when sodium octadecanoate is present in a system of globules of bitumen in water, a condition which may be represented by the diagram below (figure 3.1) sets up.

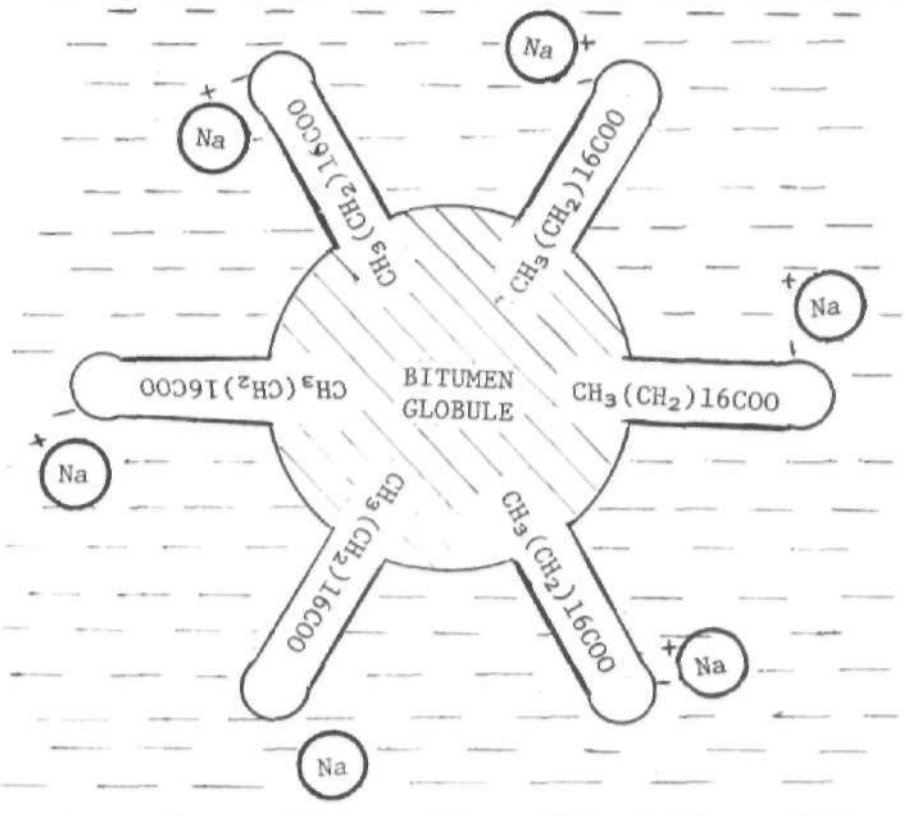


FIGURE 3.1

Diagrammatic representation of the arrangement of emulsifier around dispersed bitumen globules in an anionic emulsion.

Each bitumen globule is therefore surrounded by an absorbed film of octadecanoate ions with a negative charge on the surfaces, and it becomes much more difficult for the globules to coalesce because all have their surfaces negatively charged and so tend to repel one another. Water used for preparing anionic emulsion must be a distilled one. If hard water is use it will affect the production of emulsion because soap will form 'scum' with calcium ions in the hard water. This scum is an insoluble calcium salt of carboxylic acid.

Anionic emulsions are alkaline with PH values of 9 or more. These emulsions can be neutralized by cationic emulsions which have opposite ionic charges.

3.5.0 Breaking Mechanism of Bituminous Emulsions

3.5.1 Particle size of Dispersed Binder

The average diameter of the globules of bitumen in a road emulsion is about 2 microns. The stability and other important properties of the emulsion will depend not only on this average size but on the distribution of the particle sizes about this mean. The presence of larger particles can be a serious disadvantage because they act as centres for rapid coagulation.

3.5.2 Mechanisms

Road emulsions are produced in the first place with sufficient stability to enable them to be transported and applied without premature coagulation of the particles of binder. Immediately the emulsion has been applied to the

road. It is essential that it should break down, that is, the particles of binder in the emulsion should separate from the aqueous component and coalesce into coherent film of binder as rapidly as possible. This first visible film is given by a change in the colour of the film from the chocolate brown of emulsion to the black of the bituminous binder. The binder is by no means free from water at this stage and has not developed its full binding power. The rate of breakdown of a road emulsion is dependent on the following factors:-

(i) Evaporation of water: - After application of an emulsion to a road, evaporation starts with the same rate as for pure water with similar conditions of temperature, humidity, wind, general site conditions and method of application. When coagulation begins to occur evaporation slows down.

(ii) The porosity of the surface to which it is being applied and consequent removal of water by capillary attraction.

The loss of water by capillarity is very important when aggregate such as blast furnance slags are used. Chippings which are excessively dusty, accelerate the rate of breakdown of road emulsions but the use of such chippings is to be avoided as adhesion between the stone and binder film will be greatly reduced.

(iii) The chemical and physical influence of aggregate with which the emulsion comes in contact; This effect, has a limited effect on the rate of break down of anionic emulsions. The effect is due to the interaction between the surface of the stone and protective film of emulsifier

around the dispersed particles of binder and local coagulation of the binder occur before the emulsion has reached a concentration state.

(iv) The mechanical disturbance of the emulsion and aggregates: - As evaporation of the water continues the bitumen particles become closely packed and coalescence starts. This happens when the proportion of binder in the emulsion has risen to between 80 and 90%. It is at this stage that break down may be accelerated by mechanical disturbances. The stresses set up in the emulsion and aggregate system by rolling sometimes cause further coalescence of the bituminous binder. Before this stage, the evaporation of the water is reversible. Therefore if it rains the applied emulsion will be diluted and even washed away, and action of traffic will cause displacement of chippings. Also under cold and humid conditions of road emulsion which has partly broken down will fail to hold chippings satisfactorily under the action of traffic. And after emulsion has completely broken up there exists on the road a continuous film of bright black binder adhering to the underlying surface.

In his work, Bohn (1965) reported that the determination of the amount of bituminous emulsions that breaks on its application depends on the acidic action of the stone. The decisive factor is the reaction between the emulsifier solution, and the stone.

3.6 Classification of Anionic Emulsion.

British Standard (BS 434) classified anionic emulsion into four main classes namely class A1 liable emulsions, class A2, semi-stable emulsions, Class A3, stable emulsions and class A4, slurry seal. These main classes are also subdivided according to their binder content with exception of A3 and A4, for example A1-60, has 60% norminal binder content in the emulsion. So in all there are seven types of anionic emulsions in accordance to British Standard which are A1-60, A1-55, A1-40, A2-57, A2-50, A3 and A4.

3.7 Bituminous Emulsion Testing and Their Purposes

The following tests pertaining largely to emulsions are covered by BS 434 (1973). The tests are:-

(1) Residue on 710 μm BS Sieve

(2) Residue on 150 μm BS Sieve

(3) Stability to mixing with coarse aggregate

(4) Stability to mixing with cement

(5) Binder content

(6) Viscosity

(7) Coagulation of emulsion at low temperature

(8) Storage stability (short period test)

(9) Storage stability (long period test)

(10) Particle charge

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Residue on Sieving

Since the molecules of bitumen are larger than that of water and it is inevitable that the globules of binder in emulsion will tend to sediment. The rate at which sedimentation occurs is largely dependent on the size of the particles. It is to control the percentage of large particles in bitumen emulsions that the residue - on - sieving clauses are inserted in BS 434. The effect of the clauses are to ensure that not more than 0.05 percent by weight of the emulsion consists of particle greater than 0.71mm in diameter and not more than 0.15 percent by weight of the emulsion consists of particles greater than 0.45mm in diameter; this is to safeguard against excessive sedimentation during storage.

Stability to Mixing With Coarse Aggregates

The test included in the standard is designed to give a quantitative appraisal of stability of emulsions to mixing with aggregates and is intended to be used to differentiate between the liable emulsions which show a high degree of coagulation under these circumstances and the semi-stable and stable type which coagulate to a lesser extent.

Stability to Mixing with Sand

Stable emulsions should have sufficient mechanical and chemical stability for all purposes involving mixing with aggregates which includes those containing large

proportions of fines. Cement is used as a standard fine aggregate and the test in BS 434 gives limits of coagulation for semi-stable and stable types of emulsions.

Binder Content

The bitumen may constitute between 40% to 60% of emulsion. It is essential to know this percentage if the quantity of bituminous binder actually used in the surfacing is to be measured accurately. The binder content (including emulsifier) is the difference between 100 percent and percentage of water content.

Viscosity

The viscosity of an emulsion is a measure of the flow properties of the emulsion itself and has no relation to the viscosity of the dispersed bitumen or tar. It is determined mainly by the proportion of bitumen in the emulsion and by the particle-size distribution. The viscosity of the emulsion should be low enough to spray through conventional jets or to coat stone in a simple mixer, but at the same time the emulsion must be viscous enough not to drain off road during spraying or from the stone in mixing work.

Storage Stability (Short period test)

Emulsion must be stable enough. Some sedimentation may occur when emulsion container is left standing before use; provided, however, the sediment redisperses on agitation, the emulsion can be used satisfactorily.

Storage Stability (long period test)

When stored emulsion in a container under normal atmospheric conditions the emulsion should not separate in a form which cannot be redispersed by agitation. The test stipulates storage for three months at temperature within the range 5^{o} to $30^{o}C$.

Particle Charge

This test identifies the type of bitumen emulsion. Positive charge indicates that the emulsion is catonic, negative charge indicates that it is anionic. This is done by passing a direct current through a bitumen emulsion between two parallel plate electrodes. Deposition of bitumen on one of the electrodes indicates whether the emulsion is of the anionic or cationic type.

Coagulation of Emulsion at Low Temperatures

As all emulsion contains water they are affected by extremes of heat and cold. Exposure to temperatures below $0^{o}C$ will result in freezing and the degree of recovery on thawing depends on the type of emulsion. The test in the standard gives an indication of the reaction of an emulsion to freezing and thawing under standardised conditions.

