A STUDY ON NATURAL RUBBER LATEX MODIFICATION OF BITUMEN EMULSIONS FOR ROAD RUBBERISATION

A PROJECT REPORT SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE

B.TECH DEGREE

IN

POLYMER SCIENCE AND RUBBER TECHNOLOGY

BY

JOSHY MATHEW

DEPARTMENT OF POLYMER SCIENCE AND RUBBER TECHNOLOGY

COCHIN UNIVERSITY OF SCIENCE AND TECHNOLOGY

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Phone : 5-55723

DEPARTMENT OF POLYMER SCIENCE AND RUBBER TECHNOLOGY
COCHIN UNIVERSITY OF SCIENCE AND TECHNOLOGY
COCHIN - 682 022

Date....................

CERTIFICATE

This is to certify that the project work entitled **"A STUDY ON NATURAL RUBBER LATEX MODIFICATION OF BITUMEN EMULSIONS FOR ROAD RUBBERISATION"** submitted by **Mr. JOSHY MATHEW** is a bonafide record of the work done by him at Department of Polymer Science and Rubber Technology, Cochin University of Science and Technology under the guidance of Dr. Rani Joseph during the year 1999 in partial fulfilment for the award of B.Tech Degree in Polymer Science and Rubber Technology of Cochin University of Science and Technology.

DR. A.P.KURIAKOSE
Professor & Head.

e-mail: hodpsrt@md 2 vsnl.net.in

Fax 0484 532495

Phone : 5-55723

DEPARTMENT OF POLYMER SCIENCE AND RUBBER TECHNOLOGY

COCHIN UNIVERSITY OF SCIENCE AND TECHNOLOGY

COCHIN - 682 022

Date....................

CERTIFICATE

This is to certify that Mr. Joshy Mathew, *B.Tech Final Semester student, Dept of Polymer Science and Rubber Technology, has successfully completed a project entitled* "A STUDY ON NATURAL RUBBER LATEX MODIFICATION OF BITUMEN EMULSIONS FOR ROAD RUBBERISATION" *in the Dept. of Polymer Science and Rubber Technology under my supervision during 1st February to 30th April 1999.*

Rani Joseph

Dr. Rani Joseph

Professor

e-mail: hodpsrt@md 2 vsnl.net.in

Fax 0484 532495

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CHAPTER I

INTRODUCTION

1.1 General

Roads are important life lines of an economy. For the development of any country, efficient and adequate systems of transportation are inevitable and of the different means of transportation, road transport is the most significant one. It not only provides independent facility for road travel by a well-planned network of roads but also serves as feeder system for other modes of transportation.

1.2 Different types of pavements

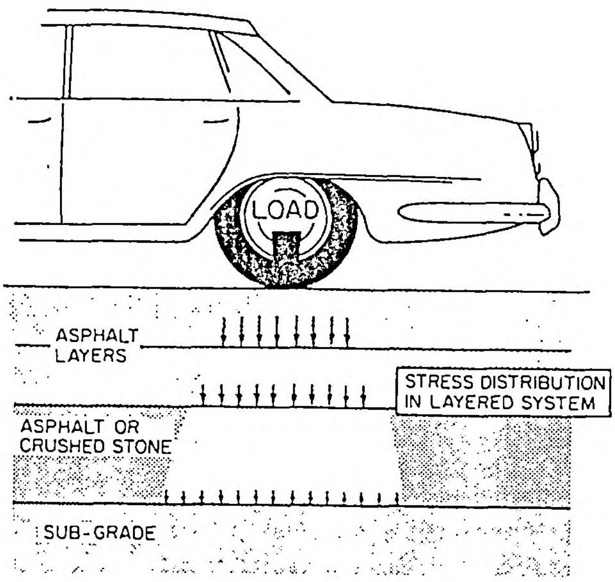
Based on the structural behaviour, pavements can be classified into two:

1.2.1 Rigid Pavements

Rigid pavements are those, which possess note worthy flexural strength or flexural rigidity. This has the slab action and is capable of transmitting the wheel load stresses through a wider area below. It does not get deformed to the shape of the lower surface as it can bridge the minor variations of lower layer. The usual rigid pavement structure is cement concrete slab.

1.2.2. Flexible Pavements

Flexible pavements are those which on the whole have low or negligible flexural strength and are rather flexible in this structural action under the loads. The flexible pavement layers transmit the vertical or compressive stresses to the lower layer by grain to grain transfer through the points of contact in the granular structure. Bituminous concrete is the commonly used material for the flexible pavements.



Load distribution on a flexible road.

Flexible pavements constitute over 95% of total road network and airfields in our country. This is due to the economic reasons and easy availability of bituminous materials.

1.3 Bitumen

Bitumens are highly viscous liquids or solids at normal temperatures and exhibit thermoplastic behaviour-softening when heated, becoming mobile liquids on further heating and returning to their original state on cooling. They are strong adhesives and, because they are essentially hydrocarbons, are excellent waterproofing agents. Bitumens are also non-volatile and highly resistant to oxidation and weathering processes. They are, therefore, very durable. Bitumens are black or black-brown in colour, and almost completely soluble in solvents such as benzene, trichloroethylene and carbon disulfide.

The predominant use for Bitumens is found in road construction. There are, however, many industrial applications of Bitumens ranging from waterproofing materials, corrosion protective to electrical uses.

Bitumens today are manufactured from the atmosphere residue of the distillation of crude oil. This atmosphere residue is then subjected to vacuum distillation for further separation of the distillable products. The vacuum residue so obtained is then air blown at temperature from 220^0 C to 280^0 C to get Bitumen.

1.4 Rubberisation of Bitumen

Basically rubberised bitumen comprises of bituminous compound into which rubber in suitable form is incorporated using appropriate techniques. When rubber powder or latex is added under stir to hot bitumen, it disperses in the form of fine particles. These particles swell and then begin to dissolve under the action of prolonged heating, ultimately to dissolve completely and a tough rubbery material called rubberised bitumen is produced. By the uniform incorporation of rubber, Bitumen under goes radical changes in its properties. The property changes depend, to a larger extent, on the type and grade of rubber used, ratio of bitumen to rubber and the method of incorporation of rubber in Bitumen.

1.5 Bitumen Emulsions

Bitumen emulsions are dispersions of very small drops of Bitumen in an aqueous medium. The droplets usually range in diameter from 1-5 microns. A satisfactory emulsion is smooth in appearance and usually brown in colour. Emulsions are used in road building and maintenance, soil stabilisation etc. Emulsions are easy to handle; for e.g., the viscosity of the Bitumen emulsion can be reduced by adding water.

Bitumen emulsions are of two types:

Anionic Emulsions: - The term anionic arises from the fact that the surfaces of the as Bitumen droplet carry negative charges. They have an alkalinity of PH 9 -11. Sodium or potassium soaps are prepared from the byproducts of rosin refining and the soaps of various complex high molecular weight organic acid and are used extensively as surface active materials for the preparation of anionic emulsion. To make reasonably stable anionic emulsions, it is necessary for some colloidal material to be associated with soap so added to the aqueous dispersion. Gums, casein and other vegetable proteins dispersed in the alkaline aqueous phase are used commercially.

