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RESEARCH ON THE EFFECTS OF SULFUR ON CHARACTERISTICS OF SULFUR BITUMINOUS BINDER (SBB) AND HOT MIX ASPHALT – SULFUR (HMAS)

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Abstract. Sulfur in SBB has been found to occur in three different forms: (1) chemically bonded, (2) dissolved in bitum, and (3) crystalline sulfur which generally exists in the form of discrete tiny particles dispersed in asphalt depending on sulfur content was added to bitumen, mixing temperature and time of a given mixture. At extraction temperatures (20 and 50°C), sulfur crystallizes and acts as a filling agent. It organizes the structure of the asphalt concrete and increases the thermal stability, hardness, and resistance of asphalt concrete to rutting. Due to the progressive restructuring of the modified sulfur in the mixture, the paving mixture made with modified sulfur takes several days to develop its final strength. This paper investigates the effect of time on the properties of SBB and HMAS. Experimental results show that by the time, the crystallization of sulfur in SBB has effects on SBB properties (penetration, softening temperature) and HMAS properties (Marshall stability, Marshall flow)

Keywords: Bitumen, sulfur, sulfur bituminous binder, hot mix asphalt, hot mix asphalt sulfur

1. INTRODUCTION

Recently, the use of sulfur to improve or replace asphalt in asphalt concrete has been successfully and it was proven both in laboratory experiments as well as in actual construction. The available advantages and low cost of sulfur created the opportunity to reduce the cost of pavement materials up to 21% [1]. On the other hand, since the end of 2003, sulfur as a by-product of petrochemical refining has increased the deterioration of air quality and regulations for air quality control had become more stringent. Products from the oil industry had to comply with low sulphur content thereby increased the attractiveness in using for sulfur in road applications [2]. By the time, Shell Corporation has developed and used sulfur for asphalt concrete which called Thiopave. Thiopave has been used in many countries such as USA, China, India, Qatar, Canada, China [2-7]. Many studies related to sulfur in asphalt concrete have been conducted to investigate its properties, evaluate its performance and ability of highway construction application. Previous researches indicated that sulfur created the increase of Marshall stability of sulfur asphalt concrete mixtures and its stiffness increases with time, which increased the resistance of wheel rutting of flexible pavement structures. The stiffness modulus was increased for a series of mixtures as well as energy savings, i.e. bitumen saving, energy savings during production, and possibly pavement thickness reduction or longer pavement life [2-7].

According to Strickland et al. 2007 [8], the results of mixed and compacted specimens indicated that it usually takes several days for an asphalt mixture made with sulfur to develop its final strength due to the progressive restructuring of the modified sulfur in the mixture. This curing period is not a problem on the road because the sulfur modified mixture generally has a higher initial stiffness than conventional asphalt.

In Vietnam, sulfur is a by-product of the oil and gas production process of Dung Quat oil refinery with an output of 18 tons per day. This output is expected to increase in the future and in other refineries produce. This study investigated some properties of SBB and HMAS according to the sulfur / bitumen ratio using materials in Vietnamese conditions and the variation of these properties of SBB, HMAS in different time to determine the appropriate time for sample maintenance before testing the mechanical properties.