The requirements for anionic road emulsions from the British Standard (BS 434) is shown in table 3.1.

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REQUIREMENTS OF ANIONIC ROAD EMULSIONS

TABLE 3.1

PROPERTY	CLASS OF ANIONIC EMULSIONS						
	A1-60	A1-55	A1-40	A2-57	A2-50	A3	A4
Residue on 710 µm (% by mass maximum)	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Residue on 150µm (gram per 100 ml maximum)	0.15	0.15	0.15	0.15	0.15	0.15	0.15
Stability to mixing with coarse aggregate (% coagulation)	20-80	20-80	20-80	20-80	20-80	20-80	20-80
Stability to mixing with cement (% coagulation)	-	-	-	2	2	2	-
Binder content (Minimum % by mass)	58	53	38	55	48	55	56
Viscosity (^{a}Engler 20°C)	6-9	5-8	4 max.	8 max.	5 max.	9 max.	8 max.
Coagulation of emulsion at low temperature	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Storage stability (Short period test) (Inversions to clear sediment, maximum)	60	60	60	60	60	60	60
Sotrage stability (Long period test) (Water content difference % maximum)	2	2	2	2	2	2	2
Particle charge	neg.	neg.	neg.	neg.	neg.	neg.	neg.

3.8.0 Theoretical Background of Anionic Emulsion Using Ash-lye As Emulsifier

Anionic emulsions are produced by using soap as an emulsifier. (Jackson et al (1984)). Soap is produced by boiling caustic lye with fat or oil. Vita (1988) also reported that soap can be produced with leach ashes and oil. Ashes: Ashes can be got from all vegetable growths as well as coal, peat and lignite when they are burnt. All of them are said to contain some amount of alkali. (Strong (1952)). Ashes are said to be made up of potassium oxide (K_2O) especially wood ash. The ashes of plant materials amount to approximately 4%. (Strong (1952). It was also estimated that the amount of potassium oxide (K_2O) in an ashes varies from 10 percent in the ashes of straw to 42 percent in hard wood. Potassium oxide, sodium oxide, calcium oxide magnesium II oxide, Iron III oxide, sulphur (in form of sulphants), silica, carbon IV oxide and tetraoxophosphate IV acid are also associated with varying amount in ashes. It was also reported that the following elements decrease in order of abundance, namely, potassium, sulphur, magnesium, phosphorus silicon, calcium, sodium, iron, aluminum, chlorine and magnanese. (Strong (1952)). When ash is dissolved in cold water the potassium oxide which is the main constituent reacts with water to form alkali (potassium hydroxide).

$$K_2O + H_2O \longrightarrow 2KOH$$

It is this potassium hydroxide that is referred to in this thesis as ash-lye. If this lye reacts with hot oil it will form potassium soap, which is soft soap.

Ellis (1981) recommended hard wood ashes for the best lye. Vita (1988) said ashes of burnt seaweed are particularly useful as these ashes produce a sodium - based lye from which hard soap can be made. And ashes from other materials such as paper, cloth or garbage can also be used to produce soap but the output will be small.

RRL (1962) recommended potassium soap as stabilizer for emulsion, This is to protect emulsion particles from coagulation. He also gave the quantity of emulsifier needed for emulsion as 0.5% to 1.0% by weight of calculated finished emulsion.

Theory behind this work centres on the amount of oil present in the cut-back bitumen. When this amount mixes with hot alkaline (ash-lye), sufficient quantity of soap needed as emulsifier for the production of anionic emulsion will be obtained.

CHAPTER FOUR

EXPERIMENTAL PROGRAMME AND DATA ANALYSIS

Experimental Programme

In accordance with the objectives of this study an experimental programme, based on factors involved in emulsion preparation and level of interest were carried out. These deal with the experimental design, materials specimens, testing procedures and analytical approach as described here in after.

4.1 Experimental Design

In order to minimize the number of tests and to facilitate interpretation of the results, the experimental design was based on the factors involved and the level of interest as applicable to tropical conditions.

4.1.1 The Factors Involved and Levels of Interest for a Bituminous Emulsion Preparation

Studies on bituminous emulsions have indicated that their performance depends on; sieve analysis, Binder content, viscosity, storage stability to mixing with coarse aggregate and particle charge analysis. RRL (1962).

For the purpose of this study the above properties of anionic emulsions were studied in relation to such factors as well as amount of bitumen and concentration of the emulsifier used. However other dependent

factors such as preparation temperature, mixing rate and time of mixing, application of stabilizer or additive, storage methods were kept constant. In effect the only two variables allowed are:

(i) Amount of bitumen

(ii) Concentration of emulsifier.

In other to study the two factors above the recommended range by Road Research Laboratory (RRL (1962)) was taken into consideration. The amount of emulsifier was kept between 0.5% to 1.0% of weight of the emulsion which corrs-pond to 1.0 to 3.0 concentration of ash-lye. The amount of the bitumen used for the study was kept between 38% to 62% of weight of emulsion in accordance to British Standards Institution recommendation (BS 434). Therefore the levels of interest utilised for obtaining the concentration of emulsifier effect on the anionic emulsion was to vary increasingly at an interval of 1.0% of the concentration of ash-lye from 1.0% to 3.0%. Thus three levels of interest were studied which are 1.0%, 2.0% and 3.0% concentrations of ash-lye.

To study the properties of anionic emulsions produced four levels of interests, were also adopted by varying the amount of bitumen content in the emulsion prepared. Thus 40%, 50%, 55% and 60% content of emulsion were used in accordance with nominal bitumen content for classes A1 and A2 of anionic emulsion of BS 434 (1973).

4.12 Phases of Experiments

A systematic approach of design based on the following phases were adopted:

Phase I: - Finding the level of significances of each of the tested property.

Phase: II - Examining the effect of concentration of emulsifier on the properties of emulsion prepared.

The nature of specimens used and the number of experiments performed were indicated in table 4.1 below.

Table 4.1 The Nature of Specimens and the Number of Experiments Performed

Binder content % of weight of Bitumen	Concentration of Ash-lye 1%	2%	3%	Total
40%	7	7	7	21
50%	8	8	8	24
55%	8	8	8	24
60%	8	8	8	24
Total	31	31	31	93

4.2 Materials Used

The various materials used in this study were as described below.

4.2.1 Ashes

In determining the ashes used in the extraction of ash-lye for this study, a preliminary investigation of ashes of some materials were conducted viz-a-viz, wood, grass and paper. Thus properties of Iroko wood, shear butter wood, sturbbon grass and paper ashes were determined.

The results of the tests conducted are given in Appendix A. The results show that high concentration of ash-lye can be got from wood, followed by grasses and finally papers.

This result is in line with what Strong (1952), Ellis (1981) and Vita (1988) said in their publications, that wood ashes produced strong-lye than any other ashes.

From this preliminary study, shear butter tree ashes were selected for this work. These ashes were used because the PH of the lyes are far more than 9.0 as recommended by Road Research Laboratory (RRL, 1962), which signify that it can be confidently used at the above stated concentration. Secondly shear butter tree is a common wood at the locality in which this experiment was conducted. The wood is used as fire-wood which implies that the cost of getting the wood is not high.

Thus shear butter tree ashes obtained from both Suleiman Hall and Ribadu Hall cafeterias of Ahmadu Bello University Zaria, Nigeria were used.

Table 4.2 below contains properties of shear butter tree ash-lye used.

Table 4.2 Properties of Ash-lye Used

Properties	Values
Type of wood burnt	Shear butter tree
Period between burning and experiment	12 - 24 hours
Unit weight	2.03 g/cm^3
Solution properties:-	
PH of 1% concentration	10.1
PH of 2% concentration	10.3
PH of 3% concentration	10.4
Electro-Potential different(mv) of:-	
1% Concentration	205
2% Concentration	220
3% Concentration	240
Boiling poing of ash-lye	97^oC
Solubility of ash-lye at S.t.p.	7.09/100g

4.2.2 Bitumen

The bitumen used was MC2 type of cut-back bitumen obtained from Civil Engineering Department, Transportation Laboratory of Ahmadu Bello University.

Table 4.3 below contains the Shell specifications for the binder.

Table 4.3: - Shell Specification for the Binder Used

Properties	Shell Specification
Viscosity bolt furol (60°C) (Sec.)	100-200
Flash point (Cleveland Open Cup Open Cup)°C	65
DISTILLATION	
Distillate to 225°C % volume	10
Distillate to 260°C % volume	15-55
Distillate to 316°C % volume	60-87
Residue from distillate to 360°C % volume	67
TEST ON RESIDUE AFTER DISTILLATION	
Penetration at 25°C (0.1mm)	120-300
Ductility, at 25°C (cm)	100
Solubility in CC¼	99.5

4.2.3 Aggregates

The aggregates used for the tests, were found around Civil Engineering Department, washed and air dried. They are crushed, rough and angular aggregates of granite origin.

4.3 Preparation of Specimens

The procedures employed in the preparation of the emulsion specimens are stated below:

Step I

The ashes collected after burning shear butter tree wood were passed through three different seives No.35, 52, and 200 BS Sieves. This was done under 24 hours after burning the wood.

Step II

The fraction that passed through Sieve BS 200 were collected and kept in a closed plastic container to avoid the ashes from absorbing moisture from the atmosphere.

Step III

A known weight of ashes was then dissolved in a known quantity of distilled water for 72 hours. At the end of 72 hours the solution was then passed through a centrifuge machine to obtain a clear solution of the ashes called Ash-lye. The residue from centrifuge cups was then collected and air-dried for a week after which it was weighed.

Step IV

The amount of ashes that dissolved in a known quantity of water was determined. The required volume of distilled water needed to make the different required concentration of ash-lye were added.

To avoid chemical reaction between the lye and the containers only glass and plastics containers were used for the storage of the lye.