Cationic Emulsions :- In the system, the surfaces of the Bitumen drops carry a positive charge. Cationic emulsions have an unusual ability to adhere to and hold together even difficult type of aggregate. Also, cationic emulsions show rapid initial set of property of great practical importance since it minimises loss resulting from rainfall.

Certain fatty diamine salts and quaternary ammonium salts have been responsible for the most successful groups of cationic bitumen emulsions. The deposition of the bitumen globules on to a surface from a cationic emulsion takes place in startling contrast to the anionic variety. The break of a cationic emulsion is primarily a chemical phenomenon which, because of the natural attraction or substantivity of the cationic agent begins to take place at the moment of contact. Surface and atmospheric conditions have little effect, if any, on adherent properties of the cationically treated bitumen and the presence of moisture on a surface is no deterrent to adhesion. The strong preferential attraction of the cationic agent actually displaces water from the surface, there by creating a common chemical bond between it and the bitumen. The bond is such that an anti-stripping effect is obtained.

Bituminous emulsions are found to have the following advantages

1. These emulsions can be formulated to match any type of aggregate, sand or grit locally available.
2. Tremendous amount of heat energy can save as it can mix with aggregates in ambient temperature.
3. It is environment and user friendly as they cause no pollution and there are no chances of suffering burns or explosion as in hot mix method.
4. It can be used in wet weather as it is water based. Again, it requires only lesser tools, plants and labour force.
5. Added productivity in getting longer working hours is another advantage.

Due to these advantages, nowadays, cold mix paving is preferred over hot mix paving.

1.5.2 Manufacture of Bitumen Emulsions

Commercial preparation of bitumen emulsions involves the use of a rugged type of colloid mill. A heavy load is placed on a colloid mill in dispersing Bitumen and the equipment must be able to with stand the stresses applied. In most type of colloid mills the hot Bitumen is drawn out into thin films between a stator and a high speed rotor. In the presence of aqueous emulsifying solution, the film breaks in to small drops found in the finished emulsions. Bitumen is usually maintained in a molten condition at a temperature of 93.3 to 121.1^0 C. Sometimes the saphonifiable materials are dissolved in the hot Bitumen to expedite the emulsification. The aqueous emulsified solution is usually kept at 82.2 to 87-8^0 C. It is a common practice to dissolve stabilizers or viscosity regulators in the emulsified solutions.

1.5.3 Classes of Emulsions

Three general classifications of emulsion are specified by ASTM namely rapid - (RS), medium – (MS), and slow – setting (SS) emulsions. Rapid setting variety is usually preferred for surface treatment medium setting verity is usually preferred for plant mixes with coarse aggregates. Slow setting type is used for fine aggregate mixes.

1.6 Natural Rubber Latex

Natural rubber latex is a colloidal dispersion of Cis 1, 4 polyisoprene and is obtained from its natural source. Hevea-brasiliensis which is cultivated in tropical regions. The presence of adsorbed proteins and phospholipids gives colloidal stability to the latex.

Freshly tapped natural rubber latex is a whitish fluid of density between 0.975 and 0.980 gm/ ml , PH between 6.5, to 7.0, and surface free energy from 40 to 45 ergs cm^{-2}. Its viscosity is variable.

Being a natural product, the compositions of fresh latex varies between wide limits. The following figures are typical.

Constituents	%
Total solids content	36
Dry rubber content	33
Proteins	1-1.5
Resinous substances	1-2.5
Ash	Upto 1
Sugars	1
Water	55-60

Latex exuding from the tree will coagulate spontaneously a few hours after tapping. The length of time for which the latex will remain fluid depends on many factors especially the ambient temperature conditions of tapping and frequency of tapping. It is also known that external bacteria contaminate the latex. These bacteria act on the non rubber substrates to produce volatile fatty acids such as formic, acetic and propionic acids. This results in coagulation and for preventing these processes, preservation is necessary.

1.6.2 Mechanism of Spontaneous Coagulation

Two general theories have been proposed to account for the phenomenon of spontaneous coagulation. The first supposes that the effect is due primarily to the phenomenon of spontaneous coagulation.. The second ascribes the effect to the liberation of fatty acid anions through the hydrolysis of various lipid substances present in latex. Such anions are then thought to be adsorbed on to the surfaces of the rubber particles, possibly partially displacing adsorbed proteins. They then interact with metallic ions such as those of calcium and magnesium, which are either present in the latex initially or are gradually released by the action of enzymes. The relevant facts accord with the second of the theories. However it is likely that all these processes play an important part in spontaneous coagulation.

1.6.3 Mechanism and Functioning of Preservation

The primary requirement of a preservative is that it should preserve against spontaneous coagulation and putrefaction. The way in which the preservative should be operative are given below :

(i) It should destroy micro-organisms, or at least suppress their activity and growth

(ii) It should contribute positively to the colloidal stability of the latex, in particular by increasing the charge on the particles.

(iii) It deactivate trace of metals, especially traces of heavy metal ions, either by sequestration in solution or by precipitation as in soluble salts. Such deactivation is desirable for two distinct reasons. In the first place, some of these metal ions are essential to the well being of those micro-organisms which cause spontaneous coagulation. Secondly, such ions - notably magnesium - tend to destabilise the latex.

Another requirements :- The ideal preservative will be characterised by a number of ancillary requirements, in addition to effectiveness in its primary function. Thus it should be obviously harmless to both people and rubber. It should not discolor the latex especially for coloured goods manufacturing. It should not impart an offensive odour. It should not interfere with latex processes. It should be cheap and convenient to handle .

1.6.4 Different Preservative Systems

Ammonia is the most popular latex preservative. Usually latex is preserved with 0.7% ammonia. But a variety of other substances also could be used with advantage along with a low level of ammonia concentration (0.2%) for effective latex preservation. For the manufacture of certain latex products, low ammonia latices are preferred. The various preservatives employed are as follows:

1.	Sodium penta chloro phenate	0.2%
	Ammonia	0.2%
2.	Sodium penta chloro phenate	0.1%
	EDTA	.1%
	Ammonia	.1%
3.	Zinc diethyl dithio carbomate	0.2%
	Ammonia	0.2%
4.	Boric acid	0.2 - 0.25%
	Lauric acid	0.03 - 0.06%
	Ammonia	0.2%

Among this any one system cannot keep the PH level near 7. For that purpose formaldehyde can be selected for preserving latex with below 7 PH. Latex is available in the market as preserved field latex, creamed latex and centrifuged latex. Generally all of them are preserved with ammonia.

1.7 Scope and Objective of the Present Work

Since cold wise paving with bituminous emulsions are preferred nowadays over hot-wise, and incorporation of rubber enhances the performance of hot-wise pavements. We thought it worthwhile to study the effect of incorporation of rubber in bituminous emulsions too. The objectives of the present work are :

1. Preparation of a workable latex and Bitumen emulsion mix by adjusting both PH of NR latex and Bitumen emulsions.
2. Development of a suitable preservative to maintain the latex PH below 7.0.
3. Optimisation of the amount of NR latex in the workable mix.
4. Effect of addition of NR latex on the viscosity of the mix and its variation with time elapse.
5. Effect of latex addition on the residue of the Bitumen emulsion.

