PRIMARY RESEARCH ON A BINDER EXTRACTION METHOD FOR ASPHALT MIX MODIFIED BY USING SEAM™ ASPHALT MIX MODIFIER

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ABSTRACT

The existing bitumen extraction methods, including solvent extraction, the ignition method, the nuclear asphalt content device and biological resolving were analysed and examined for their applicability in determining the asphalt and SEAM contents of asphalt mixtures modified with sulphur-extended asphalt modifier. In SEAM-modified mixtures, sulphur separates out scatters in the mixture and dissolves in asphalt at a temperature lower than 115 °C. Sulphur can dissolve in sodium hydroxide and react chemically with new materials. In this study, the NaOH solvent extraction method was chosen to determine the SEAM content. The conventional solvent extraction method was used to determine the total amount of asphalt and SEAM. Detailed procedures and the testing precision of the NaOH solvent extraction method are given. The test method proposed in this study can be used in highway construction to control the SEAM content.

Keywords: SEAM-modified mixture, SEAM content, sodium hydroxide solutions, extraction method.

1. BACKGROUND

SEAMTM is a pellet product for asphalt mix modification. It is the abbreviation for Sulphur-Extended Asphalt Mix modifier. It is a patented product whose constituents are sulphur, emission scavenger and plasticiser. SEAM modification does not produce an emission hazard when the SEAM asphalt mix operation temperature is below 150°C. After several road tests in China, it was found that SEAM-modified mixture did not corrode the asphalt plants and construction equipment, although this could occur if the materials are recycled. The plasticiser increases the quality of the sulphur and the strength and durability of sulphur-modified materials (Shi, 2005). The asphalt content or asphalt aggregate ratio of a conventional asphalt mixture is normally determined by the solvent extraction test (McGraw et al., 2001). Reagents commonly used in this type of test method are trichloroethylene, benzene, gasoline, etc. In this method, asphalt is extracted from the asphalt/aggregate mixture. However, SEAM can partly dissolve in trichloroethylene (Hu & Liu, 2003) at room temperature. The SEAM content cannot be precisely determined with the solvent extraction method. Other test methods for determining binder content are burning in an ignition oven (Brown & Mager, 1996), the nuclear asphalt content device (Bahia & Stakson, 2003) and biological resolving (Prowell, 1997). In the ignition method, the binder (mainly asphalt) burns off and then the total asphalt content is gasified at a high temperature. For SEAM asphalt mixes, SEAM (sulphur and other small percentages of additives) is also gasified together when the asphalt burns off. Therefore in this method the SEAM content and the asphalt content could not be separated. In the method of combining the nuclear asphalt content device and the ignition oven, both the SEAM content and the asphalt content in the SEAM asphalt mixture can be determined. However, a nuclear asphalt content gauge is rarely used in China because it contains radioactive materials and the results are influenced by various of factors such as the source of the asphalt, aggregates, sample size, gradation and human error. The reproducibility of the results is poor between different nuclear asphalt content devices. The biological resolving method is also a solvent extraction method. This method is very time consuming and its accuracy is not good either (Shi & Lv, 1999). Therefore none of the conventional extraction methods mentioned above are suitable for checking the SEAM/asphalt content in a SEAM asphalt mixture. Up to now, a proper method of determining the SEAM content and the asphalt content in an asphalt mixture modified by SEAM has not been found.

2. OBJECTIVE OF THIS PAPER

This paper describes the investigation procedure for finding a suitable method for determining the SEAM and asphalt content separately in a SEAM asphalt mixture. In this study it was found that a sodium hydroxide solution is effective for determining asphalt and SEAM content (General Administration of Technical Supervision, 1990). This method may therefore be further developed into a precise laboratory test method for daily/routine quality control of SEAM asphalt mix production.