Step V

In the preparation of the specimen emulsions the empirical batch methods (High-speed mixer methods) (Kraney (1959) and Vladimirous (1963)) of bituminous emulsion preparations were used as bases.

A known weight of bitumen which has been heated to a temperature of between 105°C to 110°C was run into the required concentration of ash-lye at a temperature of between 65°C to 70°C. The mixer were then thoroughly stirred with stirrer at the rate of 80 to 100 revolutions per minute for about 3 - 5 minutes. Each batch of emulsion which was about 1 litre in volume was prepared in the storage container. After the preparation, the containers were cooled rapidly by putting the containers in a tank of cold water. This was to limit 'Skinning' on the surface of the prepared emulsion. It was important (before the final storage of the emulsion) that any skin that appears on the surface of the prepared emulsions were removed and the containers were closed very well. This was to avoid the coagulation of dispersed emulsion particle which in turn would lead to breaking or setting of the emulsion.

4.4.1 Specimen Notation

The specimens prepared were labelled with a letter A and three figures. The letter A meant anionic emulsion the first figure signified the percentage concentration ash-lye while the last two figures gave the nominal percentage of binder content. Thus A1 - 60 signified anionic emulsion of one percent concentration of ash-lye and 60 percentage of binder content in the emulsion.

4.4 Testing Procedures

To each of the specimen prepared the following tests were carried out viz: particle charge determination; determination of residue on 710μm BS sieve; determination of residue on 150μm BS sieve; determination of stability of mixing with coarse aggregate; determination of water and binder content; viscosity determination; determination of storage stability (short period tests) and determination of storage stability (long period test). The tests were performed in accordance with British Standard BS 434.

The apparatus, materials, procedures and methods of reporting the results of the tests are given in Appendixes B to J. of BS 434 (1973).

The summary of the results obtained after the tests are as stated in Table 4.4

Table 4.4 Summary of Test Results on Prepared Emulsion

Serial Number	Specimens	Particle Charge	Residue on 710µm BS Sieve (Percentage by mass)	Residue on 150µm BS Sieve (Percentage by	Stability to mixing with coarse aggregate (% Coagulation)	Binder Content Percentage by mass	Viscosity (°Engler at 20°C	Storage Stability short period test (Inversion to clear sediment)	Storage Stability long period test (water content difference Percentage)
1	A1-40	Negative	0.070	0.20	53	40.6	4.4	45	-
2	A1-50	Negative	0.06	0.24	47	50.5	4.9	52	1.8
3	A1-60	Negative	0.05	0.30	46	55.5	7.0	50	1.8
4	A1-60	Negative	0.06	0.25	41	60.4	9.2	65	2.4
5	A2-40	Negative	0.04	0.28	38	41.2	3.0	45	-
6	A2-50	Negative	0.04	0.21	49	50.9	4.5	55	7.4
7	A2-55	Negative	0.04	0.20	58	55.9	7.2	60	1.5
8	A2-60	Negative	0.03	0.10	58	60.8	7.7	60	1.9
9	A3-40	Negative	0.06	0.25	67	41.8	6.2	50	-
10	A3-50	Negative4	0.04	0.28	55	51.5	6.4	60	2.1
11	A3-55	Negative	0.02	0.30	48	56.4	9.1	55	2.1
12	A3-60	Negative	0.05	0.30	44	61.2	9.8	75	2.8

Table 4.5 Summary of Expected Standard Results for Anionic Emulsion

No.	Specimens	Particle Charge	Residue on 0.710mm BS Sieve (Percentage by Mass (Maximum)	Residue on 0.150mm BS Sieve Percentage by mass (Maximum)	Stability of mixing with coarse aggregate (% coagulation)	Binder Content (Maximum Percentage BY Mass	Viscosity (^{o}Engler at 20^{o}C	Storage stability short period test (inversions to clear sediment Maximum)	Storage stability Long period test water content difference percentage Maximum
1	A1-40	Negative	0.05	0.15	20-80	38	4 max.	60	-
2	A1-50	Negative	0.05	0.15	20-80	48	5	60	2
3	A1-55	Negative	0.05	0.15	20-80	55	5-8	60	2
4	A1-60	Negative	0.05	0.15	20-80	58	6.9	60	2
5	A2-40	Negative	0.05	0.15	20-80	38	4 max.	60	-
6	A2-50	Negative	0.05	0.15	20-80	48	5	60	2
7	A2-55	Negative	0.05	0.15	20-80	53	5-8	60	2
8	A2-60	Negative	0.05	0.15	20-80	58	6-9	60	2
9	A3-40	Negative	0.05	0.15	20-80	38	4 max.	60	-
10	A3-50	Negative	0.05	0.15	20-80	48	5	60	2
11	A3-55	Negative	0.05	0.15	20-80	53	5-8	60	2
12	A3-60	Negative	0.05	0.15	20-80	58	6-9	60	2

4.5 Characteristics of Specimens

The emulsions prepared were dark brown in colour. They were less viscous than the bitumen used for the preparation of the emulsion. A drop of the emulsions on a glass t .ed black after a short period and sticked to the glass. A drop left for a day on glass plate became a solid drop and it was difficult to remove.

After three months when long period stability tests was to be performed most of the specimens needed to be stirred very well in order to get a good sample out of the specimen.

4.6 Analysis of Data

The mathematical analysis carried out on the test results involved the null hypthesis that both the standard samples and the prepared samples were from the same population. Therefore the main analysis carried out were to estimate the mean and variances of individual population and thereby estimate the variance difference of the population. Hence the mean differences being found and its significance was determined and checked from standard table.

For any two samples of whose details are given below were considered.

	Sample 1	Sample 2
Number of observation	N	n
Mean	A	a
Standard deviation	D	d
Standard Error	$DN^{-\frac{1}{2}}$	$dn^{-\frac{1}{2}}$

For each population their variance estimate are D^2N^{-1} and d^2n^{-1}. The estimated variance of the population of differences is $D^2N^{-1} + d^2n^{-1}$. Thus the standard error of the difference is $(D^2N^{-1} + d^2n^{-1})^{\frac{1}{2}}$.

Furthermore the mean of the differences is

$$\{A - a\}$$

If sample 1 and sample 2 belong to the same population, the mean of the differences should not differ significantly from P. Where P is significant level from standard table.

Thus $$\frac{|A - a|}{(D^2N^{-1} + d^2n^{-1})^{\frac{1}{2}}} \not> P$$

This expression is only meant for large samples for small samples it is necessary to replace the above expression by

$$\frac{A - a}{S\ (N^{-1}+n^{-1})^{\frac{1}{2}}} \pm tp$$

where S is an estimate of the standard deviation of the parent population from which both samples were drawn, and tp is the value in t-distribution table at p level of significance.

The procedure followed for the investigation of the difference between the mean of the two samples (Standard samples and prepared samples) which are small and are from the same population with the same variance were as follows:

(1) Determination of the sum and the sum of the square of each specimen in the different sample.

(2) Finding the means of each sample (A and a)

(3) Finding the variance of each sample from the formular.

$$(\text{Sum} - 1)\ s^2 = \Sigma x^2 - \frac{(\Sigma x)^2}{\text{sum}}$$

Where X is the result of each specimen.

(4) The variance of the population was determined by the formular.

$$S^2 = \frac{(N-1)\,D^2 + (n-1)d^2}{N + n - 2}$$

(where all notation were as stated earlier).

(5) Standard deviation (S) was got by finding square root of S^2.

(6) The significance of the mean difference was calculated from

$$\frac{|A - a|}{S\,(N^{-1} + n^{-1})^{\frac{1}{2}}}$$

This value must not be greater than tp. where tp was got from t-distribution table given in the Appendix B.

The degree of freedom (v)=N + n - 2, if the null hypothesis that the samples were from the same population is to be accepted.

The above analysis was performed on each numerical properties of the prepared emulsions. Taken standard results as sample 1 and tested result as sample 2.

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Table 4.6 Summary of the Analysis of Prepared Emulsion

	Mean			Variance							
	Standard Sample (1)	Test Sample (2)	Mean Difference	Standard Sample (1)	Test Sample (2)	Population Variance	Population Standard Deviation	Standard Error	Calculate tp	95% level of significance from Table t-distribution	Remark
Residue on 710μm BS Sieve	0.05	0.047	0.003	0	0.002	1.03x10-4	1.02x10-2	4.16x10-3	0.72	2.08	Accept
Residue on 150μm BS Sieve	0.15	0.243	0.092	-	0.037	0.00167	0.041	0.0167	5.56	2.08	Reject
Stability to mixing with coarse aggregate	50 (Class mean)	50.33	0.33	0	740.67	33.67	5.802	2.369	0.139	2.08	Accept
Binder Content	49.25 (Nominal)	52.23	0.98								Accept
Visco-sity	5.75	6.62	0.87	21.75	50.28	3.27	1.81	0.739	1.177	2.08	Accept
Storage Stability (Short Period test)	60	55.83	4.17	0	841.67	38.26	6.19	2.53	1.65	2.08	Accept
Storage Stability (Long Period test)	2.00	1.98	0.02	0	1.77	0.111	0.333	0.157	0.127	2.59	Accept
Particle Charge	Negative	Negative	-	-	-	-	-	-	-	-	Accept

The calculation sheets for the analysis are attached in Appendixes C1 to C7. The summary of the results got are given in table 4.6.

4.7 Discussion of Results

4.7.2.1 Emulsion Identification (Particle Charge Test)

From the results obtained from particle charge tests and recorded in table 4.4. All samples were negatively charged, which confirmed that the emulsions prepared were anionic emulsions.

4.7.2.2 Binder Content: - The binder content of the prepared specimens exceeded both the minimum binder content and the nominal binder content, that is specified in the standard (BS 434). The result of the binder content of the specimens prepared showed that on the average it is about 1.0 percent greater than nominal binder content and about 3.0 percent of emulsions total weight greater than minimum binder required. The values of the binder content obtained from experiment on each specimen are giving in Table 4.4 and Figure 4.7, while both the minimum and nominal binder requirement together with calculated results are given in Appendix C1 and Figure 4.7. These results show that each prepared specimen meets the minimum requirement.

