TITLE: A New Mix Composition to Increase the Storage Life of Indonesian Bitumen Emulsion

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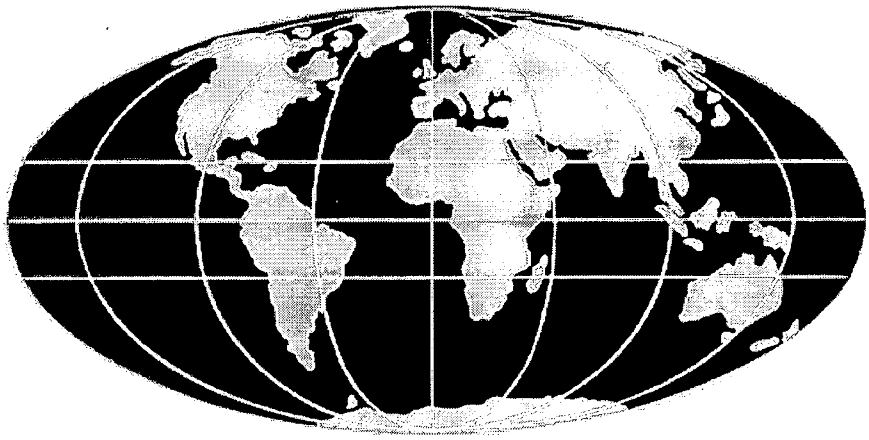

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NEW MIX COMPOSITION TO INCREASE THE STORAGE LIFE OF INDONESIAN BITUMEN EMULSION

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1 INTRODUCTION

Bitumen emulsion is often used for pavement mixes in Indonesia, especially in locations where there is a shortage of hot-mix plants. Current formulations of emulsions have a storage life of about three months. In Indonesia the typical site storage time is frequently more than three months, nine months being quite common. This situation arises because supplies to outlying areas tend to be made only at quite long intervals. The fact that emulsions are stored for longer than three months leads to poor quality asphalt or total loss of the emulsion.

IRE research in 1995 (Tjitjik, 1995) about the ways of handling bitumen emulsion showed that bitumen emulsions made in Indonesia, especially CSS type, had storage lives of only two months when retained in a drum without turning. When the drums were turned once a month, the storage life increased to, typically, five to six months.

This paper describes an IRE research project designed to find formulations for bitumen emulsion that will significantly increase storage life.

2 LITERATURE

2.1 Bitumen Emulsion

Bitumen emulsion is a mixture of two phases, that is an aqueous emulsifier solution as continuous phase and tiny droplets of bitumen as the discontinuous phase. The bitumen particles are typically in the size of 0.1 to 5 micron and are usually stabilized by an ionic emulsifier

2.2 Emulsifier

Based on the charge, there are three types of emulsifier. Those are cationic, anionic, and non-Ionic emulsifiers. Indonesia currently uses cationic emulsifiers.

2.3 The Ways of Increasing Storage Stability of Bitumen Emulsion

According to Stoke's law, the rate of settlement is determined by the following formula:

$$v = 2/9 \times gr^2 \frac{(d1-d2)}{\mu}$$

where,
v = the rate of settlement
g = gravity
r = radius of bitumen particles
d1 = specific gravity of bitumen
d2 = specific gravity of liquid phase
μ = viscosity of liquid phase

Considering the formula, we can see the rate of settlement depends upon:

- Viscosity of the continuous phase,
- Difference in specific gravity between the two phases, and
- The size of bitumen particles.

It follows that there are changes in the formulation of bitumen emulsions that may be expected to improve storage stability. They are as follows:

a) The higher the specific gravity of bitumen the faster the bitumen phase will settle. Adding kerosine to the bitumen, before emulsifying, will decrease the specific gravity of bitumen phase which should reduce the rate of settlement. Likewise, increasing the specific gravity of the aqueous phase should also reduce the rate of settlement.
b) Storage stability of bitumen emulsion should be increased by increasing the viscosity of the aqueous continuous phase.
c) Reduction of particle sizes of the bitumen phase should increase storage stability.