2. MATERIALS AND EXPERIMENTAL METHODOLOGY

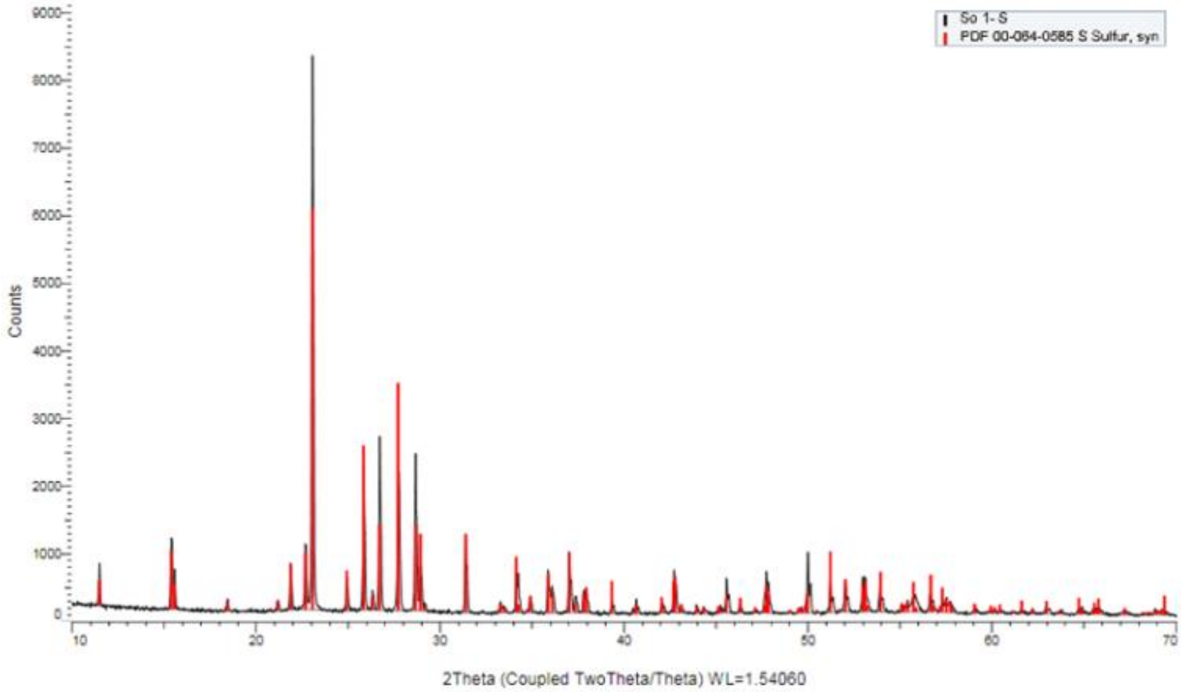
2.1. Materials Preparation

2.1.1. Materials

2.1.1.1. Bitumen

Bitumen produced in Singapore with the Penetration-Graded of 60/70 according to ASTM D946/D946M-15 which is popular used for flexible pavements in Vietnam.

Figure. 1. Sulfur byproduct from Vietnam.



Pattern List - so 1- S

Index	Color	Scan	Compound Name	Formula	System	I/Ic DB	S-Q
1		so 1- S.brml #1	Sulfur, syn	S	Orthorhombic	(1)	100.0 %

Wavelength	Space Group	a
1.54060 Å	Fddd (70)	10.46435 Å

b	c	alpha	beta	gamma
12.86555 Å	24.48598 Å			

Volume	Density
3296.54 Å³	2.067 g/cm³

Figure. 2. XRD result of Sulfur.

Table 1. Physical properties of conventional binder 60/70.

Properties of bitumen	**Unit**	**Results**
Softening Point, ASTM D 36 Standard	^oC	48.3
Ductility, 25 °C, ASTM D 113 Standard	cm	>100
Penetration 25 °C, ASTM D5 Standard	0.1mm	62
Flash point, ASTM D 92 Standard	^oC	276
Loss on Heating of 163^{o}C in 5 hours ASTM D6 Standard	%	0.045
Ratio of penetration at 163^{o}C on penetration at 25^{o}C ASTM D5 Standard	%	60.0
Solubility of bitumen in Trichloroethylene AASHTO T44 Standard	%	99.83
Density	g/cm^3	1.035
Viscosity at 60°C, ASTM D4402	Pa.s	279.642

2.1.1.2. Sulfur

Sulfur in this paper was a byproduct Dung Quat Oil Refinery which is produced at Binh Son Refining and Petrochemical Company Limited (BSR) in Vietnam with an analytical reagent more than 99% purity as shown in Figure 1. In the temperature Sulfur has crystalline with the round form of 2-3 mm diameter Figure 1 and density of 2,063 g/cm3. X-ray diffraction (XRD) spectra was used to specify feature of Sulfur at the room temperature. The XRD result of Sulfur in this paper has shown in the Figure 2.