3. EXPERIMENTAL INVESTIGATION

3.1 Methods

SEAM (the main constituent is sulphur) was blended with a hot asphalt mix at a high temperature (140 ^oC). Sulphur is present in SEAM asphalt mixture in two parts when the sulphur content in the asphalt is high enough (when the sulphur ratio is more than 30% by weight of asphalt cement): The first part is about 18% of the sulphur dissolved in the asphalt as an asphalt extender, which makes the asphalt thinner and decreases the binder viscosity. The remaining part of the sulphur (liquefied) is dispersed as micro-sulphur liquid droplets in the asphalt (already diluted by the sulphur). The melting point of sulphur is about 115 ^oC. Below the melting point, sulphur scatters in the mixture and dissolves in the asphalt. It eventually separates out and forms crystals (see Figure 1). Sulphur can react chemically with sodium hydroxide solutions and result in combinations such as Na_2S_2 and Na_2SO_3 (Sawada et al., 2001), which are soluble in water.

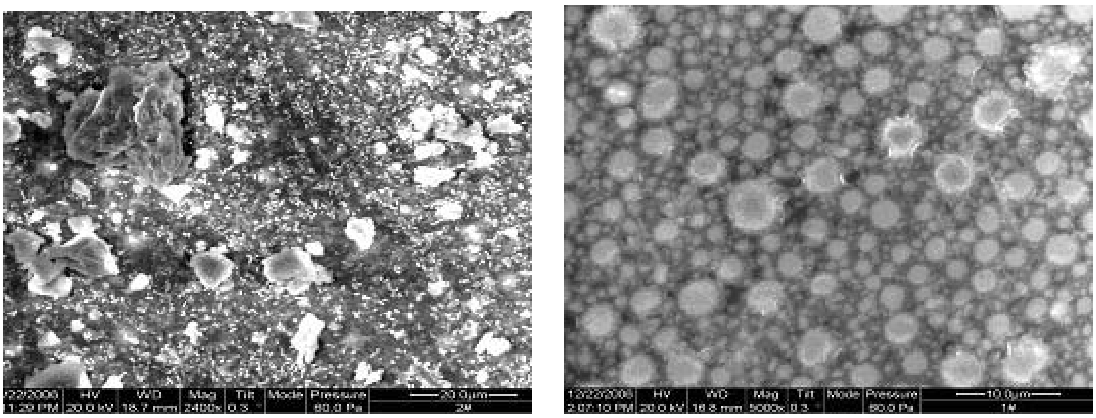

Figure 1. SEM photos of SEAM asphalt mortar at temperatures below the melting point of sulphur

3.2 Apparatus and reagents

3.2.1 Apparatus

1. Beaker: 3 000 ml
2. Vacuum filtration device
3. Oven, capable of maintaining the temperature
4. Balance, conforming to the accuracy requirements
5. Extracting apparatus
6. Graduated cylinder: 1 000 ml; filter paper: medium grade, fast filtering, 330 mm diameter; glass stirring rod; metal plate, etc. (see Figure 2).

Figure 2. Vacuum filtration device

3.2.2 Reagents

Solid sodium hydroxide
Trichloroethylene
Distilled water.

3.3 Test procedures

1. Two metal plates and a 3 000 ml beaker were weighed on the balance. They were labelled as m_1 (for metal plate 1), m_2 (for metal plate 2) and m_3 (for 3 000 ml beaker).
2. Various concentrations of NaOH solutions were prepared. In order to prepare the optimal concentration, two concentrations were used: 40/60 and 50/50 (solid NaOH/distilled water). The NaOH solutions were prepared in 3 000 ml beakers ready for use.
3. SEAM mixtures were weighed, noted as m_{mix}, and added to the NaOH solutions. The SEAM asphalt mix in the NaOH solutions was stirred thoroughly using a glass stirring rod. It was not stirred continuously. Stirring was stopped and restarted at intervals of 5 to15 minutes. Three reaction times of 1 hour, 3 hours and 6 hours were tried to determine a suitable time for the sulphur reaction in the NaOH solution.
4. The filter paper was folded along its diameter and then twice again, one fold over the other to make three segments. A hollow three-ply cone with a single-ply seam was formed (McBee et al., 1980).
5. The solutions were filtered by the vacuum pump. When the reaction (sulphur and NaOH) was completed, the solutions (with asphalt mix particles) were poured into the filter paper cone. After the first filtration there was still some sodium sulfide solution (after the reaction) left in the mix (in the beaker). After this, distilled water was poured into the beaker and the solutions were mixed for a while and then filtered again. Filtration was stopped once the asphalt mixture solutions were clear (see Figure 3). Clear liquid in the beaker indicated that it was basically free of sodium sulfide. The sulphur in the SEAM asphalt mix was mostly removed. Finally, the clear liquid in the beaker was filtered again and asphalt mix was left in the beaker.