Additional factors that should/may alter storage stability are:

a) Type and quantity of emulsifying agent.
b) Bitumen content and/or viscosity of the emulsion.

3 EXPERIMENT IN LABORATORY

The research investigated both cationic medium setting (CMS) and cationic slow setting (CSS) emulsions to find the optimum compositions by varying:

- Colloid Mill condition i.e. particle size distribution of bitumen droplets,
- emulsifier type,
- kerosene content of bitumen,
- bitumen content of the emulsion,
- emulsifier content of the aqueous phase, and
- $CaCl_2$ content of the aqueous phase.

3.1 Colloid Mill Condition

In this investigation, bitumen emulsions in many compositions were produced using a laboratory colloid mill. The gap size and the rotor speed of the laboratory colloid mill cannot be changed. Therefore, the laboratory colloid mill cannot be adjusted to achieve the same particle size distribution as the factory colloid mill. Storage stability of bitumen emulsions produced by the laboratory colloid mill are lower than those produced by the factory colloid mill. Therefore, the first step of this investigation was calibration to determine the correlation between the storage stabilities of emulsions from the two sources.

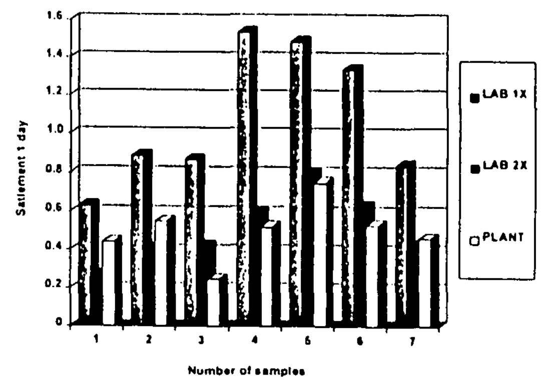
Seven bitumen emulsions were produced by each of the following processes:
- the factory colloid mill,
- the laboratory colloid mill using a single pass and
- the laboratory colloid mill with double passes.

The bitumen emulsions were compared in settlement value and the results are shown in Figure 1 below.

Settlement value is the difference in residual bitumen content, percent, between samples taken from the top and bottom of a 500 ml cylinder after storing for one or five days.
Figure 1 shows that bitumen emulsion produced by a single pass through the laboratory colloid mill had higher settlement values than those produced by the factory. Those using a double pass through the laboratory colloid mill had very similar settlement values to emulsions produced by the factory.
Hence double passes through the laboratory mill were used for the rest of the experiments.

3.2 Emulsifier Type

Figure 1. Effect of Emulsifier Type on Settlement



There are only five emulsifiers currently available in Indonesia. Three of these are for CMS, namely Asfier-103, Asfier-123, Asfier-208. Two are for CSS, namely Polyram-SL and Redicote. The research examined the changes in settlement rate caused by use of each of the above. The results are shown in Figure 1 above.

As shown in Figure 1, the emulsifiers that produced bitumen emulsions with the lowest settlements were Asfier-103 for CMS and Redicote for CSS. These emulsifiers were used for the remainder of the experiment s.

3.3 Variation of Penetration Grade Bitumen

The penetration value of the bitumen feedstock influences the storage stability of the emulsion, possibly via changes in the bitumen specific gravity (SG) and/or through the effect of bitumen hardness on particle size distribution. The influence of bitumen penetration on the settlement of emulsion is shown on Figure 2.

