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Modified Marshall Test assessment for emulsified asphalt cold mixes

K R Usman[1,2], M R Hainin[1*], M K I Satar[1], M Naqiuddin M Warid[1], Suleiman Abdulrahman[1]

[1]School of Civil Engineering, Universiti Teknologi Malaysia, 81310 Johor Bahru, Johor, Malaysia.
[2]Department of Civil Engineering Technology, School of Engineering Technology, Nuhu Bamalli Polytechnic Zaria, P.M.B 1016, Zaria, Kaduna State, Nigeria.
[*]Corresponding author : mrosli@utm.my

Abstract. Asphalt cold mixes are attractive to transportation agencies for their simplicity in set-up, less energy requirement, ease of application, and environmentally friendliness, though it suffers setbacks in terms of lack of a universally acceptable mix campaign, high water susceptibility, and low mechanical strength at initial stages of construction. However, they are widely used for rehabilitation and maintenance works, and to a lesser extent construction in low-trafficked roads. Notable among the cold mixtures is polymer modified emulsified asphalt micro-surfacing, which is used as a thin non-structural layer for the restoration of skid resistance and waterproofing. Marshall test is used to assess its performance similar to a structural wearing course layer. This study assessed the mechanical performance of cold mixtures by a modified Marshall mix employing a low viscosity cationic quick set emulsion (CQS-1h) and a cationic rapid set emulsion (RS-1k) and a 60/70 penetration grade binder. Rheological criteria assessment was conducted on both emulsions including particle charge, residue by evaporation, settlement and storage stability, solubility in inorganic solvent sieve test, and Saybolt Furol viscosity, while the residue from evaporation was tested for microstructural and mechanical performance, 65% and 63% residue by evaporation, dissolved in trichloroethylene at 95% and 100% for CQS-1h and RS-1k respectively.

1. Introduction

Cold asphalt mixtures evolved out of the need to eschew unsustainable practices associated with Hot Mix Asphalts (HMAs) and Warm Mix Asphalts (WMAs) among which includes greenhouse gas emission, hazardous fumes emission, difficulty in maintaining compaction temperatures for long haulage distances, huge energy requirement and plant requirement for its placement. WMA technology is in-between hot and cold mix asphalt which is designed to satisfy the HMA requirement at the lowest environmental impact and cost savings from reduced energy usage [1-2]. Cold emulsified asphalt slurry mixtures afford the additional advantage of less energy usage and zero pollution. They are simple to set up even in the remotest locations, cost-effective, minimum skills needed for their application and most importantly, their application is often not affected by wet weather [3]. Night application is possible with polymer improved emulsified CAMs [4-5], thus, in spite the desirability of WMA, polymer-modified emulsified cold slurry (PMECS) mixtures offer even better benefits.

Antecedents have it that polymer modified emulsified slurry mixtures were first explored in Germany towards the end of 1960s and early 1970s using highly selected well-graded dense aggregates including fillers, enhanced polymers, and notably cationic quick setting bitumen emulsion

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[6-7]. This was what later known to be as emulsified asphalt micro-surfacing. They were designed to cover entire road width whilst not destroying the expensive road markings on autobahns [8]. Subsequently, cold emulsified micro-surfacing spread to other parts of Europe, America, Australia, Africa, Asia, and France.

To date there is no universally acceptable mix design procedure and acceptance criteria requirement for cold asphalt mixtures (CAMs), mix design is usually carried out as a one-off, depending often on the type of aggregate available, gradation needed, and the nature of road to be applied on.

2. State-of-the-art

Cold asphalt micro-surfacing is a rejuvenating layer used for protecting the structural part of the pavement whilst enhancing its serviceability as mentioned in the previous section. So far, numerous researches and even the industry uses polymer-modified quick set and slow to medium set emulsions [9] for micro-surfacing and cold mix maintenance mixtures respectively [6,10-11]. Among the overarching issues with cold mix, patching mixtures remain the low early strength, slow rate of curing, slow rate of strength gain, high void/porosity, and high moisture susceptibility.

Specification for cold mix asphalt micro-surfacing as specified by ISSA A 143 is presented in Table1

Table 1. Requirements for emulsified asphalts and residue for use in cold mix asphalt.