2.1.1.3. Aggregate

Coarse and fine aggregate were bazan from Phu Man Quarry in Thanh Oai, Ha Noi and were fully crushed. Filler was limestone from Kien Khe in Ha Nam.

HMAS and HMA were using the same 12,5 mm aggregate gradation (Figure 3).

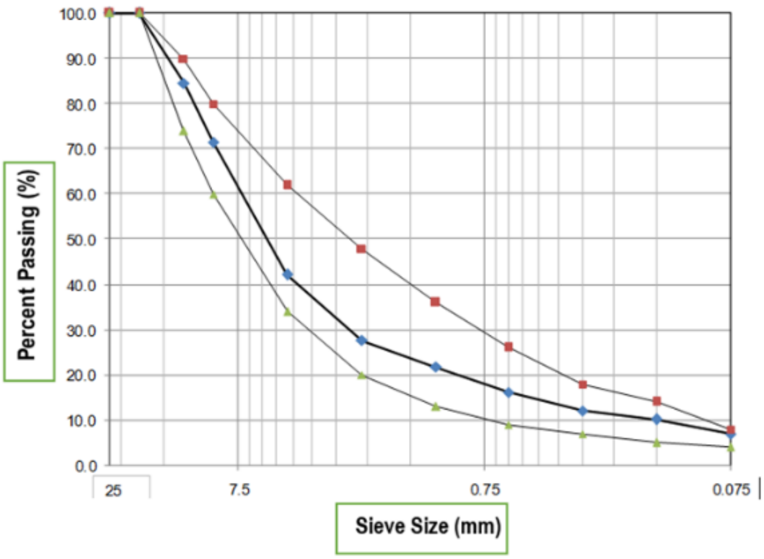


Figure 3. Design Aggregate Gradation Curve.

2.1.2. Sample Preparation

2.1.2.1. Sulfur bitumen binder (SBB)

Bitumen is heated to a temperature of 135^{0}C, sulfur heated at 60°C is slowly added to the bitumen mixture. The stirring speed of 1000 revolutions per minute ensures that the sulfur melts and diffuses evenly in the mixture. The stirring time is 5 minutes, and during mixing, always maintain the temperature of 135^{0}C. Sulfur was added in the bitumen with the ratio of 10/90; 20/80, 30/70 and 30/60 by weight as named SBB1 (S/B ratio = 10/90); SBB2 (S/B ratio = 20/80); SBB3 (S/B ratio = 30/70); SBB4 (S/B ratio = 40/60). Samples after mixing are poured into molds and cured at room temperature. A bitumen sample 60/70 without sulfur was simultaneously prepared in this research as a reference sample. The results presented in this study were the mean values of six measurements.

2.1.2.2. Preparation of hot mix asphalt sulfur (HMAS)

For this study, a mix design was conducted for each of the control and HMAS with mass sulfur percentages 30%, 40% using the Marshall Method according to with VietNamese Standard Test Method TCVN 8820-2011 (Standard Practice for Asphalt Concrete Mix Design Using Marshall Method) and Decision 858/QĐ-BGTVT, 2014.