Figure 2. The Influence of Penetration on Settlement of Emulsion

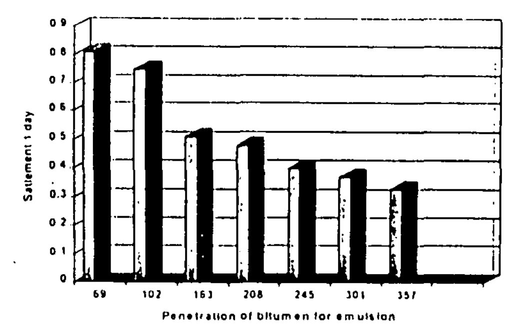
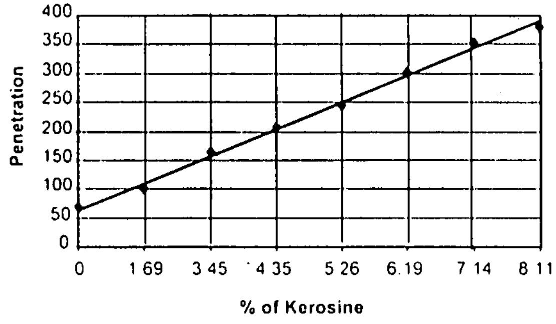


Figure 3. The Effect of Adding Kerosine on Penetration



Bitumens produced in Indonesia by Pertamina are AC pen 60/70 and AC pen 80/100. The literature indicates that pen 180/200 is a more common bitumen grade for use in production of emulsion. In order to produce a bitumen of 180/200 grade, 60/70 was blended with kerosene. The effect on the penetration grade, of adding kerosene to 60/70 bitumen is shown in Figure 3.

As may be seen in Figure 3, above, 4.3% kerosene must be added to the bitumen to increase the penetration value to 200. This also has the effect of reducing the bitumen SG.

3.4 Variation of Bitumen Content

Increasing the bitumen content of a bitumen emulsion increases both emulsion viscosity and storage stability. The influence of bitumen content on storage stability and viscosity of bitumen emulsion is shown in Figures 4 and 5.

Figure 4. The Influence of Bitumen Content on Settlemet of Emulsion

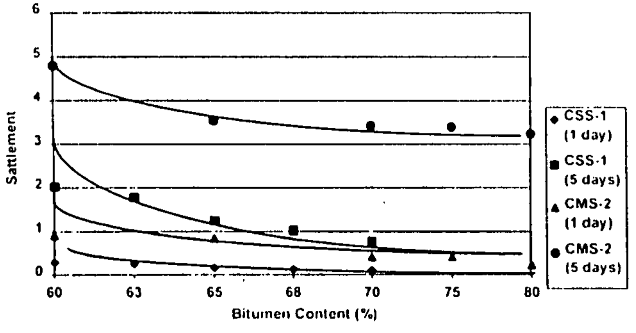


Figure 5. The Influence of Bitumen Content on Settlement of Emulsion

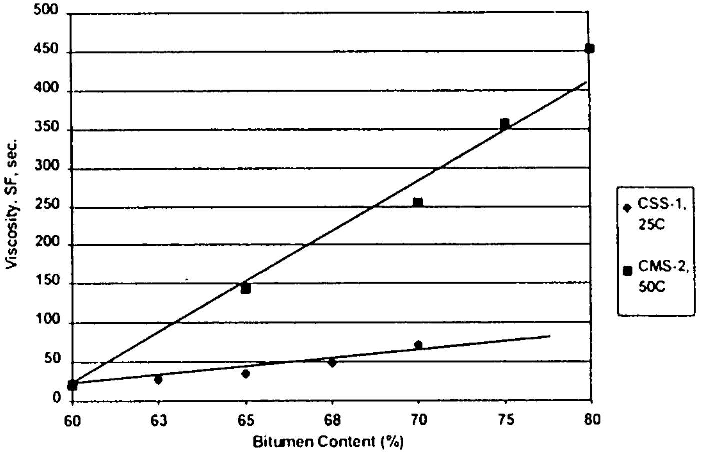


Figure 5, above, shows that a higher bitumen content leads to increased storage stability of bitumen emulsion. Bitumen contents used in current production are designed to achieve emulsion viscosities complying with the relevant specifications. The specified viscosity range for CSS-1, at a temperature of 25°C, is 20 to 100 seconds. For CMS-2 it is 50 to 450 seconds at a temperature of 50°C. Considering Figure 6, the ideal viscosity of CSS-1 is 60 seconds which can be reached at bitumen content of 70 to 72 %. The minimal residue content of CMS-2 is 65 %, so bitumen containing about 10 % of kerosene would leave 65% residue if 72 % of cutback bitumen emulsion were used in the emulsion.