Furthermore since on the average the prepared specimen also exceeded the nominal binder content this implies that the specimen prepared contain more binder content thus there is tendency for the viscosity of the prepared specimens to increase above the required value.

4.7.2.3 Residue on Sieving Through Sieve 710um

Tables 4.4, 4.5 and Figure 4.1 show the specimens results and standard values respectively. These results show that the residue of the prepared sample after passing through sieve 710um, ranges between 0.02 to 0.07 percent of the total weight of the emulsion. They have 0.047 of total weight of the emulsion on the average as residue which is less than the maximum average of 0.05 percent. t-tests performed on the specimens results at 95 percent level of confidence showed that emulsion prepared from ash-lye, on sieving the emulsion through sieve 710um will not exceed maximum requirement with an error not more than 0.05 percent of the total weight of emulsion. The calculation of this analysis is given in Appendix C2.

4.7.2.4 Residue on Sieving Through Sieve 150um:

From the results of the tested specimens shown in Table 4.4 together with the standard results shown in Table 4.5 and Figure 4.2, the value of tested results ranges between 0.10 to 0.30 percent of the total weight

of the emulsion; giving an average of 0.24 percent for all the prepared specimens as against 0.15 percent of maximum limit allowed in the standard (BS 434).

This shows that the emulsion prepared contain more particles that are larger than 150μm in diameter than the required amount. This means that the specimens may be more viscous than the standard specimen and that they are prone to coagulation early if they stand for a long period. In other words, the best result can be achieved only if the specimens are used within three months from the day of preparation. The mathematical analysis performed on the results shows that the standard error of the difference in the mean of the prepared specimens and the standard specimens is0.0167 percent of the total weight of emulsion and the level of confidence in using this emulsion is not up to 95%. The calculation for the mathematical analysis for this test is shown in Appendix C3.

4.7.2.5 Stability to Mixing with Coarse Aggregates:-

Tables 4.4, 4.5 and Figure 4.3 show the specimen results and the standard values, then both respectively. These results show that the percentage of coagulation of the prepared specimens ranges between 38 and 58 which is within the interval of the range specified by the standard (BS 434) that is 20 to 80. This implies that when mixing coarse aggregates with these prepared

emulsions the breaking down of the emulsion and coating of the aggregates with bitumen will not take place too early in the mixing cycle. But it will occur at moderate rate. The mathematical analysis performed on the test results is as indicated in Appendix C4. The standard error of the difference between the mean of the prepared specimens and the standard results is 2.37 percent of coagulation, and that the emulsion will pass stability to mixing with coarse aggregates test with 95% confident.

4.7.2.6 Viscosity:- From the results of the viscosity test performed on the specimen at 20°C shown in Table 4.4 together with required standards shown in Table 4.5 and Figure 4.4, only emulsions A1-50, A1-55, A2-40, A2-50, A2-55 and A2-60 meet the standard requirement which are 4.0, 5.0, 8.0 and 9.0° Engler for emulsions A-40, A-50, A-55 and A-60 respectively, while specimens A1-40, A1-60, A3-40, A3-50, A3-55 and A3-60 which have viscosity value of 4.4, 9.2, 6.2, 6.4, 9.1, 9.8 respectively did not meet the standard requirement.

The increase in the viscosity may be attributed to increase in the binder content of the prepared specimens above the nominal binder content specified. It may also be due to the particle size of the prepared emulsion which contain more than the required particle size greater than those that pass through sieve 710μm but retained on 150 MUsieve.

In their recommendation RRL (1962) specified that an emulsion in which the viscosity lies between 6 and 13 ^{o}Engler can be sprayed by bulk spraying machine while emulsion with viscosity range between 6 and 9 ^{o}Engler can be sprayed by hand operated spraying machine. With hand application, viscosity range between 10 to 20^{o} Engler will be adequate. Going by this recommendation, it shows that most of the results got are within the range. When mathematical analyses were performed on the tested results, the result showed that the emulsion prepared will flow readily with 95% level of confidence and the error that may result will not be more than 1.11^{o}Engler.

4.7.2.7 Short Period Storage Stability:- From the results shown in Table 4.4, Table 4.5, and Figure 4.5 for tested results, standard results and both results respectively, the range of short period stability measured ranges between 45 to 75 as against 60 for maximum value. The average of tested specimens is 55.83 of short period stability measurement. This means that the specimens prepared will be stable enough on the average; if they are left standing in a closed container for a short period before use. This was confirmed by the mathematical analysis performed on tested results as shown in Appendix C6, which indicated that the stability of the emulsion prepared by the above method and materials can be assured with 95% level of confidence error that will be associated with it will not be more than 2.53 of the standard short period storage stability measurement.

4.7.2.8 Long Period Storage Stability:- For long period stability only specimen As-50, As-55 and As-60 were considered and the results got are as shown in Table 4.4 together with required standard in Table 4.5 and graphical view in Figure 4.6. The results got range between 1.4 to 2.8% of the total weight of emulsion. On the average, the differences in the water content of the prepared specimens for a period of three months from the time of the preparation was not more than 1.98 percent of total weight of the emulsion as against 2.0 percent allowed.

This implies that only few of the prepared specimens can be relied on when they are stored in a closed container for a period of three months at a temperature within the range of 5^{o} to 30^{o}C under atmospheric conditions. The result of the mathematical analysis confirms that this can be achieved with 95% level of confidence with an error not more than 0.10% of the total weight of emulsion.

From the above discussion and the summary of the analysis as indicated in Table 4.6. It implies that, with an exception of residue test on sieve 150µm the samples of anionic emulsion produce by the use of ash-lye as emulsifier can be used with a confidence limit of 95% in tropics.

4.7.2.9 Performance of Ash-lye Concentration:- Figure 4.1 to Figure 4.7 show pictorially the deviation of the tested results from standard values and Table 4.7 shows the number of specimens which meets the standard and the number of specimens which failed to meet the standard against the ash-lye concentration.

One can easily conclude that 2% of ash in the ash-lye solution performed very well within the experimental limit follow by 1% and lastly by 3%. This is because out of 31 tests performed with 2% concentration, 28 passed while 3 failed as against 3% concentration in which out of the same number of the tests performed 18 passed and 13 failed. In the case of 1% concentration 19 passed and 12 failed. Figure 4.8 also shows these deviations against the expected number.

Table 4.7 Performance of Ash-lye concentration in each property tested of prepared Emulsion

Properties Test	1% concentration of Ash-lye		2% concentration of Ash-lye		3% concentration of Ash-lye		Total
	Number that passed	Number that failed	Number that passed	Number that failed	Number that passed	Number that failed	
Residue on 710µm BS Sieve	1	3	4	0	3	1	12
Residue on 150µm BS Sieve	0	4	1	3	0	4	12
Stability on mixing with Coarse aggregate	4	0	4	0	4	0	12
Viscosity	1	3	4	0	0	4	12
Storage stability (short Period)	3	1	4	0	3	1	12
Storage stability (long Period)	2	1	3	0	0	3	9
Binder content	4	0	4	0	4	0	12
Particle Charge	4	0	4	0	4	0	12
	19	12	28	3	18	13	93
	31		31		31		93