2.1.3. Field Latex

DRC	....	41.5
TSC	....	43.6
PH	....	6.3

2.1.4. Field Latex Preserved With 20% Formaldehyde Solution

DRC	....	40.1
TSC	....	42.3
PH	....	5.80

2.1.5 Bitumen emulsion R.S type

Characteristics

Appearance	....	brownish viscose liquid
Odour	....	faint fishy odour
Specific gravity	....	1.02
PH	....	6.3
Asphalt content (TSC)	....	60%
Brook field Viscosity	....	154 contipoise at 50 RPM

2.1.6. Bitumen emulsion M.S type

Appearance	....	Brownish viscose liquid
Odour	....	Faint fishy odour
Specific gravity	....	1.023
PH	....	2.3
Asphalt content	....	62%
Brook field viscosity	....	136 at 50 RPM.

2.1.7. Paraformaldehyde

Appearance	....	White powder
Odour	....	Odour of formaldehyde

It is also called polyoxymethylene paraformagene. Also enoneously referred to as Triformol or as thioxy methylene polymerised formaldehyde $(CH_20)_n$ obtained by concentrating formaldehyde solution.

White crystal powder having an odour of formaldehyde slowly soluble in cold, more readily in hot water with evolution of formaldehyde. Insoluble in alcohol, eather etc.

2.1.8. Acetic acid 5% soln.

Prepared by mixing 50ml glacial acetic acid in 950ml. distilled water.

2.2. Testing of latex

2.2.1. Total Solid Content (TSC)

The total solids content is a measure of non volatile non rubber constituents and rubber hydro carbon. For the determination of total solid content, about 2g of the latex sample was accurately weighed into a petrydish. It was made into a thin film at the bottom of the petrydish. The test portion was dried in an air oven at a temperature of 70^0 C for 16 hours, cooled in a decicator and weighed. The drying process was repeated till concordant values were obtained

$$\text{TS \%} = \frac{\text{Mass of dried film}}{\text{Mass of test portion}} \times 100$$

2.2.2. Dry Rubber Content (D.R.C.)

The D.R.C. of the latex is the percentage by weight of the latex which is precipitated by acetic acid under closely defined conditions. About 10g of well mixed sample was accurately weighed in to a beaker. It was then diluted with 20ml of water. Up to 8ml of acetic acid per gram of sample was added with stirring gently for a period of 5min. When the scrum was clear. Small particles of coagulum were collected by rubbing with the main bulk. The coagulum was washed in running water and the thickness reduced to 2mm with a hand roller. The coagulum was dried at 70^0 C for 16 hours, cooled in a decicator and weighed. Drying and weighing were repeated till concordant values were obtained.

$$\text{Dry rubber content, (DRC) \% by weight} = \frac{\text{Weight in gm of dried coagulum} \times 100}{\text{Weight in gram of the sample taken for test}}$$

2.2.3. Alkalinity

Alkalinity is expressed as the amount ammonia content present in the latex. Well mixed and stirred sample was taken in a stoppered conical flask. About 2 to 5 gm of latex was taken from this and diluted to 150^0 CC with water and titrated against standard acid. Methyl red was used as the indicator and the end point was taken as the appearance of a orange red colour.

$$\text{\% of ammonia in latex} = \frac{NV \times 17}{W \times 10}$$

Where N = Normality of acid

V = Volume of acid

W = Weight of latex taken

2.2.4. Volatile fatty acid number (VFA number)

VFA number is the number of gms. of KOH required to neutralise the volatile fatty acids in a latex sample containing 100gm of total solids. Volatile fatty acids are formed by the action of micro-organisms upon some of the serum carbohydrates. It is a measure of the effectiveness of the preservative and the time delay of preservation after elution of the latex from the tree.

50gm of latex was coagulated with 50ml Of 30 percent ammonium sulphate w/v, pressed out the serum and 25 ml. of the serum was acidified with 5ml 50% sulphuric acid. 10ml. of this acidified serum was pipetted out in to the inner tube of a mark ham still. A drop of anti-foaming agent was added. Steam was allowed to pass into the mark ham still and the steam supply was adjusted to collect 3 to 6ml of distillate per minute until 100ml of the distillate had been collected. It was titrated with 0.02 N Ba $(OH)_2$ in the microburette using phenolphthalein as indicator. The end point was the appearance of a faint permanent pink coloration. A blank determination was carried out and this value was subtracted from the sample value to get the volume of alkali.

$$\text{VFA number} = VN\,[\,67.32 + 0.66\,(100\text{-DRC})\,]\,/\,TS$$

Where V = Volume of Ba $(OH)_2$

N = Normality of Ba $(OH)_2$

TS = Total solids content of latex

DRC = Dry rubber content of latex

2.2.5.Determination of Penetration Value of Bitumen and NR Modified bitumen

Penetration is a measurement of hardness or consistency of bituminous material. It is the vertical distance traveled or penetrated by the pointer of a standard needle into the bituminous material under specific condition of load, time and temperature. This distance is measured in one tenth of a millimeter. This test is used for evaluating consistency of bituminous materials.

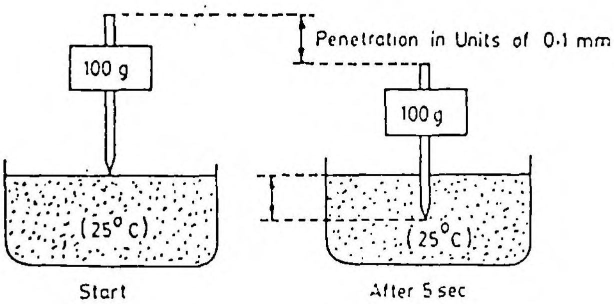
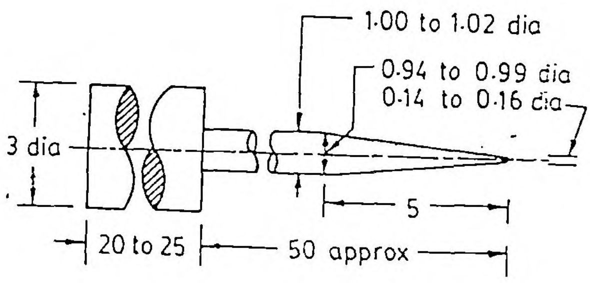


Fig. 6.1 Concept of penetration test



All dimensions in millimetres

2.2.5.1. Apparatus

(i) Container : A flat bottomed cylindrical metallic dish 55mm in diameter and 35 mm in depth is required

(ii) Needle : A straight, highly polished, cylindrical hard steel rod, as per dimensions given in figure