3.5 Variation of Emulsifier Content

The amount of emulsifier used in bitumen emulsion depends on the bitumen content of the emulsion. There is an optimal content of emulsifier to achieve the highest value of storage stability. If the content of emulsifier is too low, the bitumen droplets will soon precipitate. If the content of emulsifier is too high it can cause the emulsion to bubble and excessive air content can destroy the emulsion. The influence of emulsifier content to storage stability of bitumen emulsion is shown in Figures 6 and 7.

Figure 6 shows that the optimum content of emulsifier Asfer-103 is 0.4 % for CMS-2 bitumen emulsion. Figure 7 shows that the optimal content of Redicote emulsifier is 0.48 % for CSS-1 bitumen emulsion. At those emulsifier contents, the emulsion had the lowest value of settlement.

Figure 6. The Influence of Emulsifier Content on Settlement of Emulsion CMS-2 Type

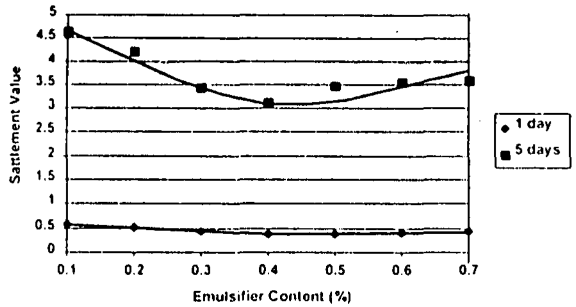
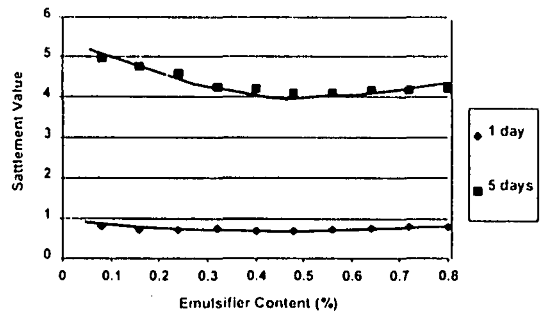


Figure 7. The Influence of Emulsifier Content on Settlement of Emulsion CSS-1 Type

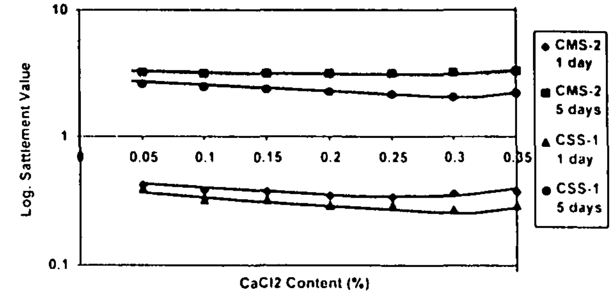


3.6 Variation of $CaCl_2$

Sometimes cationic emulsifiers combine with unexpected anions and are precipitated as sediment. Calcium Chloride, in cationic bitumen emulsion, acts as a stabilizer that inhibits such sedimentation. $CaCl_2$ also increases the density of emulsifier solution and hence increases the storage stability of the emulsion. The influence of $CaCl_2$ on storage stability of bitumen emulsion is shown in Figure 8.

Figure 8, below, indicates that the optimal content of $CaCl_2$ is 0.3 %. This condition gives the best storage stability of CMS-2 bitumen emulsion. The optimal content of $CaCl_2$ could be different if the quality of water in the emulsion changed.

Figure 8. The Influence of $CaCl_2$ Content to Sattlement of Emulsion



3.7 Optimal Composition of Bitumen Emulsion

Using the results of the experiments described above, the optimal composition of bitumen emulsion was determined and is presented in Tables 1 and 2.