Percentage of Passes

1% Concentration = $\frac{19}{31}$ x 100% = 61.29%

2% Concentration = $\frac{28}{31}$ x 100% = 90.32%

3% Concentration = $\frac{18}{31}$ x 100% = 58.06%

Total No. Passed = $\frac{19 + 18 + 18}{93}$ x 100% = 69.89%

FIGURE 4.1 EFFECT OF ASH-LYE CONCENTRATION ON RESIDUE ON 710μm BS SIEVE FOR PREPARED EMULSION

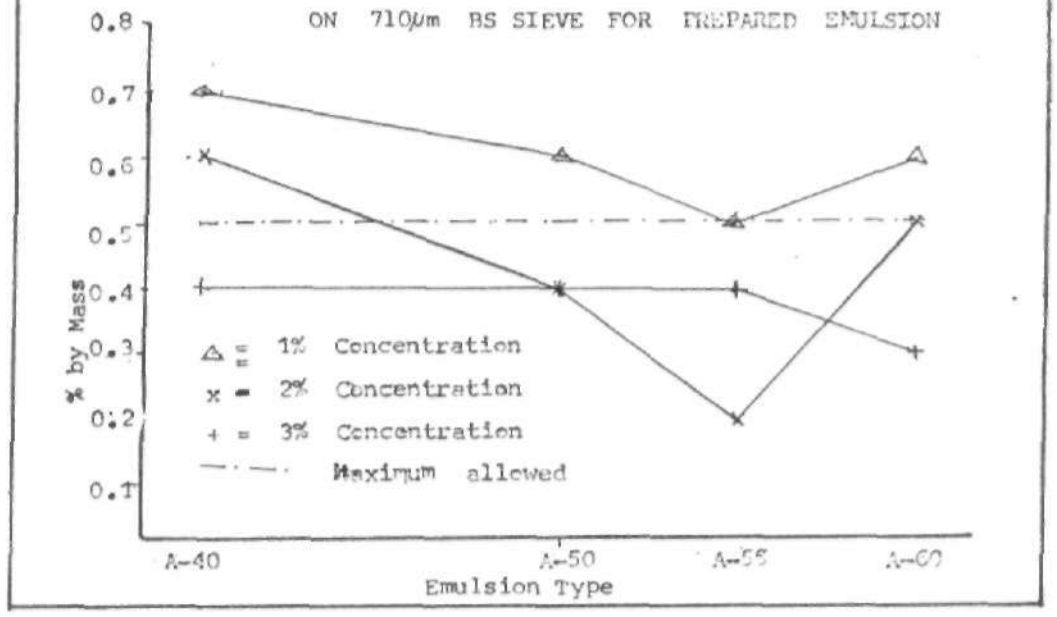
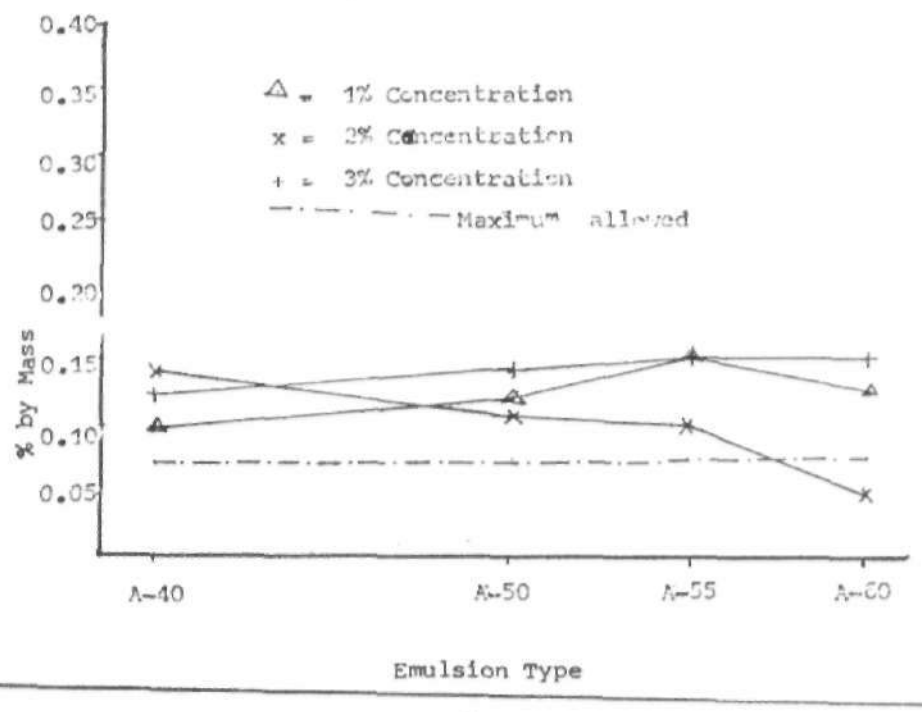
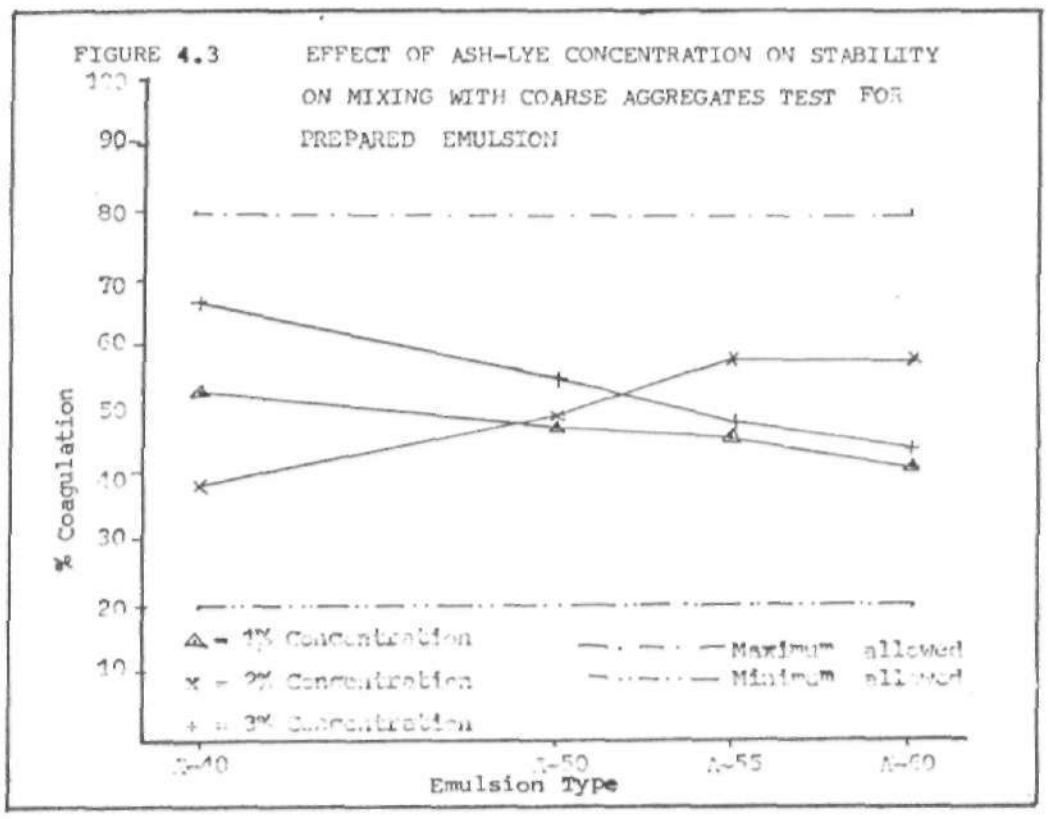


FIGURE 4.2 EFFECT OF ASH-LYE CONCENTRATION RESIDUE ON 150μm BS SIEVE FOR PREPARED EMULSION




FIGURE 4.3
EFFECT OF ASH-LYE CONCENTRATION ON STABILITY ON MIXING WITH COARSE AGGREGATES TEST FOR PREPARED EMULSION
% Coagulation
90
80
70
60
50
40
30
20
10
Δ = 1% Concentration
× = 2% Concentration
+ = 3% Concentration
Maximum allowed
Minimum allowed
A-40
A-50
A-55
A-60
Emulsion Type

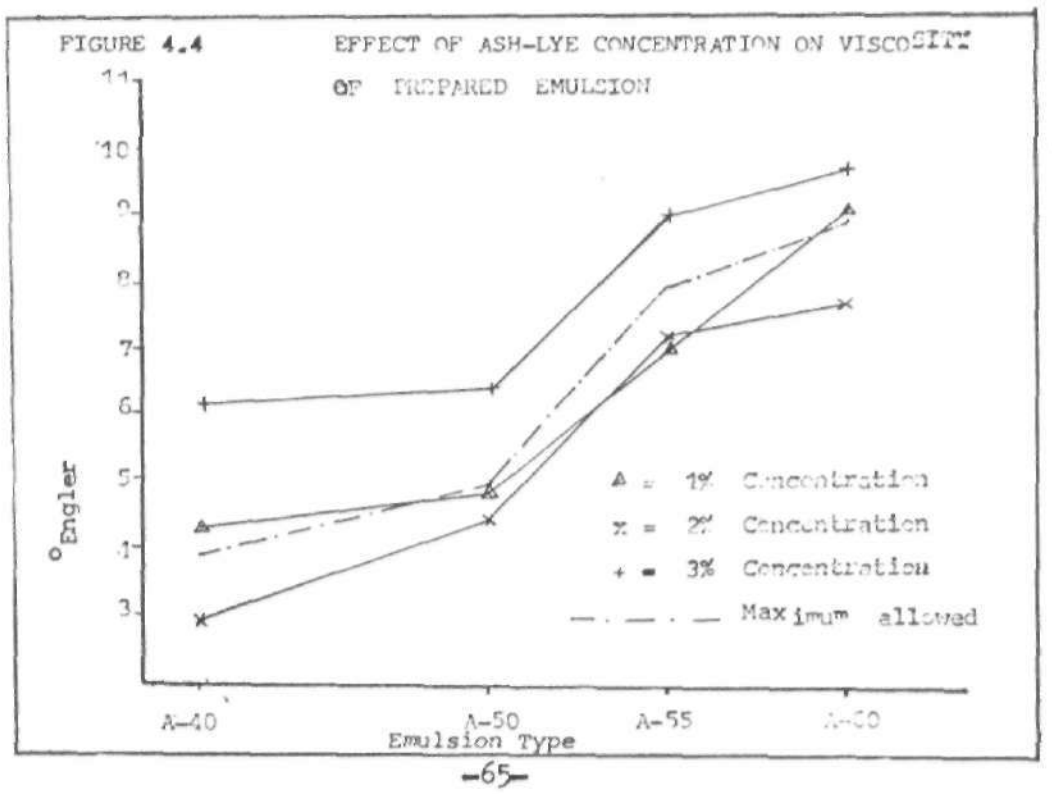

FIGURE 4.4
EFFECT OF ASH-LYE CONCENTRATION ON VISCOSITY OF PREPARED EMULSION
°Engler
10
7
6
5
3
Δ = 1% Concentration
× = 2% Concentration
+ = 3% Concentration
Maximum allowed
A-40
A-50
A-55
A-60
Emulsion Type