TEST	STANDARD SPECIFICATION		SPECIFICATION
	ASTM	AASHTO	JKR/ISSA A 143
TEST ON EMULSIFIED ASPHALT			
Particle charge	D7402		Positive
Saybolt Furol Viscosity	D 7496	T 59	15 – 50 secs
Storage stability (1 day)	D 6930	T 59	1% Maximum
Settlement test (5 days)	D 6930	T 59	1% Maximum
Residue by evaporation	D 6934		62% Minimum
TEST ON EMULSIFIED ASPHALT RESIDUE			
Penetration	D5		40 - 90mm
Softening point	D 36		57 ^{0}C/54 ^{0}C (135 ^{0}F) Min.
Viscosity (rotational)	D 4402		N.A
Solubility in trichloroethylene	D 2042		90% min

For decades, the tradition has been the use of quick set emulsion in cold mix pavement maintenance and micro surfacing mixtures [12-13], in addition, medium to slow set is also applicable in patching cold mixtures, but the use of a rapid and quick set emulsions is quite uncommon in cold mixtures. Thus, the standard requirements pulled from both ISSA A143 and JKR as depicted in Table 1 is used as a guide to accessing the potentiality of employing these grades of emulsions in comparison to CQS-1h for such purposes.

3. Materials and experimental detail

3.1 Bitumen Emulsions

Emulsions were originally developed at the beginning of the nineteenth century traditionally for dust control in farms and rural access paths to strengthen the surfaces and to reduce respiratory diseases associated with dust inhalation, by the end of nineteenth-century emulsions gained acceptance for paving applications.

Emulsions are dispersions of tiny repulsive globules of bitumen ‘discontinuous phase’ in water ‘continuous phase’ kept in suspension with the help of a surface-action agent (SurFactAnt) commonly referred to as an emulsifier. The stable suspended discontinuous phase become destabilised by chemical alteration, by contact with aggregate (substrate of opposite charge), and by mechanical agitation [14]. Granite aggregate is used for mixtures in this study.

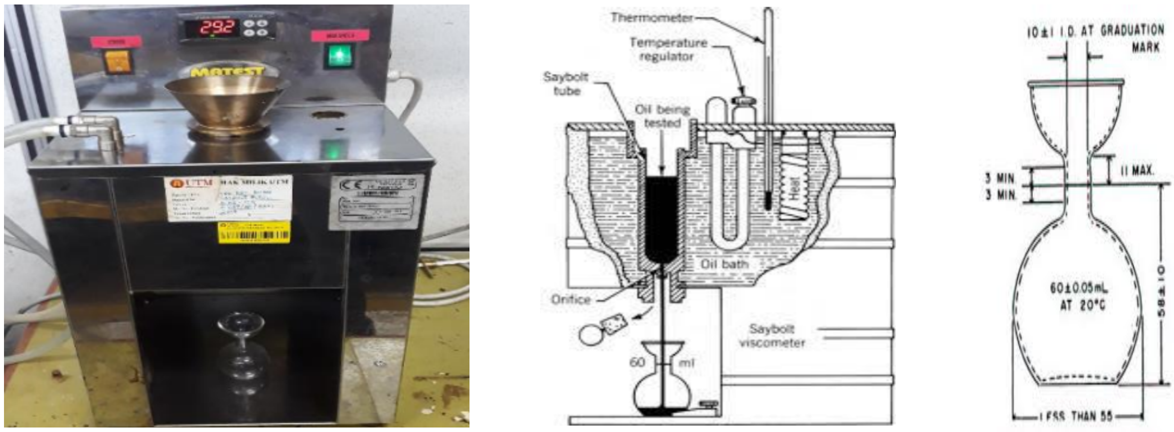
Dense-graded samples were mixed at the optimum binder content under ambient temperature, wrapped in foil paper, cured at 40ºC in a forced draft oven for 24 hours after which they are compacted at 50 blows per face and tested for Marshall parameters. Results were compared with the public works department’s requirements (Jabatan Kerja Raya) JKR-2008 for HMA mixtures and International Slurry Surfacing Association (ISSA A143) specifications for emulsified asphalts emulsions used for cold mix emulsified asphalt micro-surfacing mixtures used for pavement maintenance.