Table 1. Composition of Bitumen Emulsion CMS-2 Type

Element of emulsion CMS-2 Type	Composition	
	Modified	Conventional
Bitumen, %	65.00	63.00
Kerosine, %	7.00	7.00
Emulsifier, %	0.40	0.30
Hcl, %	0.40	0.30
$CaCl_2$, %	0.30	0.10
Water, %	27.90	29.30

Table 2. Composition of Bitumen Emulsion CSS-1 Type

Element of emulsion CMS-2 Type	Composition	
	Modified	Conventional
Bitumen, %	65.20	59 00
Kerosine, %	2.80	1.00
Emulsifier, %	0.48	0.40
Hcl, %	0.48	0.40
$CaCl_2$, %	0 30	0 10
Water, %	30 74	39.10

3.8 The Monitoring Quality Result of Modified Bitumen Emulsion

Monitoring the quality of modified bitumen emulsion CMS-2 and CSS-1 shows that it can be placed in 9 months and will still comply with the specification.

Table 3. Quality of Bitumen Emulsion CMS-2 Type

Characteristic	Specification		CMS-2 Modified				CMS-2 Conventional			
	Min	Max	0 M	3 M	6 M	9 M	0 M	3 M	6 M	9 M
Viscosity, SF at 50°C, s	50	450	257,6	257,3	257,8	257,7	252,6	252,3	253,5	252,8
Settlement test, 1 day, %	-	1	0,36	0,47	0,53	0,78	0,42	0,45	0,63	0,88
Settlement test, 5 days, %	-	5	3,18	3,55	3,87	4,22	3,45	3,47	4,12	4,75
Coating ability and water resistance										
- Coating dry aggregate	good		good	good	good	good	good	good	good	good
- Coating after spraying	good		good	good	good	good	good	good	good	good
- Coating wet aggregate	good		good	good	good	good	good	good	good	good
- Coating after spraying	good		good	good	good	good	good	good	good	good
Particle charge test	Positive		+	+	+	+	+	+	+	+
Sieve test, %	-	0,1	0	0	0	0	0	0	0	0
Water content, %	-	-	28,20	27,21	27,45	27,33	26,48	27,27	26,51	26,13
Oil distillate, %	-	12	6,1	6,5	6,3	6,2	6,2	6,3	6,3	6,4
Residue, %	65	-	66,8	66,8	66,2	66,5	65,5	65,3	65,6	65,4
Test on residue										
- Penetration, 25°C, 100 g, 0,1 mm	100	250	214	215	215	215	215	214	214	215
- Ductility, 25°C, cm	40	-	>140	>140	>140	>140	>140	>140	>140	>140
- Solubility in Trichloroethylene, %	97,5	-	99	99	99	99	99	99	99	99
Specific Gravity	-	-	0,958	0,958	0,958	0,958	0,958	0,958	0,958	0,958

Note: M = Months (Age of bitumen emulsion)

Table 3, above, shows that the quality of modified CMS-2 bitumen still meets the specification after 9 months. Table 4, below, shows that the quality of modified CSS-1 bitumen emulsion still meets the specification after 9 months. Whereas the quality of conventional CSS-1 will not meet the specification in 3 months, and it will precipitate in 6 months.

Table 4. Quality of Bitumen Emulsion CSS-1 Type

Characteristic	Spesification		CSS-1 Modification				CSS-1 Conventional			
	Min	Max	0 M	3 M	6 M	9 M	0 M	3 M	6 M	9 M
Viscosity, SF at 25°C, s	20	100	48,6	48,6	48,5	48,6	20,2	19,5	Bitumen Emulsion has Settled	
Sattlement test, 1 day, %	-	1	0,13	0,16	0,17	0,85	0,75	2,60		
Sattlement test, 5 days, %	-	5	1,03	1,23	1,82	4,23	4,30	5,23		
Particle charge test	Positif		+	+	+	+	+	+		
Sieve test, %	-	0,1	0	0	0	0	0	0		
Cement mixing test, %	-	2	1,30	1,32	1,33	1,76	1,80	1,83		
Water content, %	-	-	31,62	31,82	31,62	31,62	39,22	39,25		
Oil destilate, %	-	-	3,31	3,05	3,45	3,34	0,50	0,71		
Residu, %	57	-	65,44	67,74	65,46	64,62	59,81	59,75		
Test on residu										
- Penetration, 25°C, 100 g, 0,1 mm	100	250	135	135	134	135	122	123		
- Ductility, 25°C, cm	40	-	>140	>140	>140	>140	>140	>140		
- Solubility in Trichloroethylene, %	97,5	-	99	99	99	99	99	99		
Specific Gravity	-	-	0,962	0,962	0,962	0,962	0,960	0,960		