FIGURE 4.5 EFFECT OF ASH-LYE CONCENTRATION ON STORAGE STABILITY (SHORT PERIOD TEST) OF PREPARED EMULSION

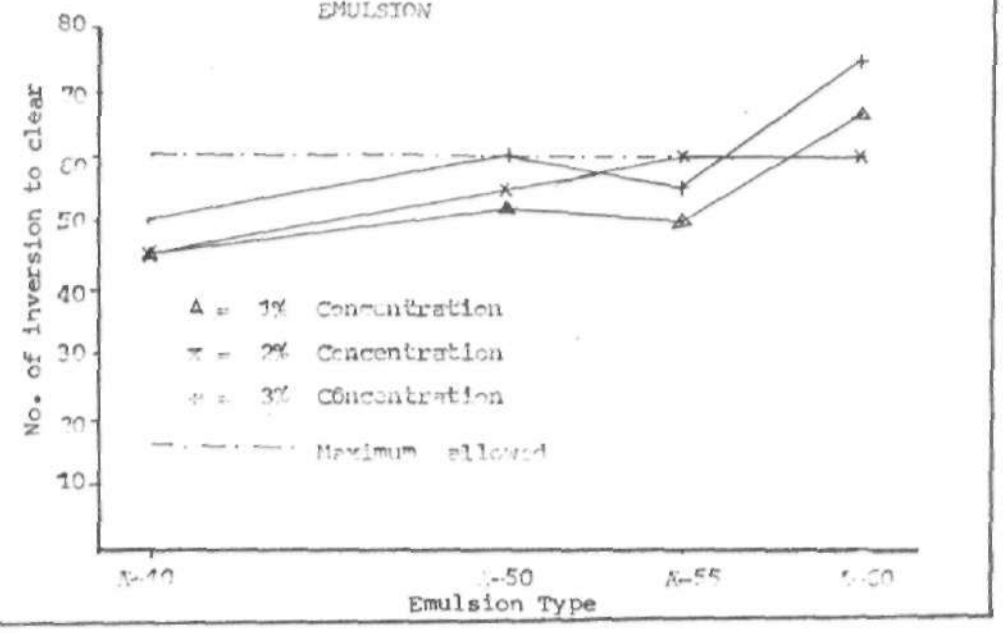
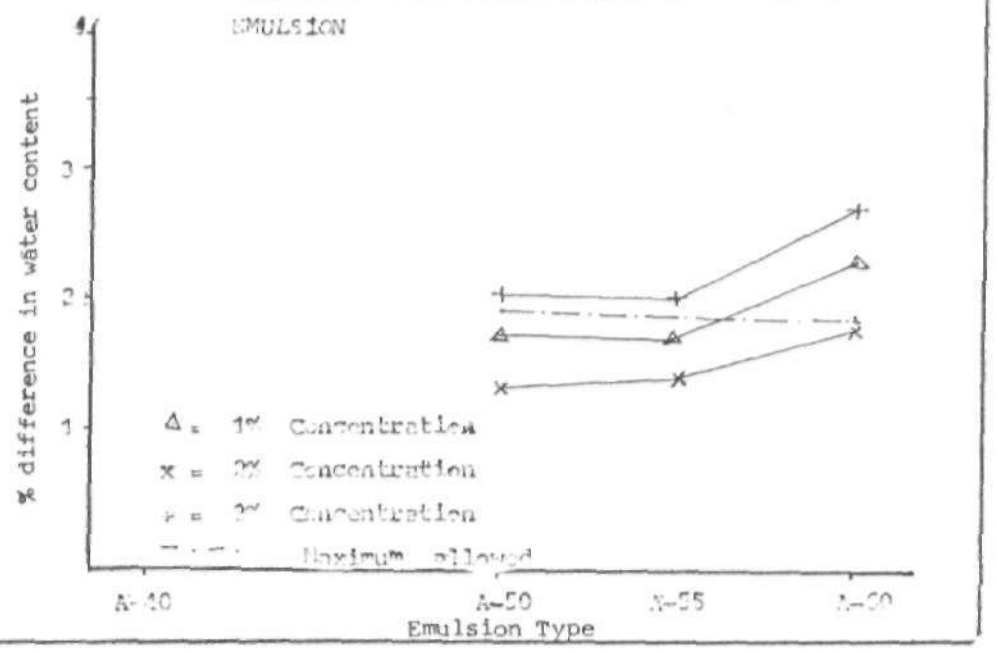
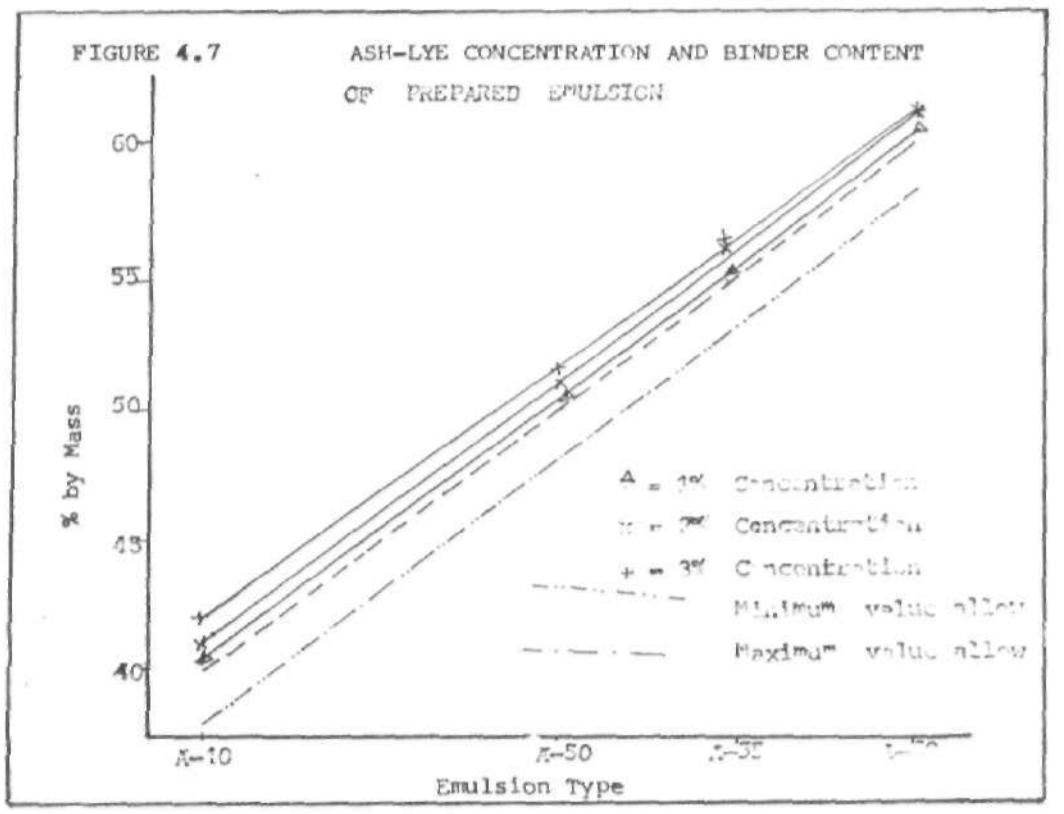
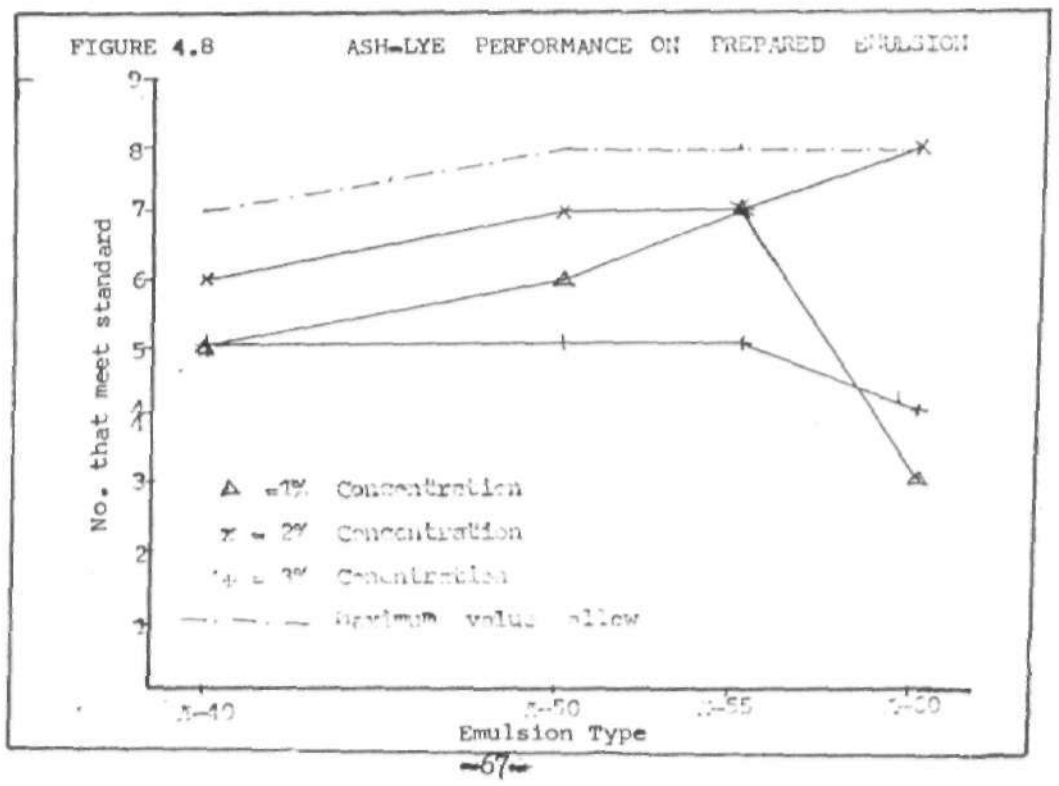


FIGURE 4.6 EFFECT OF ASH-LYE CONCENTRATION ON STORAGE STABILITY (LONG PERIOD TEST) OF PREPARED EMULSION




FIGURE 4.7
ASH-LYE CONCENTRATION AND BINDER CONTENT
OF PREPARED EMULSION
% by Mass
60
55
50
45
40
A-10
A-50
Emulsion Type
Maximum value allow


FIGURE 4.8
ASH-LYE PERFORMANCE ON PREPARED EMULSION
No. that meet standard
9
8
7
6
5
4
3
2
1
Emulsion Type

CHAPTER FIVE

5.0 FINDINGS, CONCLUSIONS AND RECOMMENDATIONS

In summary, the findings, conclusions and recommendations on this work is as stated below.

5.1 Findings

Within the limits of experimental error, weather influence and other variables, the following findings on the study were made:

(1) That these three materials; bitumens, ashes and water can be used to prepare emulsion.

(2) That distilled water and wood ashes produced better results when used as emulsifier.