3.1.1 Particle Charge (ASTM D 7402-09).
The emulsified asphalt in this study is conditioned at a temperature of 25ºC in a water bath before being tested. Conditioning and testing were conducted based on ASTM D 244 [15] and later ASTM D 7402-09. The sample under testing and a visible emulsion deposition on the cathode at the end of the test are depicted in Figure 1(a) and (b) respectively. An initial current setting of 8 mA was used; a stop-clock is used to time the immersion for up to 30 minutes. The test is considered complete by taking the earliest of either the current or stop clock to reach 2 mA or 30 minutes respectively. The deposition of asphalt on the negatively charged electrode indicates a cationic emulsion, while an anionic emulsion will register a visible deposition on the anode electrode.

(a) (b)

Figure 1. (a) Emulsified asphalt under Particle charge testing, (b) visible deposition of emulsion on the electrode after the test.

3.1.2 Saybolt Furol Viscosity (ASTM D2397 -13).
Saybolt Furol measures the time in seconds in which an emulsified asphalt at certain specified temperature will flow under gravity and fill a 60ml pycnometer via a distance of 125mm through a standard notched orifice. Sample conditioning and safety measures were exercised by utilising a representative sample from both emulsions as specified in ASTM D2397 – 13. Test results are presented in Say bolt Furol Seconds (SFS), the specification spelled out a minimum and maximum SFS to be 20 and 100 seconds respectively for emulsions used in cold mixtures. The apparatus for Say bolt Furol and its schematics are shown in Figure 2 (a) and (b). The time was measured with a stop-clock at the point the emulsion reaches the ring line on the bottle as in Figure 2(C).



(a) (b) (c)

Figure 2. (a) Experimental set-up for Say Bolt Furol apparatus (b) Schematics (c) pycnometer details

3.1.3 Storage Stability and Settlement Tests (ASTM D6930-10).

The storage stability test is essential for ascertaining the suitability of emulsion for cold mixtures. The stable charges imparted by emulsifiers are meant to break upon contact with any substrate of opposite charge, this could occur due to pH change, hydrophilic-lipophilic balance or acid addition [14], intense mechanical agitation and coalescence due to expiration [7,16].

Conditioned emulsion samples were filled in the glass cylinders and kept on a flat surface, allowed to stand undisturbed at room temperature for 24 hours for storage stability and five days for settlement. After the designated time, 50g of the settled emulsion was removed from the top and bottom of the cylinder and put in a clean, previously weighed clean 1000ml glass beaker containing a glass rod. These samples were then subjected to 3hours oven drying at 165ºC + 5ºC, after which the percentage residue from each beaker computed.

Both JKR [17] and ASTM [15] specifications require that this percentage be less than 1% in any case, otherwise, the emulsion integrity is in question, hence, it may be rejected for use in cold mixtures.

3.1.4 Residue by evaporation (ASTM D6934-08).

This test measures the bitumen content in an emulsion by subjecting a sample to heating. After conditioning the previously stirred emulsion to achieve homogeneity, 50g each was weighed into a clean 1000ml glass beaker weighed empty and together with a glass rod. A total of six beakers – three each for RS-1K and CQS-1h emulsions were placed in an oven set at 165ºC and heated for 3 hours, cooling at room temperature and subsequent weighing comes thereafter.

The result is reported in percentage representing the amount of asphalt cement in the initial emulsion .

3.1.5 Penetration test on residue (ASTM D5-10).

The residue obtained from both emulsions was sieved through a 300µm sieve, cooled and stored in clean labelled containers for subsequent testing.

The residue was made sufficiently fluid by heating to less than 60ºC and poured into 55 x 35mm containers, allowed to cool at ambient temperature for 1.5 hours. Triplicate samples were prepared for both RS-1k and CQS-1h emulsion residues in labelled penetration cups as specified in ASTM D5. An average value for three readings was taken to be the penetration value.

3.1.6 Softening point test (ASTM D36-08).

Ring and ball apparatus was used for the softening point tests on residues from both RS-1k and CQS-1h emulsions. It records the average temperature at which two steel balls, housed on brass rings supported on a flat metal plate passes through the softened bitumen and just touches the base of the apparatus.

Distilled water was used in a 500ml beaker, whilst a central metal cover guides a centrally placed thermometer to the top surface of the plate holding the rings and also supporting the two balls on either side. The temperature of the water was brought to 5°C at the beginning of the test and a rate of heating of 5°C + per minute was maintained up to the point of failure. Two test set-ups were conducted and the average reported.

3.1.7 Viscosity test (ASTM D4402-02).
Measurement of the ratio between the force per unit area (shear stress) and the rate of speed at which the intermediate layers of the asphalt move with respect to each other measured in reciprocal second (sec-1) respectively gives the apparent viscosity of the Newtonian or non-Newtonian fluid. It is an important test to know the behaviour of the binder during handling, mixing and compaction on site.