(iii) Penetration apparatus

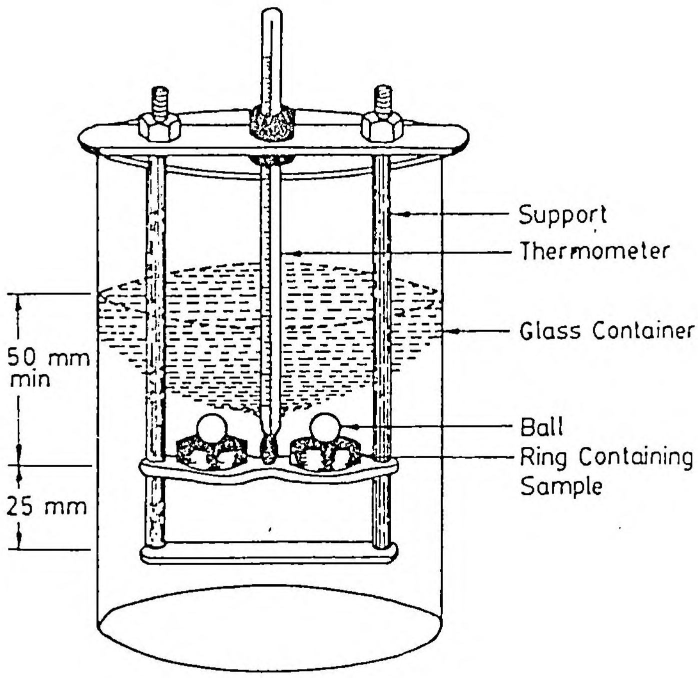
2.2.5.2. Procedure

Softened the material to a pouring consistency at a temperature not more than 90^0C above the approximate softening point and stirred it thoroughly until it was homogeneous and is free from air bubbles and water. Poured the melt into the container to a depth of 10mm in excess of the expected penetration allow it to cool in air for one hour. Then placed along with the dish in a water bath at 25 ^{0}C and allowed it to remain 1 to 1 ½ hours. The test was carried out at 25^0 C. The steps are (i) placed the specimen (ii) cleaned the needle (iii) adjusted the needle top to make just contact with the surface of the specimen (iv) made the dial to read zero (v) released needle for exactly five seconds (vi) read the dial (vi) made three readings in the vertices of a equilateral triangle each away from minimum one cm. from each and from edge of the container.

2.2.6. Determination of Softening Point

The softening point of a bitumen or modified bitumen is the temperature at which the substance attains a penticular degree of softening. As per IS 334-1982, it is the temperature (in ^0C) at which a standard ball passes through a sample of bitumen in a standard mould and falls through a height 2.5 cm. When heated under water or glycerin at specified conditions. It is a measure of the fluidity of the bitumen at high temperature.

2.2.6.1 Apparatus



Ring and ball apparatus

Softening point was determined by ring and ball apparatus it consists of

(a) Steel balls - two numbers each of 9.5mm. diameter and weighing 3.5 to 0.05 gm

(b) Brass rings - two numbers each having depth of 6.4mm. The inside diameter at bottom and top is 15.9mm and 17.5mm respectively.

(c) Ball guides to guide the movement of steel balls centrally.

(d) Support - that can hold rings in position and also allows for suspension of a thermometer. The distance between the bottom of the rings and the top surface of the bottom plate of the support was 25mm.

(e)Thermometer and water bath is also used.

2.2.6.2 Procedure

(i) Preparation of test specimen

Heated the material to a temperature between 110^0 - 140^0 C until, it became completely fluid and free from air bubbles and water. Poured the bitumen into the ring, which was heated approximately the molten material and applied glycerin and starch mixture before it filled, cooled for 30 minutes in air, then placed the ring on assembled apparatus after removing excess material with a warmed sharp knife. Filled the bath with distilled water to a height of 50 cm above the specimen. The temperature of the bath was reduced to 5^0 C. Applied heat to bath and stirred so that the temperature raises at a uniform rate of 5 ± 0.5^0 C per minute. Noted the temperature at which any of the steel ball with bituminous coating touches the bottom plate.

2.2.7. Determination of ductility

The Ductility test gives a measure of adhesive property of bitumen and its ability to stretch. In a flexible pavement design, it is necessary that binders should form a thin ductile film around the aggregates so that the physical inter locking of the aggregates is improved. Ductility of a bituminous material is measured by the distance in centimeters to which it will elongate before breaking when two ends of standard briquette specimen of the material are pulled apart at a specified speed.

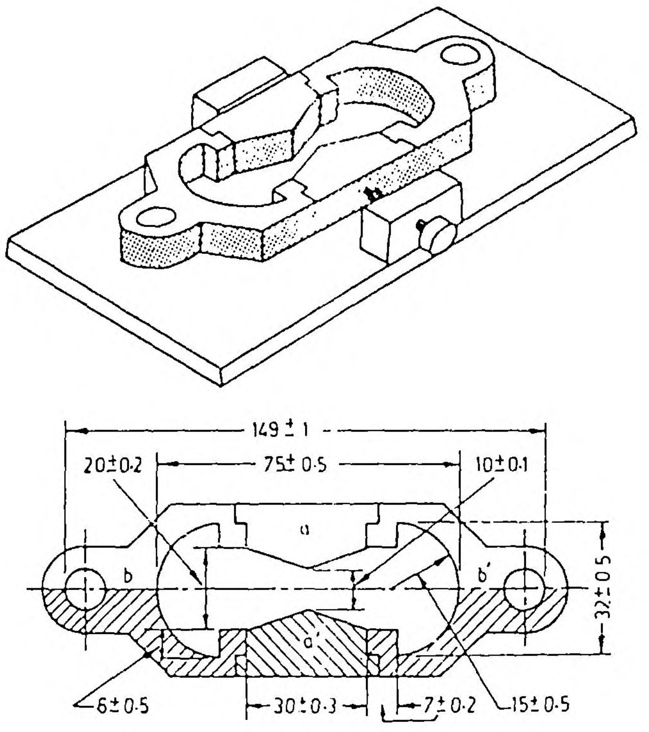
Apparatus

The apparatus for the standard ductility test as per IS: 1208/ 1978 consists of the following

1. Briquette mould: The figure of the mould assembly ad dimensions are given in the figure.
2. Water Bath: It can maintain $\pm$ .1^0 C of specified temperature.

3. Testing machine: For pulling the briquette of bituminous material apart, apparatus used which is so constructed that specimen can be continuously submerged in water while the two clips are being pulled apart horizontally at a uniform speed of 50 $\pm$ 2.5 mm. per minute

4. Thermometer : Range 0-44 ^{0}C and readable up to .2^{0} C



All dimensions in millimetres

Briquette mould

Procedure : Melted the bituminous test material completely at a temperature 130^0C . It became thoroughly fluid, poured the fluid in to the mould assembly which placed on a brass plate after. Before that a mould releasing agent glycerin was applied where ever adhesion was not desire. After that kept the total assembly with molten material in air for 45 minutes. After that kept the total assembly in water bath at 25^0 C for 1½ hours. Removed the sample with mould assembly from the water bath and separate the specimen. Trimmed the specimen with a hot knife. Hooked the clips carefully on the machine with out causing any initial strain. Adjusted the pointer to read zero. Started the machine and pulled at a speed of 50 mm. per minute. Noted the distance at which the bitumen thread of specimen broke. Took it as ductility.