(3) That heating of bitumen that will be used for the preparation of emulsion above 120°C (when batch process method is to be adopted) should be avoided because this will cause the water in the ash-lye to evaporate by gaining heat from the bitumen while mixing thereby making the resulting emulsion to be more viscous than the required standard.

(4) Excess and rigorous mixing must also be avoided during preparation if batch process is in use because it will cause the water molecules in the ash-lye to gain kinetic energy which will be converted into internal energy hence to latent heat of evaporation, consequently making the emulsion to be more viscous.

(5) Prepared emulsion should be stored properly in a container that is air tight so as to avoid setting

CONCLUSIONS

On the basis of the experimental programme executed and the analysis of the results discussed in Chapter 4 together with the review of existing knowledge on the bituminous emulsions and ashes. The following conclusions can be made:

5.2.1 (1) Ash-lye made from wood of shear butter tree when used beneficially in place of Sodium Octadecanoate (soap) as emulsifier in bituminous emulsion making, will produce an emulsion which will pass on sieving through sieve 710μm sieve tests, the stability to mixing with coarse aggregates tests, the viscosity test, the storage stability for both short and long period tests and the particle charge test.

The results of mathematical analysis also show that the above tests can be achieved with 95% confidence limit.

5.2.2 (2) The results obtained in chapter 4 reveals that the best results can be got by using 2% concentration of ash-lye.

5.2.3 (3) The result got from sieve analysis using sieve 150μm over the emulsion prepared indicated that the emulsion contained more large particles than the required amount.

5.2.3 (4) Though the work did not take into consideration the cost implication of the process, it is believed that the use of ash-lye as an emulsifier in the production of anionic emulsion will be viable, cost wise.

5.3 Recommendations

(1) Ash-lye is recommended as an emulsifier for the production of anionic emulsions; subject to the following further research works.

(2) A detailed study of the effect of the mixing temperature for the components.

(3) Detailed study on the rate and the duration of the mixing during production of emulsions.

(4) Further study on the optimum concentration of ash-lye for the production of bituminous emulsions.

(5) Field performance of the emulsion prepared using ash-lye as emulsifier.

(6) The performance of anionic emulsion using ash-lye as emulsifier with American Standard.

(7) Detailed study on the method that can be adopted for its production in a rural area.

(8) Study on the economic effect and deforestration impact.

(9) Economic study of using ash-lye as emulsifier.

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APPENDIX A

	Shear Butter Tree			Iroko Tree			Gross(Sturbbon Gross)			Paper		
Ash-lye concentration	1%	2%	3%	1%	2%	3%	1%	2%	3%	1%	2%	3%
P H	10.1	10.3	10.4	9.4	9.7	9.8	8.8	9.0	9.2	7.4	7.8	8.0
Electro-Potential different (MV)	205	220	240	160	180	180	120	130	145	60	80	110
Specific gravity	2.03			1.87			1.49			1.12		
Solubility	7.0			5.7			6.0			8.7		
Boiling Point	97°C			97°C			97°C			97°C		

Appendix B

Percentage points of the t-distribution

P	25	10	5	2	1	0.2	0.1	$\frac{120}{\nu}$
$\nu = 1$	2.41	6.31	12.71	31.82	63.66	318.3	636.6	
2	1.60	2.92	4.30	6.96	9.92	22.33	31.60	
3	1.42	2.35	3.18	4.54	5.84	10.21	12.92	
4	1.34	2.13	2.78	3.75	4.60	7.17	8.61	
5	1.30	2.02	2.57	3.36	4.03	5.89	6.87	
6	1.27	1.94	2.45	3.14	3.71	5.21	5.96	
7	1.25	1.89	2.36	3.00	3.50	4.79	5.41	
8	1.24	1.86	2.31	2.90	3.36	4.50	5.04	
9	1.23	1.83	2.26	2.82	3.25	4.30	4.78	
10	1.22	1.81	2.23	2.76	3.17	4.14	4.59	12
12	1.21	1.78	2.18	2.68	3.05	3.93	4.32	10
15	1.20	1.75	2.13	2.60	2.95	3.73	4.07	8
20	1.18	1.72	2.09	2.53	2.85	3.55	3.85	6
24	1.18	1.71	2.06	2.49	2.80	3.47	3.75	5
30	1.17	1.70	2.04	2.46	2.75	3.39	3.65	4
40	1.17	1.68	2.02	2.42	2.70	3.31	3.55	3
60	1.16	1.67	2.00	2.39	2.66	3.23	3.46	2
120	1.16	1.66	1.98	2.36	2.62	3.16	3.37	1
∞	1.15	1.64	1.96	2.33	2.58	3.09	3.29	0

The function tabulated is t_P defined by the equation

$$\frac{P}{100} = \frac{1}{\sqrt{(\nu\pi)}} \frac{\Gamma(\frac{1}{2}\nu + \frac{1}{2})}{\Gamma(\frac{1}{2}\nu)} \int_{|t| \geq t_P} \frac{dt}{(1 + t^2/\nu)^{\frac{1}{2}(\nu+1)}}.$$

If t is the ratio of a random variable, normally distributed with zero mean, to an independent estimate of its standard deviation based on ν degrees of freedom, $P/100$ is the probability that $|t| \geq t_P$.

Interpolation ν-wise should be linear in $120/\nu$.

Other percentage points may be found approximately, except when ν and P are both small, by using the fact that the variable

$$y = \pm \sinh^{-1} \{\sqrt{(3t^2/2\nu)}\},$$

where y has the same sign as t, is approximately normally distributed with zero mean and variance $3/(2\nu - 1)$.

APPENDIX C1

Analysis On Binder Content for Prepared Emulsion

Serial Number	Sample Name	Minimum Result (x1)	Test Result (x2)	Nominal Result
1	A1-40	38.0	40.6	40
2	A1-50	48.0	50.5	50
3	A1-55	53.0	55.5	55
4	A1-60	58.0	60.4	60
5	A2-40	38.0	41.2	40
6	A2-50	48.0	50.9	50
7	A2-55	53.0	55.9	55
8	A2-60	58.0	60.8	60
9	A3-40	38.0	41.8	40
10	A3-50	48.0	51.5	50
11	A3-55	53.0	56.4	55
12	A3-60	58.0	61.2	60
Sum	12	591.0	626.7	615.0

Mean of (x1) = $\frac{591}{12}$ = 49.25

Mean of (x2) = $\frac{626.6}{12}$ = 52.23

Mean of (X2) - Mean of (x1) = 52.23 - 49.25

= 2.98

= 3.0

Nominal mean = $\frac{615}{12}$

= 51.25

Mean of X2) - nominal mean

= 52.23 - 51.25

= 0.98 ≈ 10

APPENDIX C2

Analysis On Residue after Sieving Through 710μm Sieve for Prepared Emulsion

Serial Number	Sample Name	Standard Result (x1)	Test Result (x2)	$(x1)^2 x10^{-3}$	$(x2)^2 x10^{-3}$
1	A1-40	0.05	0.07	2.50	4.90
2	A1-50	0.05	0.06	2.50	3.60
3	A1-55	0.05	0.05	2.50	2.50
4	A1-60	0.09	0.06	2.50	3.60
5	A2-40	0.05	0.04	2.50	1.60
6	A2-50	0.05	0.04	2.50	1.60
7	A2-55	0.05	0.04	2.50	1.60
8	A2-60	0.05	0.03	2.50	0.90
9	A3-40	0.05	0.06	2.50	3.60
10	A3-50	0.05	0.04	2.50	1.60
11	A3-55	0.05	0.02	2.50	0.40
12	A3-60	0.05	0.05	2.50	2.50
Sum	12	0.60	0.56	30.00	28.40

Mean of (x1) $= \frac{0.60}{12} = 0.05$

$(N-1)\,D^2 = \Sigma(x1)^2 - \frac{(\Sigma x1)^2}{N}$

$= 0.03 - \frac{(0.60)^2}{12}$

$= 0$

Mean of (x2) $= \frac{0.56}{12} = 0.047$

$(n-1)d^2 = \sum(x2)^2 - \frac{(\Sigma x2)^2}{n}$

$= 0.0284 - \frac{0.56^2}{12}$

$= 0.0022$

Hence $s^2 = \frac{(N-1)B^2 + (n-1)d^2}{N+n-2} = \frac{0 + 0.0022}{22} = 1.03 \times 10^{-4}$

$s = 1.02 \times 10^{-2}$

The estimate of standard error of means difference is

$s\sqrt{\frac{1}{N} + \frac{1}{n}} = 1.02 \times 10^{-2} \sqrt{\frac{1}{12} + \frac{1}{12}} = 4.16 \times 10^{-3}$

$t = \frac{|A - a|}{s\sqrt{\frac{1}{N} + \frac{1}{n}}} = \frac{|0.05 - 0.047|}{4.16 \times 10^{-3}} = \frac{0.003}{4.16 \times 10^{-3}} = 0.72$

$V = N + n - 2 = 12 + 11 - 2 = 22.$

At 95% level of significant, t from table (appendix B) = 2.08

2.08 is greater than 0;72. Therefore the hpotesis that both sample were of the same parent is accepted.