Note: M = Months (Age of bitumen emulsion)

4. USING MODIFIED EMULSION IN FIELD EXPERIMENT

A field experiment was performed in which Slurry Seal was mixed and laid to compare the use of modified and conventional CSS-1 emulsions as a binder. The experiments used emulsions, both modified and new, that had been stored for 3, 7 and 9 months. A control section was also laid using new, conventional CSS-1 emulsion.

4.1 Specification of Slurry Seal Mixture

Here are four specification of four types of Slurry Seal mixture.

Table 5. Specification of Four Types of Slurry Seal Mixture

Characteristic of Slurry Seal Mixture	Specification			
	Type I	Type II	Type III	Type IV
Gradation of aggregate, % passing:				
Sieve size : 1/2 "	-	-	-	100
3/8 "	-	100	100	85 - 100
No. 4	100	90 - 100	70 - 90	60 - 85
No. 8	90 - 100	65 - 90	45 - 70	40 - 60
No. 16	65 - 90	45 - 70	30 - 50	28 - 45
No. 30	40 - 60	30 - 50	18 - 34	19 - 34
No. 50	25 - 42	18 - 30	10 - 25	12 - 25
No. 100	15 - 30	10 - 21	7 - 18	7 - 18
No 200	10 - 20	5 - 15	5 - 15	4 - 8
Residual bitumen content, % weight of dry aggregate	10,0 - 16,0	7,5 - 13,5	6,1 - 11,0	5,5 - 8,0
Application rate, kg/m² based on mass of dry aggregate	3,5 - 5,0	5,5 - 8,0	8,0 - 18,0	10,0 - 15,0
Condition of Slurry Seal Mixture	- Semi fluid - Smooth - Homogenous - No emulsion run off	- Semi fluid - Smooth - Homogenous - No emulsion run off	- Semi fluid - Smooth - Homogenous - No emulsion run off	- Semi fluid - Smooth - Homogenous - No emulsion run off

4.2 The Quality of Material for Field Experiment of Slurry Seal

The quality of bitumen emulsion and aggregate used in this field experiment is shown in Tables 6 and 7.

Table 6. The Quality of Bitumen Emulsion

No.	Characteristic of Bitumen Emulsion	Specification		Quality of Bitumen Emulsion CSS-1				Units
				Modified			Conven-tional	
		Min	Max	3 M	7 M	9 M		
1	Viscosity, SF at 25°C	20	100	68,5	69,2	67,3	20,5	seconds
2.	Settlement test, 1 day	-	1	0,15	0,34	0,68	0,65	%
3.	Settlement test, 5 days	-	5	1,25	2,31	4,07	3,77	%
4.	Particle charge test	Positive		+	+	+	+	-
5.	Sieve test, %	-	0,1	0	0	0	0	%
6	Cement mixing test, %	-	2	1,50	1,70	1,80	1,50	%
7	Water content, %	-	-	29,7	27,6	28,3	38,4	%
8	Oil distillate, %	-	-	3,25	3,32	3,12	0,50	%
9.	Residue, %	57	-	64,2	64,8	63,7	58,6	%

Table 6. The Quality of Bitumen Emulsion (continued)

No.	Characteristic of Bitumen Emulsion	Specification		Quality of Bitumen Emulsion CSS-1				Units
				Modified			Conventional	
		Min	Max	3 M	7 M	9 M		
10.	Test on residue:							
	- Penetration, 25°C, 100 g, 0,1 mm	100	250	134	134	135	123	0,1 mm
	- Ductility, 25°C, cm	40	-	>140	>140	>140	>140	cm
	- Solubility in Trichloroethylene, %	97,5	-	99	99	99	99	%
11.	Specific Gravity	-	-	0,9585	0,9588	0,9587	0,9607	-