6. The filter paper containing fine aggregate, mineral filler and asphalt was cleared in the metal plate filled with water, and the cleaned filter paper was discarded. The mixture in the bottom of vacuum filtration device was poured into the other metal plate. Finally, the two metal plates and the beaker were placed in the oven in which the temperature was held at 145 ^oC and did not exceed 150 ^oC. Once the moisture in the blending material had been removed, the plates and the beaker were taken out of the oven.
7. The two metal plates and the beaker containing asphalt mixtures were weighed again and labelled as m_4 (for metal plate 1 with asphalt mix particles) m_5 (for metal plate 2 with asphalt mix particles) and m_6 (for beaker with asphalt mix particles). The SEAM content was obtained by the following formula:

   $m_4 – m_1 = m_7$

   $m_5 – m_2 = m_8$

   $m_6 – m_3 = m_9$

   SEAM weight (removed from SEAM asphalt mix) = $m_{mix} – (m_7 + m_8 + m_9)$
8. The total SEAM and asphalt contents were determined according to the criteria of the traditional trichloroethylene extraction method (Ministry of Communications, 2000).
9. Both the SEAM content and the asphalt content of the SEAM modified asphalt mix were obtained.

Figure 3. Clear mixture solutions

3.4 Notes

1. Because NaOH solutions have a high concentration and is strongly corrosive, attention should be paid to safety during the test and the solution should be prevented from splashing anywhere on the body.
2. NaOH is a strong alkali, which absorbs water very easily and then releases a lot of heat. Therefore when the NaOH solutions are being made, water is first poured into the container and then the NaOH powder is poured into the water. This order must not be reversed.

4. RESULTS AND DISCUSSION

4.1 Mix gradations

AC-25 mixes and modified AC-13 were selected for the test. Limestone aggregates and 70 pen asphalt binders were used in the mixtures. The gradations of the mixtures are shown in Table 1.

Table 1. Gradation of SEAM mixtures

Mixture types	Proportion of aggregate(%)						Asphalt (%)	SEAM (%)
	31.5~16	16~9.5	9.5~4.75	4.75~2.36	2.36~0	Mineral filler		
AC-25	20	26	17	10	24	3	2.79	1.86
AC-13	/	22.5	34.5	12	27.5	3.5	3.73	1.60

4.2 Test results and discussion

The test results are given in Tables 2 and 3.

Table 2. Results of the NaOH extraction tests

List	**1**	**2**	**3**	**4**	**5**	**6**
Mixture type	AC-25	AC-13	AC-25	AC-13	AC-25	AC-13
Mixture weight (g)	1 002.4	1 000.5	1 001.2	1 000.7	1 000.8	1 003.6
Concentration of NaOH solutions	50:50	40:60	40:60	40:60	40:60	40:60
m_1 (g)	618.9	641.9	641.9	605.8	605.8	618.9
m_2 (g)	431.7	376.1	375.9	453.0	340.9	340.6
m_3 (g)	453.0	522.5	522.5	437.3	453.0	451.2
Soaking time (h)	1	1	3	3	6	3
m_4 (g)	459.5	533.7	531.2	452.4	463.5	464.7
m_5 (g)	432.8	378.6	377.4	456.7	343.2	343.5
m_6 (g)	1 598.8	1 615.8	1 615.7	1 573.3	1 574.9	1 591.9
SEAM extraction weight (g) – m_s	14.9	12.9	17.2	14.4	18.9	14.2
SEAM calculation weight (g) – m_{st}	17.8	15.2	17.8	15.2	17.8	15.2
E_s (%)	-16.3	-15.1	-3.4	-5.3	6.2	-6.6