APPENDIX C3

Analysis of Residue after sieving through 150um sieve

Serial Number	Sample Name	Standard Result (x1)	Test Result (x2)	$(x1)^2 \times 10^{-2}$	$(x2)^2 10^{-2}$
1	A1-40	0.15	0.20	2.25	4.00
2	A1-50	0.15	0.24	2.25	5.76
3	A1-55	0.15	0.30	2.25	9.00
4	A1-60	0.15	0.25	2.25	6.25
5	A2-40	0.15	0.28	2.28	7.84
6	A2-50	0.15	0.21	2.25	4.41
7	A2-55	0.15	0.20	2.25	4.00
8	A2-60	0.15	0.10	2.25	1.00
9	A3-40	0.15	0.25	2.25	6.25
10	A3-50	0.15	0.28	2.25	7.84
11	A3-55	0.15	0.30	2.25	9.00
12	A3-60	0.15	0.30	2.25	9.00
Sum	12	1.8	2.91	27.00	74.35

Mean of (x1) $= \frac{1.8}{12} = 0.15$ Mean of (x2) $= \frac{2.91}{12} = 0.243$

$(N-1)D^2 = \Sigma(x1)^2 - \frac{(\Sigma x1)^2}{N}$ $(n-1)d^2 = \Sigma(x2)^2 - \frac{(\Sigma x2)^2}{n}$

$= 0.27 - \frac{1.8^2}{12}$ $= 0.7435 - \frac{2.91^2}{12}$

$= 0$ $= 0.037$

Hence $s^2 = \frac{(N-1)p^2 + (n-1)d^2}{N+n-2} = \frac{0 + 0.037}{22} = 0.00167$

$s = 0.041$

The estimate of standard of means difference is

$s\sqrt{\frac{1}{N} + \frac{1}{n}} = 0.041\sqrt{\frac{1}{12} + \frac{1}{12}} = 0.0167$

$V = N + n - 2 = 12 + 12 - 2 = 22$

At 95% level of significant, t from table (appendix B) = 2.08

t calculated $= \frac{|A - a|}{s\sqrt{\frac{1}{N} + \frac{1}{n}}} = \frac{|0.15 - 0.243|}{0.0167} = 5.56$

2.08 is less than 5.56. Therefore the null hypotesis that both samples were drawn from the same parent population is rejected.

Appendix C4

Analysis of Stability on Mixing with Coarse Aggregates for Prepared Emulsion

Serial Number	Sample Name	Standard Result	Class Mean (x1)	Test Result	$(x1)^2$	$(x2)^2$
1	A1-40	20-80	50	53	2500	2809
2	A1-50	20-80	50	47	2500	2209
3	A1-55	20-80	50	46	2500	2116
4	A1-60	20-80	50	41	2500	1681
5	A2-40	20-80	50	38	2500	1444
6	A2-50	20-80	50	49	2500	2401
7	A2-55	20-80	50	58	2500	3364
8	A2-60	20-80	50	58	2500	3364
9	A3-40	20-80	50	67	2500	4489
10	A3-50	20-80	50	55	2500	3025
11	A3-55	20-80	50	48	2500	2304
12	A3-60	20-80	50	44	2500	1936
Sum	12		600	604	30000	31142

Mean of (x1) = $\frac{600}{12}$ = 50 Mean of (x2) = $\frac{604}{12}$ = 50.33

$(N-1)D^2 = \sum(x1)^2 - \frac{(\sum x1)^2}{N}$ $(n-1)d^2 = \sum(x2)^2 - \frac{(\sum x2)^2}{n}$

$30000 - \frac{600^2}{12}$ $= 31142 - \frac{604^2}{12}$

$= 0$ 740.67

Hence $s^2 = \frac{(N-1)D^2 + (n-1)d^2}{N + n - 2}$ $= \frac{0 + 740.67}{22} = 33.67$

$s = 5.802$

The estimate of standard error of means difference is

$$s\sqrt{\frac{1}{N} + \frac{1}{n}} = 5.802\sqrt{\frac{1}{12} + \frac{1}{12}} = 2.369$$

$$t = \frac{|A - a|}{s\sqrt{\frac{1}{N} + \frac{1}{n}}} = \frac{|50 - 50.33|}{2.369} = 0.139$$

$V = N + n - 2 = 12 + 12 - 2 = 22$

At 95% level of significant, t from table (appendix B) = 2.08

2.08 is greater than 0.139. Therefore the null hypotesis that both samples were drawn from the same parent population is accepted.

APPENDIX C5

Analysis of Viscosity For Prepared Emulsion

Serial Number	Sample Name	Standard Range	Class Mean (xl)	Test Result (x2)	$(xl)^2$	$(x2)^2$
1	A1-40	4	4.0	4.4	16.00	19.36
2	A1-50	5	5.0	4.9	25.00	24.01
3	A1-55	5-8	6.5	7.0	42.25	49.00
4	A1-60	6.9	7.5	9.2	56.25	84.64
5	A2-40	4	4	3.0	16.00	9.00
6	A2-50	5	5	4.5	25.00	20.25
7	A2-55	5-8	6.5	7.2	42.25	51.84
8	A2-60	6.9	7.5	7.7	56.25	59.29
9	A3-40	4	4	6.2	16.00	38.44
10	A3-50	5	5	6.4	25.00	40.96
11	A3-55	5-8	6.5	9.1	42.25	82.81
12	A3-60	6-9	7.5	9.8	56.25	96.04
Sum	12		69	79.4	418.5	575.64

Mean of (xl) = $\frac{69}{12}$ = 5.75, Mean of (x2) = $\frac{79.4}{12}$ = 6.62

$(N-1)D^2 = \Sigma(xl)^2 - \frac{(\Sigma xl)^2}{N}$ $(n-1)d^2 = \Sigma(x2)^2 - \frac{(\Sigma x2)^2}{n}$

$418.5 - \frac{69^2}{12}$ $= 575.64 - \frac{79.4^2}{12}$

$= 21.75$ $= 50.28$

Hence $s^2 = \frac{(N-1)D^2 + (n-1)d^2}{N + n - 2} = \frac{21.75 + 50.28}{22} = 3.27$

$s = 1.81$

The estimate of standard error of means difference is

$s\sqrt{\frac{1}{N} + \frac{1}{n}} = 1.81\sqrt{\frac{1}{12} + \frac{1}{12}} = 0.739$

$t = \frac{|A - a|}{s\sqrt{\frac{1}{N} + \frac{1}{n}}} = \frac{|5.75 - 6.62|}{0.739} = 1.177$

$V = N + n - 2 = 12 + 12 - 2 = 22$

At 95% level of significant t from table (Appendix B) is 2.08
2.08 is greater than 1.177. Therefore the null hypotesis that both samples were drawn from the same parent population is accepted.

APPENDIX C6

Analysis on Storage Stability for short period on Prepared Emulsion

Serial Number	Sample Name	Standard Result (x1)	Test Result (x2)	$(x1)^2$	$(x2)^2$
1	A1-40	60	45	3600	2025
2	A1-50	60	50	3600	2500
3	A1-55	60	50	3600	2500
4	A1-60	60	65	3600	4225
5	A2-40	60	45	3600	2025
6	A2-50	60	55	3600	3025
7	A2-55	60	60	3600	3600
8	A2-60	60	60	3600	3600
9	A3-40	60	50	3600	2500
10	A3-50	60	60	3600	3600
11	A3-55	60	55	3600	3025
12	A3-60	60	75	3600	5625
Sum	12	720	670	43200	38250

Mean of (x1) = $\frac{720}{12}$ = 60 Mean of (x2) = $\frac{670}{12}$ = 55.83

$(N-1)D^2 = \Sigma(x1)^2 - \frac{(\Sigma x1)^2}{N}$ $(n-1)d^2 = \Sigma(x2)^2 - \frac{(\Sigma x2)^2}{n}$

$= 43200 - \frac{720^2}{12}$ $= 38250 - \frac{670^2}{12}$

$= 0$ $= 841.67$

Hence $s^2 = \frac{(N-1)D^2 + (n-1)d^2}{N + n - 2} = \frac{0 + 841.67}{22} = 38.26$ $s = 6.19$

The estimate of the standard error of means difference is

$$s\sqrt{\frac{1}{N} + \frac{1}{n}} = 6.19\sqrt{\frac{1}{12} + \frac{1}{12}} = 2.53$$

$$t = \frac{|A - a|}{s\sqrt{\frac{1}{N} + \frac{1}{n}}} = \frac{|60 - 55.83|}{2.53} = 1.65$$

$$V = N + n - 2 = 12 + 12 - 2 = 22$$

At 95% level of significant t from table = 2.08

2.08 is greater than 1.65. Therefore the null hypotesis that both sample were drawn from the same parent population is accepted.

APPENDIX C7

Analysis on Stability for long period on Prepared Emulsion

Serial Number	Sample Name	Standard Value (x1)	Test Result (x2)	$(x1)^2$	$(x2)^2$
1	A1-50	2.0	1.8	4.00	3.24
2	A1-55	2.0	1.8	4.00	3.24
3	A1-60	2.0	2.4	4.00	5.76
4	A2-50	2.0	1.4	4.00	1.96
5	A2-55	2.0	1.5	4.00	2.25
6	A2-60	2.0	1.9	4.00	3.61
7	A2-50	2.0	2.1	4.00	4.41
8	A2-55	2.0	2.1	4.00	4.41
9	A2-60	2.0	2.8	4.00	7.84
Total	9	18.0	17.8	36.00	36.97

Mean of (x1) = $\frac{18.0}{9}$ = 2.0 Mean of (x2) = $\frac{17.8}{9}$ = 1.98

$(N-1)D^2 = \Sigma(x1)^2 - \frac{(\Sigma x1)^2}{N}$ $(n-1)d^2 = \Sigma(x2)^2 - \frac{(\Sigma x2)^2}{N}$

$= 36 - \frac{18^2}{9}$ $= 36.97 - \frac{17.8^2}{9}$

$= 0$ $= 1.77$

Hence $s^2 = \frac{(N-1)D^2 + (n-1)d^2}{N + n - 2}$ $= \frac{0 + 1.77}{16}$ $= 0.111$

$s = 0.333$

The estimate of standard error of the means difference is

$s\sqrt{\frac{1}{N} + \frac{1}{n}}$ $= 0.333\sqrt{\frac{1}{9} + \frac{1}{9}}$ $= 0.157$

$$t = \frac{|A - a|}{s\sqrt{\frac{1}{N} + \frac{1}{n}}} = \frac{0.02}{0.157} = 0.127$$

At 95% level of significant t from table = 2.59

2.59 is greater than 0.127. Therefore the null lypotesis that both samples were drawn from the same parent population is accepted.

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