2.2.8.Determination of Viscosity of the Mix By Brook Field Viscometer

2.2.8.1. Instrument

Brook field viscometer is an instrument which gives viscosity in centipoice . It consists of a rotor, a dial, a pointer, motor, dial raising lever, speed selector etc. It has a set of spindles and can be selected any of them depending on the viscosity of the liquid.

2.2.8.2. Procedure

Leveled the viscometer and selected the spindle No.2, took approximate 200ml. bitumen emulsion in a 250ml beaker, inserted the spindle inside the emulsion so that the level of the bottom stud of the spindle be just above the bottom surface of the beaker. Selected a speed and allow to rotate the spindle for one minute. After one minute switched off the spindle and raised the dial to the pointer simultaneously, read the pointer coincide value in the dial and multiplied it with the multiplying factor. Multiplying factor is depended on the spindle and RPM. A table is also available for finding the multiplying factor with the instrument.

2.2.10. Determination of PH

2.2.10.1. Instrument

Digital PH meter: It consists of an electrode assembly digital display etc.

2.2.10.2 Procedure

Prepared buffer solutions of 7 PH , 9PH, and 4 PH in distilled water and calibrated with 7 PH solution by turning the calibrating screw. Then checked with 4 PH and 9PH solutions, washed the electrode with distilled water and removed the end drop of water. Immersed it into the beaker containing the liquid. A 250ml beaker was used with above 100ml solution. Stirred the liquid for sometime till the reading of in digital dial become steady. Taken the reading as PH.

2.2.11. Preparation of Bitumen Emulsion Latex Blend Specimens for Testings

Weighed the bitumen emulsion and latex for the desired proportion and mixed it thoroughly with a glass rod for 5 minutes. Then poured it into a flat vessel then dried it in air oven at 60^0C for 24 hours with intermittent stirring, softened rubberised bitumen over a hot plate at desired temperature (at 125°C) stirred it well continuously to remove all the water present in it. Then poured the bitumen to the specimen container for penetration and moulds for softening point and ductility.

2.3.1. Preparation of Pure Formaldehyde From Para - Formaldehyde

Polyoxymethylparaformagene. Also erroneously referred to as Triformol or as thioxy methylene polymerised formaldehyde $(CH_2O)_n$ obtained by concentrating formaldehyde solution. It's a white crystal powder having an odour of formaldehyde slowly soluble in cold water, more readily in hot water with evolution of formaldehyde in soluble in alcohol, ether, soluble in fixed alkali hydroxide solutions.

Weighed 200gm paraformaldehyde crystal and carefully placed in a 3 neck round bottom flat containing 800ml water. A stirrer was inserted through the centre neck with an air tight assembly. A thermometer was placed through one of the side necks, and an air condenser was placed with glass beads in the another side neck. Total assembly was placed in a heating mantle and heated through the control so as to maintain the system at 80^0C. The condenser helped to reduce the pressure inside vaporising formaldehyde. After 5 hrs. dissolution completed. Now we obtained 20% pure formaldehyde solution.

2.3.2. Blending of Bitumen Emulsion With NR Latex

It is a simple process as bitumen and latex are in emulsion form.For an effective blending bitumen emulsion with latex is possible only in a miscible range of PH. If two of them are in miscible range blending is very easy. Just by stirring and adding we can obtain a homogeneous blend.

CHAPTER III

Results and Discussion

Part I: PH optimisation of both latex and bitumen emulsion (cationic) to obtain a workable mix.

The centrifuged latex having ISI specification was found to have a PH of 11 (Ref 2:1:1). When it was added to the cationic bitumen emulsions of PH 6.3 (RS) and PH 2.3 (MS) in different proportion immediate coagulation took place. Thus the mixes were found to be non workable.

Hence low ammonia, TMTD, ZnO stabilised latex (LATZ) of PH 9.85 (Ref. 2.1.2) was tried in the cationic bitumen emulsions as above. Here also immediate coagulation took place proving the mixes were non workable mix we reduced the PH of the above latex by adding formaldehyde solution (ref. 2.3.1.) at different levels there by preparing latices of different PH levels varying from 9.85 to 7.7. The addition of acetic acid 10% solution in bitumen emulsions changed their PH. We tried latices of different PH and different PH bitumen emulsions. And the observations made are given in the table I.

Table No : 1

Results of latex bitumen emulsion blending

Quantity of latex taken I ml

Quantity of bitumen emulsion taken 9 ml

PH of Latex	PH of Bitumen Emulsion	Observations
11	2.3	immediately coagulated
11	6.3	Do
9.85	2.3	Do
9.85	6.3	Do
8.5	2.3	Do
8.5	6.3	Do
7.7	2.3	Do
7.7	6.3	Do
7.63	2.3	Do
7.65	4.0	Do
7.65	6.3	Do
6.3	6.3	obtain a workable mix
5.85	6.3	Do
5.85	2.3	Do
6.3	2.3	Do

Since centrifuged latex failed to give a workable mix even after reduction in its PH, we extended our study using field latex of PH 6.3 (Ref.2.1.3.) This when blended with cationic bitumen emulsions gave a workable mix. But since fresh field latex cannot be preserved for period over two hours, due to bacterial action and spontaneous coagulation; fresh formaldehyde was prepared (Ref.2.3.1.) and added into the latex as a preservative.

The formaldehyde added latex had a PH of 5.85 and this also gave a workable mix with bitumen emulsion RS and MS.

The formaldehyde added latex was kept for a period of 30 days at room temperature, neither coagulation, nor significant reduction in PH was observed. This shows that the formaldehyde prepared by dissolving paraformaldehyde in water can act as a preservative in latex of low PH value.

Thus we selected the PH of bitumen emulsion 2.3 and latex at 5.85. The further studies were carried out on these materials.

Part II

Study on the viscosity variation of Bitumen emulsion and gelling characteristics of bitumen emulsion on addition of latex at different percentages.

Each measurement was made by taking 250 ml sample and adjusting the spindle at a height of 1.5 cm above from the bottom of the beaker. The tests were carried out at 29^{0}C. Each stage the viscosity was measured 15 minutes after blending. The results obtained are given in the table II.

From the table it can be observed that the viscosity remains constant upto a latex level of 3%. Thereafter a slight increase in the viscosity was seen upto a latex level of 10%. Afterwards a sharp increase in viscosity was seen upto a latex level of 25%. Above 25% of latex level the emulsion broke giving rise to a solid gel.

Observations with the bitumen emulsion, latex, and their blends indicate that the viscosity reduces as the RPM of the viscometer spindle increases. This is an indication of the thixotrophic characteristic of the blends and emulsion.

Table II

Effect of Viscosity on Addition of Latex

Mix No.	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10	A11	A12	A13	A14	A15	A16	A17	A18
% of latex incorporated	0%	100% Pure latex	.5%	1%	1.5%	2 %	25 %	3%	4 %	5 %	6 %	7 %	8%	10 %	15%	20%	25%	30%
RPM	VISCOSITIES																	
50	136	200	136	144	144	144	146	148	152	152	180	188	216	224	544	780	>800	>800
20	180	240	180	180	180	180	180	190	200	200	210	220	270	290	840	1400	1900	>2000
10	240	300	240	240	240	240	240	240	260	260	280	260	340	420	1280	2500	3500	>4000
5	320	400	320	320	320	320	320	320	360	360	360	360	440	520	2048	3600	4800	>8000

Table III gives the viscosity variations with time of a bitumen emulsion containing 10% latex. It was seen that the viscosity initially got reduced with time upto an interval of 10 minutes. Afterwards the viscosity showed a regular increase with time and finally after 45 mts. gelling took place. It was also observed that viscosity decrease as the RPM increases as expected.