Table 3. Results of the trichloroethylene extraction tests

Mixture type	**Mixture weight (g)**	**SEAM and asphalt extracted weight (g)**	**Asphalt extracted weight (g) – m_a**	**Asphalt calculated weight (g) – m_{at}**	**E_a (%)**
AC-25	1497.8	62.4	36.7	39.3	-6.6
AC-13	1253.2	68.3	47.2	49.2	-4.1

It can be seen from Table 2 that the error percentage of SEAM between extracted mass and theoretically true mass is 16.3% when the concentration of the sodium hydroxide solution is 50/50. Because the concentration is high, the sodium hydroxide solution can react chemically with sulphur and asphalt binder at the same time. The result of the SEAM content is not precise.

According to the results of List 2 to List 6 in Table 2, the same concentration was used in the tests but the soaking time was different. The accuracy of the results for 3 hours' and 6 hours' soaking time is better than for 1 hour because sulphur cannot completely react with sodium hydroxide solutions in 1 hour. When 6 hours' soaking time was used in the tests, the accuracy of the results was good. However, the test time is too long, which is disadvantageous for guiding the construction in time, and the solutions can also react with the asphalt binder.

The traditional method using trichloroethylene extraction can be used to determine both SEAM content and asphalt content of SEAM-modified asphalt mix. According to the results of List 3 and List 4 in Table 2, the SEAM extracted mass in the sample is calculated by formula (2) and the mass of extracted asphalt in the sample is calculated by formula (3). The error of the results is acceptable. It can be seen from Table 3 that the weight of asphalt between extracted mass and theoretically true mass is close and the error percentage is stable.

4.3 Calculations of SEAM and asphalt contents

1. To calculate the mass of asphalt mix particles without SEAM (m) in the original sample:

$$m = (m_4 - m_1) + (m_5 - m_2) + (m_6 - m_3) \quad (1)$$

where:

m_4 = mass of metal plate 1 with asphalt mix particles
m_1 = mass of metal plate 1
m_5 = mass of metal plate 2 with asphalt mix particles
m_2 = mass of metal plate 2
m_6 = mass of beaker with asphalt mix particles
m_3 = mass of beaker.

2. To calculate the mass of SEAM in the original sample:

$$m_s = m_{mix} - m \quad (2)$$

where:

m_s = mass of extracted SEAM in sample
m_{mix} = mass of sample.

3. To calculate the mass of asphalt in the original sample:

$$m_a = m_{ex} - m_{sc} \quad (3)$$

where:

m_a = mass of extracted asphalt in sample
m_{ex} = mass of extracted SEAM and asphalt using trichloroethylene extraction
m_{sc} = mass of calculated SEAM in sample according to m_s.

4. To calculate the error percentage of SEAM and asphalt content in the original sample:

$$E_s = \frac{m_s - m_{st}}{m_{st}} \quad (4)$$

$$E_a = \frac{m_a - m_{at}}{m_{at}} \quad (5)$$

where:

E_s = the error percentage of SEAM between extracted mass and theoretically true mass
E_a = the error percentage of asphalt between extracted mass and theoretically true mass
m_{st} = theoretically true mass of SEAM in sample
m_{at} = theoretically true mass of asphalt in sample.

5. CONCLUSIONS

1. The recommended concentration of sodium hydroxide solution is 40/60 based on the test results obtained in this study.
2. The accuracy of extracting SEAM using a 3-hour soaking time is better than for a 1-hour soaking time. A soaking time of 3 hours is suggested for mixing the asphalt mixture thoroughly in NaOH solution.
3. This new extraction method of using NaOH solution can be used to determine the SEAM/asphalt content of a SEAM asphalt mix with acceptable accuracy. This method allows a daily/routine quality check of SEAM asphalt mix production.

6. RECOMMENDATIONS

According to the findings obtained in this study, the following recommendations are made on NaOH solvent extraction testing:

1. Further investigation of the chemical reaction mechanisms of NaOH solutions and asphalt is needed.
2. It is necessary to develop a better testing device to accelerate filtration and improve testing efficiency.

7. REFERENCES

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