Table 7. The Quality of Fine Aggregate ex Cilacap

No.	Characteristic of Aggregate	Specification Min	Specification Max	Result of Test	Units
1	Gradation of aggregate, % passing				
	Sieve size: 3/8 "	-	-	100	%
	No. 4	-	-	97,5	%
	No 8	-	-	82,9	%
	No. 16	-	-	58,6	%
	No 30	-	-	39,5	%
	No 50	-	-	29,7	%
	No 100	-	-	16,3	%
	No 200	-	-	7,3	%
2	Specific Gravity.				
	- Bulk	2,500	-	2,630	
	- SSD	2,500	-	2,690	
	- Apparent	2,500	-	2,790	
3	Absorbtion	-	3,00	1,20	%
4	Density	1,200	-	1,530	kg/l
5	Sand Equivalent	50	-	72,53	%
6	Clay Lumps	-	0,25	0,15	%
7.	Organik impurities content	-	3	< 3	%

4.3 Design of Slurry Seal for Field Experiment

The gradation of aggregate, ex Cilacap, is shown in Table 7 above, and meets the specification of Slurry Seal Type II.

4.4 Bitumen emulsion CSS-1 Content

The content of bitumen emulsion CSS-1 type is stated base on the following formula:

P = 0.05A+0.1B+0.5C
where:
P = Percentage of bitumen emulsion
A = Percentage of aggregate that contain in No.8 sieve (17.1 %)
B = Percentage of aggregate passing No.8 sieve and retained on No.200 (75.6 %)
C = Percentage of aggregate that pass in No.200 sieve (7.3 %)

For modified CSS-1 bitumen emulsion content, multiply the calculated content of bitumen emulsion from the formula by the correction factor of residue content 58.6÷64.2. The value of 58.6 is the residue % of conventional bitumen emulsion and 64.2 is the residue % of modified bitumen emulsion.

Hence:
Modified CSS-1 bitumen emulsion content = 10.35 %
Conventional CSS-1 bitumen emulsion content = 12.07%

4.5 The Content of Cement in Slurry Seal Mixture

Portland cement, chalk, and fly ash may all be used as mineral fillers in order to meet the specified aggregate gradation or to modify the time-to-break of the bitumen emulsion. We use Portland cement in this trial in order to achieve the break time of 30 minutes, the time needed in field experiment from mixing to pouring the mixture using locally produced equipment. The result of adding cement filler to Slurry Seal is described in Tables 8 and 9.

Table 8. Bitumen Emulsion CSS-1 Modification

Percent of Cement	Breaking Time	
0	60 minutes	-
0,5	45 minutes	-
1	30 minutes	Optimum
1,5	20 minutes	-
2	15 minutes	-

Table 9. Bitumen Emulsion CSS-1 Conventional

Percent of Cement	Breaking Time	
0	60 minutes	-
0,5	45 minutes	-
1	30 minutes	Optimum
1,5	20 minutes	-
2	15 minutes	-

4.6 Optimising Added Water

Adding water in Slurry Seal is to get mixture with characteristics semi fluid, smooth, homogenous and without emulsion run off. The result of adding water to Slurry Seal is described in Tables 10 and 11.

Table 10. Adding Water for Slurry Seal CSS-1 Modification

Adding Water	Semi fluid	Smoothly	Homogenous	No run off
15	No	No	No	No
20	No	No	Yes	No
24	Yes	No	Yes	No
26	Yes	No	Yes	No
28	Yes	No	Yes	No
30*)	Yes	Yes	Yes	No
32	Yes	Yes	Yes	Yes
34	Yes	Yes	Yes	Yes

Note : *) = Optimum

Table 11. Adding Water for Slurry Seal CSS-1 Conventional

Adding Water	Semi fluid	Smoothly	Homogenous	No run off
10	No	No	No	No
14	No	No	Yes	No
16	Yes	No	Yes	No
18	Yes	No	Yes	No
19	Yes	No	Yes	No
20*)	Yes	Yes	Yes	No
21	Yes	Yes	Yes	No
22	Yes	Yes	Yes	Yes