Triplicate samples of 11g each were prepared in steel moulds, conditioned in the oven at 135°C for 2 hours, transferred into a thermostatically controlled chamber, normalised before running the test. Spindle number 24 was used and a starting temperature of 135°C and 165°C at 20 revolutions per minute.

3.1.8. Solubility in trichloroethylene (ASTM D6934-08).
The ability of the asphalt emulsion residue to dissolve in technical grade trichloroethylene signifies the presence or otherwise of active to non-active components in the emulsion. A high percentage of insoluble material signifies the inability of the binder to efficiently coat aggregate – which is regarded as an important parameter in cold mixes [18], especially in quantifying moisture susceptibility[19].

The percentage of insoluble material in the residue from emulsion obtained at 110 °C $\pm$ 5 °C oven drying and filtration signifies the presence or otherwise of unwanted material in the base bitumen, thus, emulsion quality.

4. Results and discussion

Result obtained is compared with both specifications set in JKR-2008 (the Malaysian Road Transport standard specification for Roads – section 4: flexible pavements) and the ISSA A143 standard as depicted in Table 2. A result comparison of the two emulsions was conducted to ascertain the level of correlation or otherwise of the two results.

Furthermore, the lesser Saybolt Furol Seconds (SFS) for CQS-1h as compared to RS-1k could not be far-fetched from the fact that globules of asphalt were well dispersed within the continuous phase (water) more than RS-1k leading to a frictionless and fast flow in lesser time (sec) than RS-1k. Generally, the five (5) days settlement result for both emulsions was the same, coinciding with the peak limit of the specification of 1.0%.

Interestingly, the residue by evaporation result for RS-1k was above the minimum requirement of 62% by a unit increase, in spite of the fact that CQS-1h has a very thick viscous residue, which literally ought to be heavier than RS-1k residue, but, the reverse was the case in actuality.

The viscosity measured – though not a criterion set by either of the earlier mentioned specifications, yet, it gives a vital information for mixing and compaction, especially, if modified Marshall test is intended to further study the mechanical behaviour of the emulsion residue as suggested by Kumar and Ryntathiang [20] and highlighted in ISSA A143.

4.1. Comparison of the rheological and mechanical properties of the rapid set and quick set emulsions

Both RS-1k and CQS-1h exhibited similar rheological characteristics, perhaps, owing to the nature of their respective formulation parameters. The penetration value for CQS-1h was rather harder than RS-1k, but more promising was the fact that the result for solubility in trichloroethylene indicated that RS-1k has a more active constituent than CQS-1h as it dissolved fully in this hydrocarbon solvent as presented in Table 2.