Table III

Effect Of Viscosity on Keeping 10% Latex Blended Bitumen Emulsion with Time

TIME / RPM	Immediately after blending	After 5 mts	After 10 mts	After 15 mts	After 30 mts	After 45 mts	After 1 hr
50	272	264	248	304	456	640	> 800
20	340	320	320	360	630	1020	> 2000
10	460	440	420	480	720	1200	> 4000
5	680	640	600	720	960	1600	>8000

Part III

Characteristics of the residue of latex-bitumen emulsion blend.

The specimens are prepared as per the procedure mentioned in 2.2.6.

Table IV

Effect of Penetration

Mix No.	% of Rubber Content	Penetration
B1	0	88
B2	.5	86
B3	1	84
B4	1.5	83
B5	2	80
B6	3	77
B7	5	73
B8	7.5	68
B9	10	61

Table gives the penetration values of different blends of rubber content ranging from 0-10%. The results show a regular decrease in the penetration value with increase in rubber content. This is a clear evident for the hardening effect provided by rubber to bitumen and also the proper dispersion of rubber in the bituminous emulsion.

Table V

Effect on Softening Point

Mix No.	% rubber incorporated	Softening Point ^{0}C
B1	0	41.5
B2	.5	42
B3	1	43
B4	1.5	43.5
B5	2	44.5
B6	3	45
B7	5	47
B8	7.5	51
B9	10	55

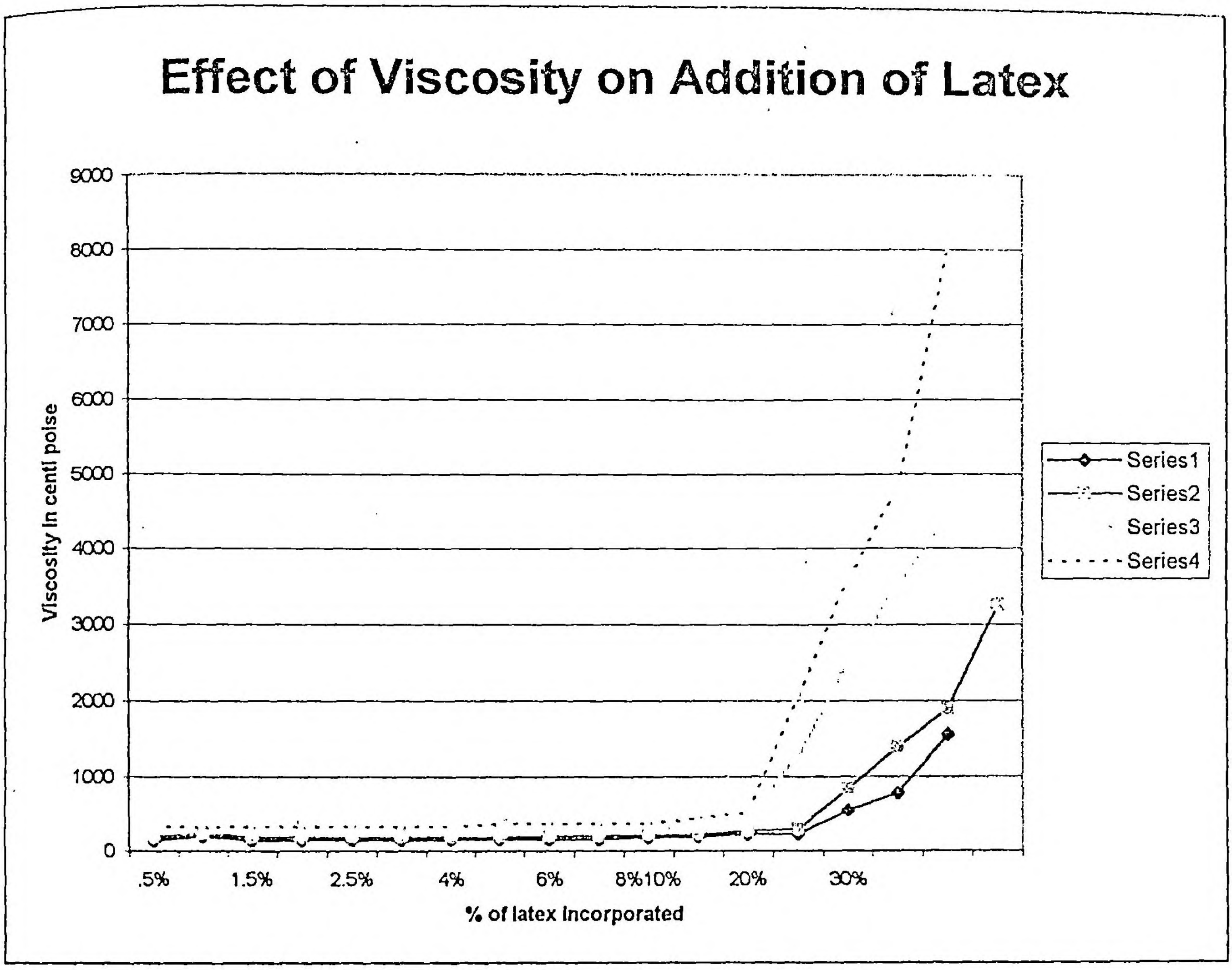
Table V gives the softening point values of the residue of the bituminous emulsion latex blends having rubber contents from 0-10%. The softening point values show a regular increase with increased substitution of rubber. This indicate that rubber blended bitumen emulsion can perform well even at elevated road temperature conditions. These results also supplement the hardening effect provided by rubber to bitumen and also the proper dissolution of rubber in the bitumen matrix.

