LABORATORY STUDIES ON SOLVENTLESS DENSE GRADED COLD MIX USING REDICOTE E-4868 EMULSION

1.0 Introduction

The use of cold mix in road repair and construction in desirable for environmental, economic and logistical reasons (refs). The manufacture of fully coated densely graded mixes requires a very special type of bitumen emulsion in the absence of any solvent. To be able to fulfil this requirement an emulsion must be very slow setting to prevent premature break during mixing. Unfortunately this results in very slow curing of a mix after laying and compaction.

The use of cement, and to a lesser extent hydrated lime, have long been known to improve the setting rate and water resistance of cold mix (Refs). These claims have been shown to be true for Redicote E-4868 emulsion mixes. Further studies have been carried out to look at the behaviour and properties of the cement-water-emulsion mastic in the absence of aggregate. A new technique has been developed to measure the effect of compaction loads and cement on the coalescence rate of bitumen emulsion onto an aggregate. The performance of emulsions made with bitumens from two different crudes and the beneficial effects of cement have been assessed.

2.0 Materials

2.1 Ordinary Portland cement - OPC

2.2 Aggregate - Granite from ECC's quarry at Croft in Leicestershire?

Porphyritic granite from Bardon Hill quarry in Leicestershire.

20, 14, 10, 6 mm, dust and < 300 µm fractions were used.

2.3 Bitumen Emulsion

100 pen bitumens from Venezuelan and Middle Eastern crudes were used.

Bitumen		62%
Redicote E-4868		1.2% on emulsion
c. HCl	20 pH	2.5 on Waterphase
Water	20	100%

3.0 Experimental

3.1 Curing behaviour of mixes

The Nottingham Asphalt Tester (NAT) was used to non-destructively test samples their curing period to observe the stiffness modulus build up over time.

Densely graded Granitic aggregate from ECC's Croft quarry was dry mixed with cement in a Hobart mixer. Pre-wet water was added and mixed in to evenly wet the aggregate. Redicote E-4868 bitumen emulsion was then added and mixed until maximum coating had been achieved (usually 1-2 mins). Mixes were compacted in Marshall moulds with 50 blows of a Marshall hammer to each side of the specimen. Cores were left in their moulds overnight before being extruded. Samples were then stored at 20°C and 40-50% relative humidity throughout the curing period.

Hot mix cores were produced in the same way using 100 pen Venezuelan bitumen and a mix temperature of 140°C.

Cores were tested for stiffness modulus of various time intervals on the NAT. The test temperature was 20°C and the target deformation was normally 10 μm.

3.2 Cement, water, emulsion mixes.

Mixes of cement, water and emulsion were produced to assess the breaking behaviour and final penetration of the mastic.

Pre-wet water was added to cement and mixed thoroughly. As cement:emulsion ratio was increased the amount of water added was increased accordingly to improve workability. Emulsion was then added and mixed for a further 2 mins. Samples were sealed and inspected at various intervals. The percentage break of the mix was estimated according to the viscosity and general consistency. After break had occurred in all samples, the break water was poured off and the penetration of the cured mastics measured.

3.3 Bitumen Coalescence.

A method has been developed to measure the degree of bitumen coalescence occurring in a mix immediately after compaction.

A 300 μm to 1.18 mm fraction of Porphyritic aggregate from Bardon Hill quarry was thoroughly rinsed with water to ensure complete removal of < 300 μm material, and dried in an oven at 120°C. A 200-g portion was then dry mixed with cement (if required) before addition of pre-wet water (2 g) followed by bitumen emulsion (20 g). The mix was then placed in a Marshall mould and compacted using a static load press. The core was immediately removed from the mould, broken up using a spatula and placed in a glass jar. A 2% quaternary amine solution (300g) was then added and the jar rolled for 20 mins to thoroughly redisperse any un-coalesced bitumen. The washed mix was then repeatedly rinsed with water and decanted until all free bitumen had been removed. The samples were then dried to constant weight in an oven at 60◦C.

The retained bitumen content was analysed by Xylene extraction using Soxhlet apparatus.

3.4 Effect of bitumen type

A range of tests were carried out to assess the effect of bitumen type on mix properties of an emulsion.

Small scale mixes were produced using 200 g portions of densely graded Porphyritic aggregate mixes from Bardon Hill quarry. Cement (1%) was added, if required, followed by pre-wet water (2.5%) and bitumen emulsion (8.1%). The mix times and coating were noted. Samples were fully cured in an oven at 60◦C and then subjected to boiling in water for 3 mins after which the retained coverage was estimated.