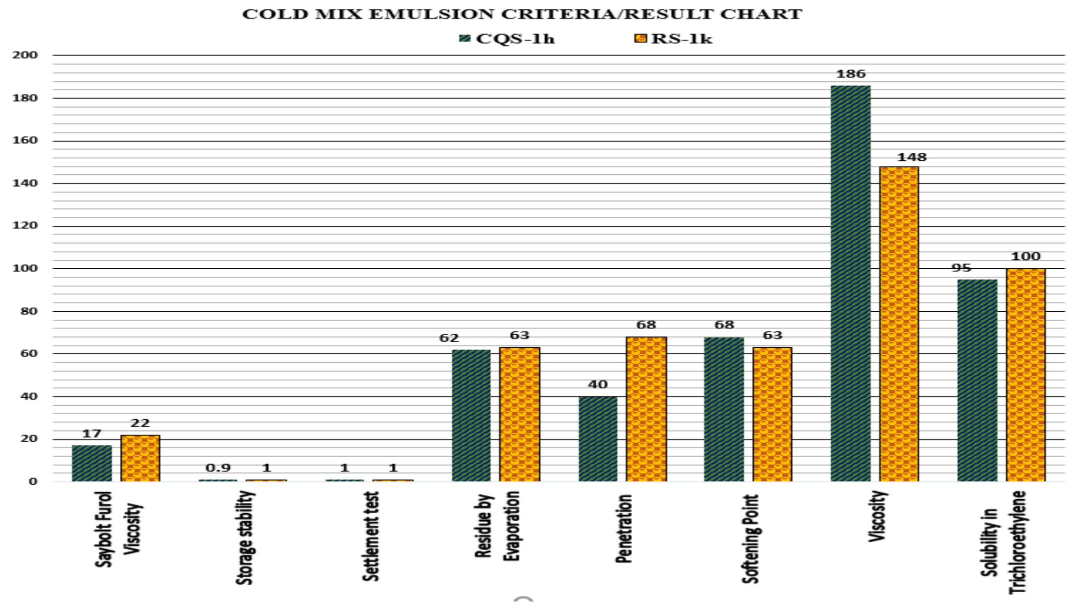


Figure 3. Emulsion and emulsion residue test result

Significant margins are noticeable in the viscosity of residue, followed by a penetration test, and solubility test as depicted in Figure 3. Nevertheless, no marked difference was recorded for the remaining tests; however, both emulsions have passed minimum requirements set by the requirements as in Table 2.

Table 2. Rheological result of emulsion and their residues

TEST	STANDARD SPECIFICATION		RESULT		JKR/ISSA A143 SPECIFICATION
	ASTM	AASHTO	CQS-1h	RS-1k	
Particle charge	D7402		Positive	Positive	Positive
Saybolt Furol Viscosity	D 7496	T 59	17	22	15 – 50 secs
Storage stability (1 day)	D 6930	T 59	0.9	1.0	1% Maximum
Settlement test (5 days)	D 6930	T 59	1.0	1.0	1% Maximum
Residue by evaporation	D 6934		62	63	62% Minimum
TEST ON EMULSIFIED ASPHALT RESIDUE					
Penetration (mm)	D 5	T 49	40	68	40 - 90
Softening point	D 36	T 53	68	63	54%/57% minimum
Viscosity (rotational)	D 4402		186/198*	148/160*	N.A
Solubility in trichloroethylene	D 2042		95	100	95% minimum

* these means mixing/compaction temperatures

4.2. Modified Marshall Result.

The Marshall Stability results in figure 4 indicated that the stability of RS-1k is in close range to that of the control bitumen, but perhaps, due to greater coating and adhesion of the CQS-1h is what leads to lower stability values.

Table 3. Modified Marshall results for (a) 60/70 bitumen, (b) Quickset emulsion, and (a) Rapid set emulsion

From Graphs	60/70 Bitumen (a)		CQS-1h Emulsion (b)		RS-1k Emulsion (c)		JKR Specification
Stability, S	11500 N	ok	9900 N	ok	1200 N	ok	> 8000 N
Flow, F	3.8 mm	ok	3.7 mm	ok	4.0 mm	ok	2.0 - 4.0 mm
Stiffness, S/F	2530 N/mm	ok	2150 N/mm	ok	2850 N/mm	ok	> 2000 N/mm
Air Void in Mix (VTM)	4.6 %	ok	4.5 %	ok	12.5 %	Not ok	3.0 - 5.0 %
Voids in Aggregate Filled with Bitumen (VFA)	74%	ok	75.5%	ok	63%	Not ok	70 - 80 %

Form Table 3 it can be deduced that in spite of the fact that mixtures of CQS-1h have lower stability compared to control and RS-1k mixtures, yet, it satisfied all the basic requirements set out by JKR for AC10 HMA mixtures. The flow exhibited by the CQS-1h mixtures perhaps lead to a lesser mixture stiffness, which narrowly passed with an increase of 150 N/mm

5. Conclusion

The rheological evaluation of the two grades of emulsion indicated a high potentiality of rapid set emulsion RS-1k to be used for both cold mix asphalt and in emulsified micro-surfacing mixture with a little or without any polymer modification. The residue base of RS-1k was higher than even the CQS-1h, perhaps, the little higher Say bolt Furol seconds signifies a more viscous feature than the CQS-1h. it can be concluded that RS-1k emulsion and its residue exhibited similar behaviour to CQS-1h, as such can be tried for use in cold mix emulsified asphalts and even micro-surfacing mixtures. Though, the latter may require some form of polymer modification. The penetration values of the residues indicated that RS-1k is possibly formulated from a 60/70 penetration grade bitumen, as such can render self for further testing like the modified Marshall test for cold mix slurry mixtures.

High air voids were obtained with RS-1k and this could be attributed to poor coating and bonding of the aggregate by the emulsion. Moreover, CQS-1h exhibited more desirability for use in cold mix asphalt for wearing surface because it has a comparable void content to the control.

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