Table VI

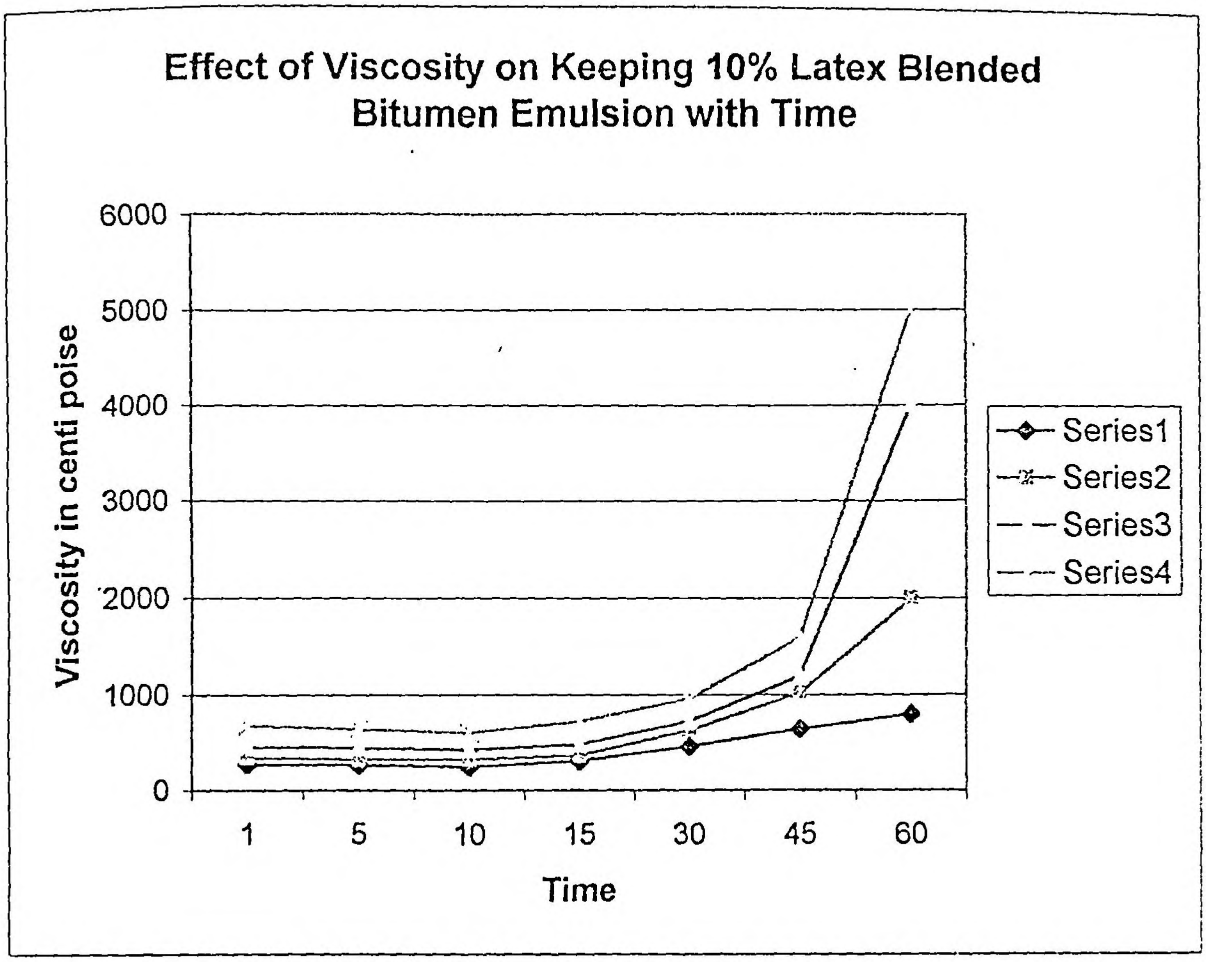
Effect on Ductility

Mix No.	% rubber incorporated	Ductility
B1	0	120
B2	.5	122
B3	1	118
B4	1.5	118
B5	2	110
B6	3	107
B7	5	100
B8	7.5	80
B9	10	39

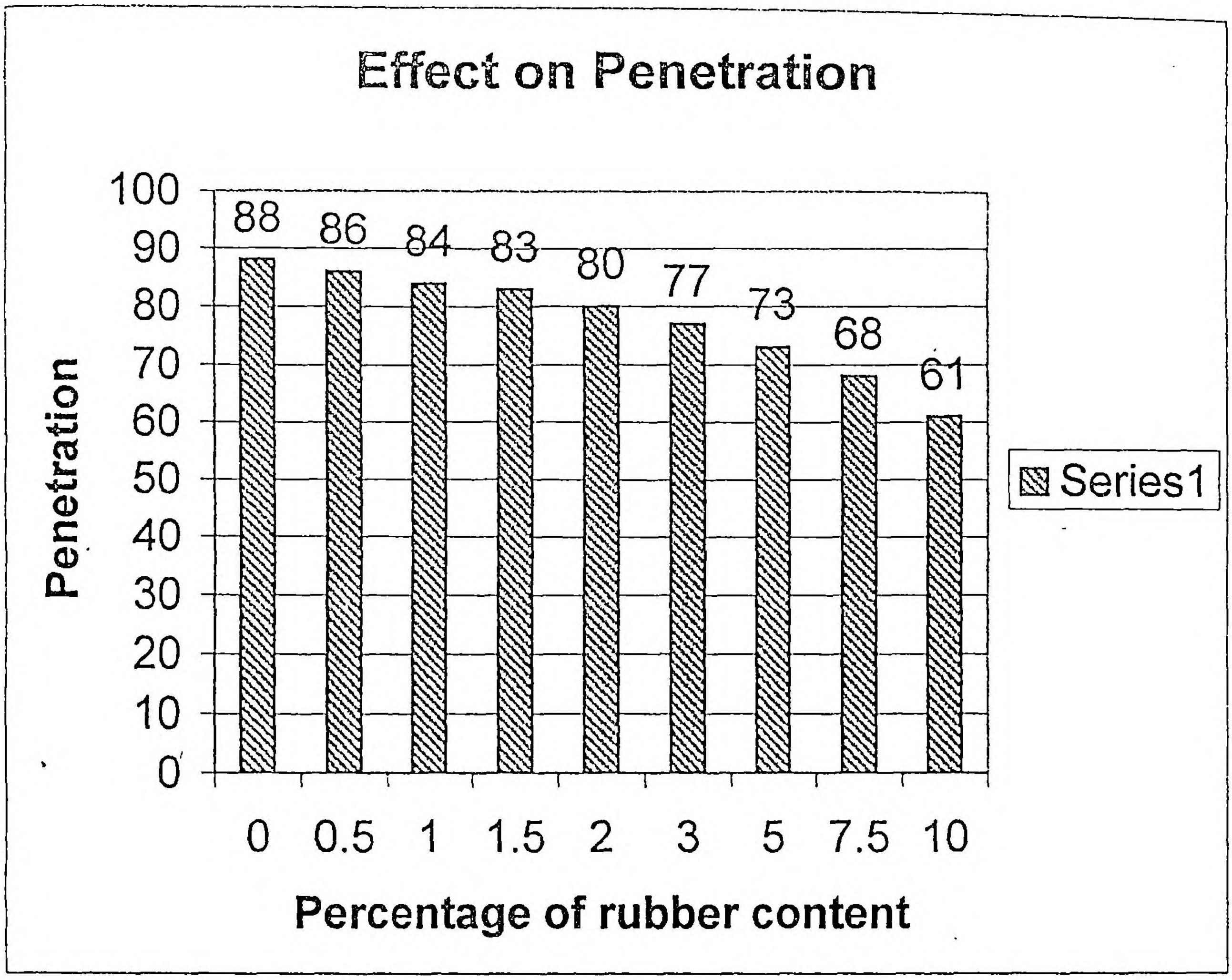
Table VI gives the effect of rubber on the ductility values. The results show that the ductility decreases with increased substitution of rubber in the bitumen emulsion. It is due to the stiffening effect of rubber on the bitumen matrix. Even though ductility values are showing a decreasing in trend, it has no much impact on the workability since here bitumen is used in the emulsified form than as a hot mix.