Note : *) = Optimum

4.7 The Result of Field Experiment of Slurry Seal Mixture

The trial of Slurry Seal was laid in Jalan Jenderal Sudirman KM 1-Cilacap on February 5 1997, in Jalan Gatot Subroto KM 5 Cilacap on May 5 1997 and in Jalan Sutomo KM. 7 Pekalongan. The Slurry Seal was mixed and laid using simple equipment that can easily be built in any part of Indonesia. The equipment is illustrated in Figure 10.

4.8 Pavement Condition and Traffic Before and After the Experiment

The condition of Slurry Seal and traffic when the experiment was done is given in Tables 12 and 13.

Table 12. Visual Condition of Slurry Seal Mixture

Age of Bitumen Emulsion	Slurry Seal Mixture of CSS-1 Modification	Slurry Seal Mixture of CSS-1Conventional
< 3 months	-	good
3 months	good	-
7 months	good	-
9 months	good	-

Figure 10. Simple Equipment for Slurry Seal

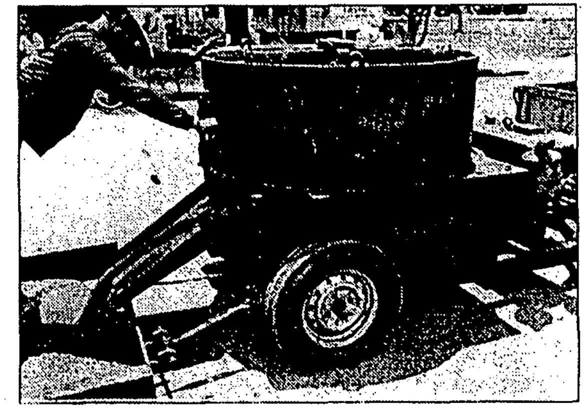


Table 13. Pavement Condition and Traffic Before and After Application of Slurry Seal

No	Survace Condition	CSS-1 Modification 3 Months Age			CSS-1 Modification 7 Months Age			CSS-1 Modification 9 Months Age		
		Before Aplication	After Aplication		Before Aplication	After Aplication		Before Aplication	After Aplication	
			Mod	Con.		Mod.	Con.		Mod.	Con
1.	Texture deepth, Average, cm	0.08	0.06	0.06	0.08	0.08	-	0.07	0.6	0.6
2.	Skid Resistance	47.5	62.5	64.7	48.3	63 7	-	52 9	63 4	65 3
3.	Survace Condition	45.3 % Raveling	good	good	52.2 % Raveling	baik	-	65,3 % Raveling	good	good
4.	Trafic per day	521	521	521	5186	5186	-	5605	5605	5605

Note: Mod = Modification   Con. = Conventional

5. CONCLUSIONS

a) Because of the remoteness of some of its parts, Indonesia needs bitumen emulsion with high storage stability, that can be stored about 6 to 9 months.

b) The storage stability of Indonesian bitumen emulsions, CMS and CSS, can be increased by changing their composition.

c) The optimum composition for CMS-2 bitumen emulsion is 65% bitumen, 7% kerosine, 0.40% emulsifier (Asfier-103), 0.40% H Cl, 0.30% $CaCl_2$ and 27.90% water.

d) The optimum composition for CSS-1 bitumen emulsion is 65,20% bitumen, 2.80% kerosine, 0.48% emulsifier (Redicote), 0.48% H Cl, 0.30% $CaCl_2$ and 30.74% water.

e) The study has shown that bitumen emulsions CMS-2 and CSS-1, with optimum composition, can be stored for up to 9 months.

f) A field experiment demonstrated that changing the composition of bitumen emulsion CSS-1 to that described above does not effect the characteristics of Slurry Seal produced with this material.

g) Slurry Seal, made with the new emulsion, was placed successfully after storing the emulsion for 9 months. A simple mix-and-spread machine was used that is designed for local production.