Emulsion adhesion tests were performed by coating washed and dried 14 mm Porphyrite with 8% emulsion and curing in an oven at 60◦C over night. The coated stones were then covered with water and again stored overnight at 60◦C. The remaining percentage coverage was estimated.

Marshall type specimens were fabricated using the method detailed above using a densely graded Porphyritic aggregate mix. Cores were tested after days storage at 60◦C. Other cores were subjected to vacuum saturation under water for ½ hr followed by 24 hrs at normal pressure. Testing was carried out on the Marshall apparatus at 60◦C.

3.5 Zeta potential measurements

In order to investigate the electrochemical effect of cement on a bitumen emulsion Zeta potential measurements were made.

OP cement was added to bitumen emulsion and left to stand for 2 hrs. Samples were taken and dispersed in distilled water. The pH of the dispersion was adjusted as required, using 0.1 μ HCL or 0.1 NaOH, and samples taken for Zeta potential measurements on a Brookhaven Zetaplus.

Stated briefly, Zeta potential is a measurable property of particles dispersed in water, which is related to the actual surface charge on these particles.

4.0 Results and discussion

4.1 Curing behaviour of mixes.

Figure shows the stiffness moduli of cores with different levels of cement over a number of weeks.

Fig 1 : Stiffness modulus v time for mixes with different cement contents.

It can be seen from figure 1 that the stiffness modulus of the cold mix cores increased with time. The rate of this increase was faster with increasing cement addition level. The overall stiffness at any particular time was also increased by cement.

The stiffness of the hot mix cores remained constant over time and the addition of cement did not have any significant effect. This result indicates that the cement acted as a hydraulic binder in the cold mix cores and not merely as additional filler.

4.2 Cement, water, emulsion mixes.

Figure shows that the rate of break of the bitumen emulsion increased with increasing cement level. At cement levels above 4% the mixes showed complete break in under 6 hrs. With more realistic levels of 1 & 2% cement the break times were only 24 and 48 hrs respectively.

The fact that resilient modulus tests of cores produced from actual mixes showed a steady increase over many weeks suggests that although the cement, water, emulsion mastic incapable of curing within 48 hrs, the evaporation of the break water takes a lot longer. This may indicate that less dense aggregate gradings may be favourable but other tests have shown that more open textures perform less well due to inferior aggregate packing stability. Particularly in early life the stability of a cold mix is dependent on the aggregate structure.

It can be seen from figure that the penetration of the cured mastic decreased rapidly with increasing cement. At 2-3% cement the penetration was equal to that of the original bitumen used in the emulsion.

Figure : Penetration of cured cement, water, emulsion mixes

4.3 Bitumen coalescence

From figure it can be seen that the degree of coalescence onto the aggregate increased with increasing compaction loads. Emulsion coalescence is therefore dependant on compaction load.

Figure :   Percentage binder content on aggregate v compaction load.

4.4 Effect of bitumen type

Shows the results of comparative tests between emulsion made with bitumen extracted from Venezuelan and Middle Eastern crudes with and without cement. Venezuelan bitumen was superior in terms of initial coverage and resistance to stripping under the action of water. This did not carry through to the Marshall tests as the difference here was not significant.

Cement was found to have a beneficial effect on adhesion and Marshall stability performance. However, a reduction in initial coating was observed due to shortened workability of the emulsion caused by accelerated break.

Figure :   Comparison of Venezuelan and Middle Eastern bitumen emulsions.

4.5 Zeta potential measurements

From figure   it can be seen that the additional of cement had a noticeable effect on the Zeta potential of a bitumen emulsion. At weakly acidic to neutral pH's (ie 4-7), the emulsions with cement are less negative or even cationic. This may be one of the causes of the increase in curing rate found in mixes with negative siliceous aggregates incorporating cement. An emulsion with some cationic sites would have a greater affinity for a negative aggregate giving rise to increased rate of coalescence of emulsion onto the aggregate surface.

Figure   :   Zeta potentials of bitumen emulsions with and without cement.

5.0 Conclusions

From the studies carried out the following conclusions can be drawn.

5.1 Very well coated, workable cold mixes can be produced using Redicote E-4868 emulsion on true densely graded granitic aggregate mixes.

5.2 The curing rate, final strength and resistance to water damage of compacted mixes can be improved by the addition of OPC.

5.3 OPC decreases the break time of bitumen emulsion and produces a harder composite binder on curing.

5.4 Coalescence of a bitumen emulsion onto an aggregate is dependent on the degree of compaction.

5.5 The addition of OPC to a bitumen emulsion makes it more cationic in nature.

5.6 Bitumen emulsions containing Venezuelan bitumen are superior to those with Middle Eastern in terms of coating and adhesion. Mechanical properties of mixes are little affected.