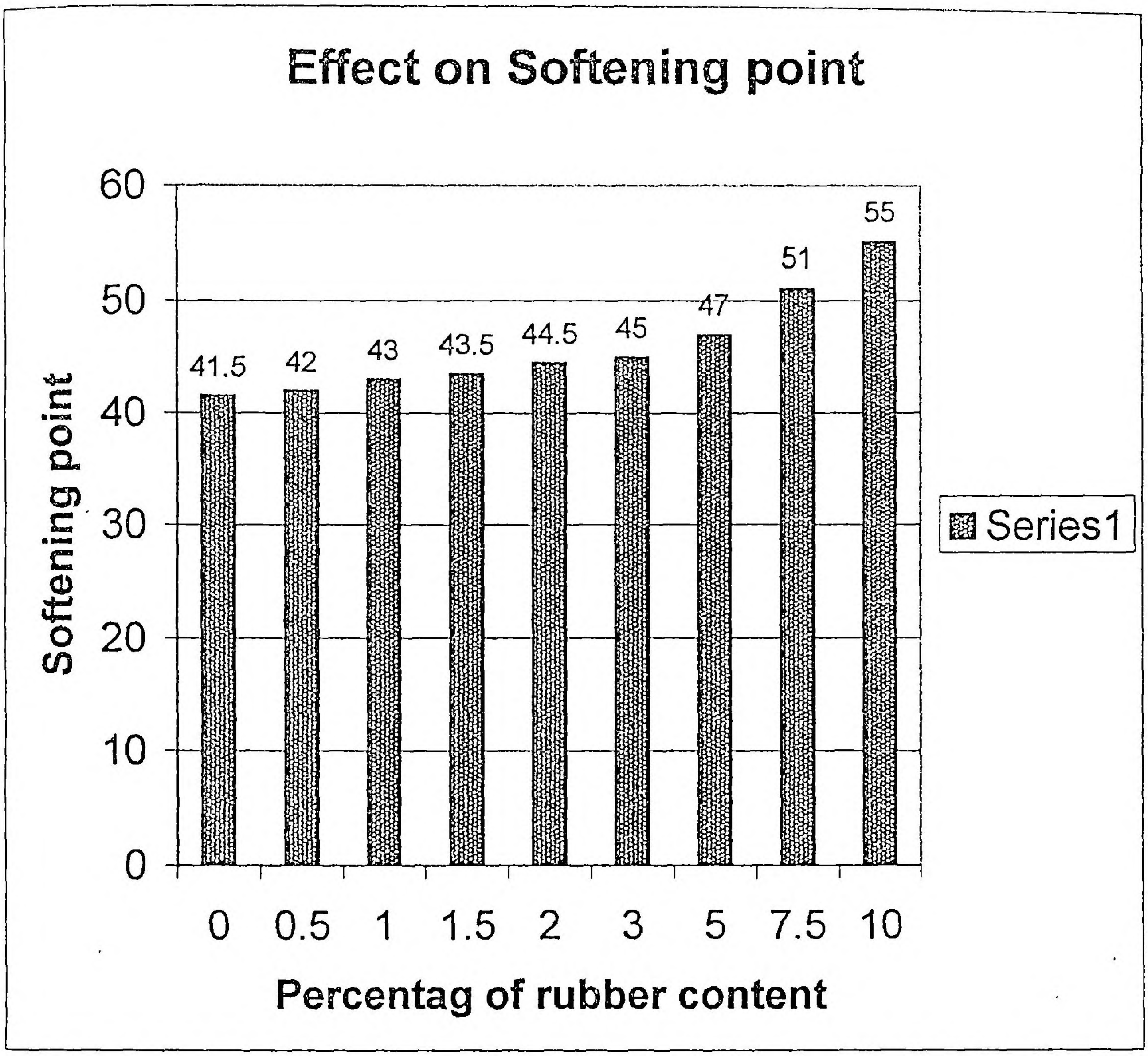
Series 1 at 50 RPM
Series 2 at 20 RPM
Series 3 at 10 RPM
Series 3 at 5 RPM



Series 1 at 50 RPM
Series 2 at 20 RPM
Series 3 at 10 RPM
Series 3 at 5 RPM



Effect on Penetration
Penetration
100
90
80
70
60
50
40
30
20
10
0
88
86
84
83
80
77
73
68
61
Series1
0
0.5
1
1.5
2
3
5
7.5
10
Percentage of rubber content



Effect on Softening point
Softening point
Percentag of rubber content
0
10
20
30
40
50
60
0
0.5
1
1.5
2
3
5
7.5
10
41.5
42
43
43.5
44.5
45
47
51
55
Series1

CHAPTER IV

Summary and Conclusion

* Rubberised bitumen production in the bitumen emulsion latex blend form is very easy and can be done at room temperature in a very simple manner. The incorporation of rubber in the bituminous emulsions will increase the road life as indicated by the increased softening point value, and the decreased penetration values which is a clear evidence for the hardening effect provided by rubber and proper blending of rubber with the bitumen.

* Another point of advantage is that more % of rubber can be incorporated in the bitumen emulsions compared to rubberisation of dry bitumen.

* It is possible to obtain controlled gelling of bitumen emulsion latex blend by adjusting the PH and quantity of latex and bitumen emulsion so it can be formulated to a desired gelling time and for matching any type of aggregate, sand or grit, locally available.

* In rubberisation of dry bitumen it is a usual practice to add kerosene to bring down the viscosity of the hot mix to a workable level which gets evaporated during the paving process causing atmospheric pollution. This emulsion type cold mix process of rubberisation also helps to avoid these environmental problems and wastage of the much-needed precious petroleum products.

* Being a cold process it can save much wealthy forest assets during the rubberisation and paving process. Environmental and user friendly as this causes no pollution and there are no chances of suffering burns or explosion as in hot mix method. It also gives longer working hours and can be used in wet weather as it in water based. It required only lesser tool, plant and labour force.

Further Challenges

* Development of a more suitable preservative system for latex to keep the latex PH level less than 7.
* In corporation of cross-linking agents in these latex modified emulsions can be tried to get enhanced and improved properties.

References

1. Asphalt by Palzakar, Chemical Publishing Company, ink. Newyork .
2. Asphalt science and Technology edited by Arthur M. Usmani Maxel Dekker, Inc New york – Basel – Hong kong.
3. Hand book of Natural Rubber Production in India, published by RRII Kottayam.
4. Indian Standards IS: 5430 (1981)
5. RRIM training Manual on analytical chemistry. Latex and analysis.
6. 'Rubberised Roads for superior performance' by Mr.K.S. Gopala Krishnan, Rubber Asia, July August 1998
7. 'An insight into rubberised roads' by Mr. K.S. Gopala Krishnan.
8. Rubber and its cultivation published by Rubber Board.
9. Laboratory manual in High Way Engineering by Ajay .K. Duggal and Vijay.P. Puri - Wily Eastern Limited.
10. Indian Standards IS : 8887 (1995)
11. " IS :1203(1978)
12. " IS : 1208 (1978)
13. Quick setting and special grade emulsions - a brochure of Tiki Tar Industries Bombay.