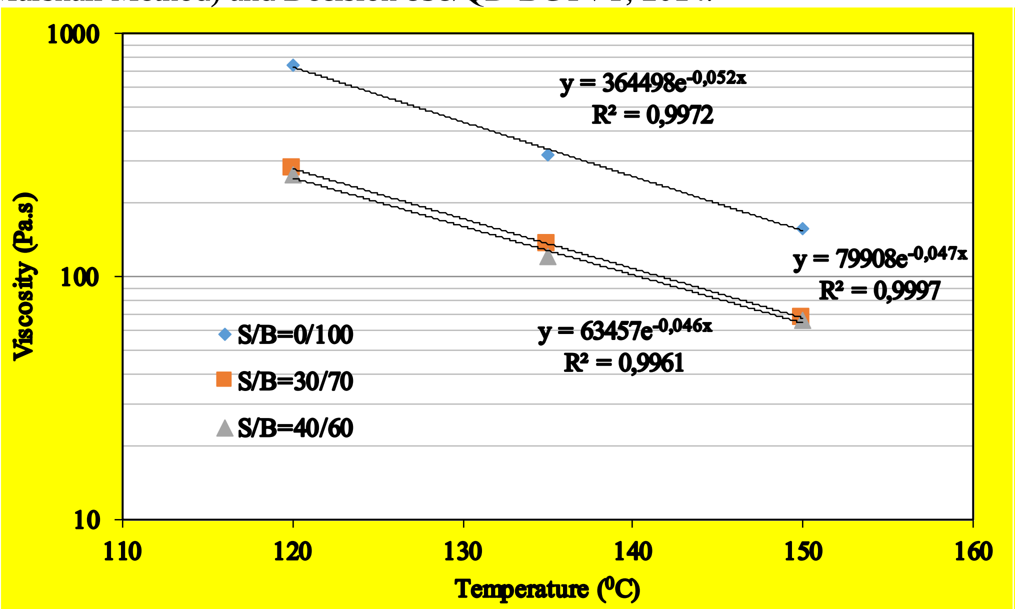


Figure 4. Viscosity - temperature diagram of SBB samples.

For three mixes, the optimum binder content was determined corresponding to 4,5 ÷5 percent air voids (Recommended by Decision 858).

For each HMAS, the content of SBB was determined according to Equation 1.

$$SBB(\%) = \frac{100AR}{100R - P_s(R - G_{bitum})} \quad (1)$$

Where:

A = weight percentage of binder in conventional mix design, %;
R = G_{sulfur}/G_{bitum} (R = 1,99 for this study);
P_s = weight percentage of Sulfur in SBB, %;
G_{bitum} = specific gravity of the bitumen, 1,035g/cm^3;

G_{sulfur} = specific gravity of the bitumen, 2,067g/cm^3.

The mixing and compaction temperatures were determined from viscosity – temperature diagram of bitumen, SBB samples show in Figure 4.

Table 2. Range of mixing and compaction temperature of SBB concrete samples.

No.	Binder Type	Mixing Temp., (°C)	Compaction., (°C)
1	Bitumen 60/70 (S/B = 0/100)	147 ÷151	137 ÷ 141
2	SBB_30/70 (S/B = 30/70)	128 ÷ 133	118 ÷ 122
3	SBB_40/60 (S/B = 40/60)	126 ÷ 131	116 ÷ 120

To mix the HMAS samples in the laboratory, the aggregate is heated at 135°C for 12 hours. Parallel to the aggregate heating process, mixing SBB mixture with S/B ratio was prepared according to 2.1.2.1. When the aggregate meets the temperature, the process of "dry mixing" the aggregate with mineral powder is carried out for 30 seconds in a mixing tank heated to 135oC. The "wet mixing" process with SBB binder is then carried out in 60 seconds, ensuring a homogeneous mixture. All the samples HMAS were short-term aged in the oven at a temperature of 125oC for 2 hours before compaction. The reference HMA mixture was mixed at 155°C and compacted at 145°C.

2.2. Experimental Methodology

2.2.1. Penetration Testing

The penetration test is used to characterizes consistency of bitumen. Higher values of penetration indicate softer consistency. The testing apparatus was setup as in Figure . For the penetration test, a 100 g needle penetrated into a bitumen sample for 5 s at 25 °C, according to the ASTM D 5 Standard. The penetration of bitumen and SBB was determined according to ASTM D5 / D5M - 19a at the age of 0, 7, 14, 30 days.

Figure 5. Penetration test.

2.2.2. Softening Point testing

The softening point was evaluated according to the ASTM D36 Standard. Softening temperature is a specification to assess the quality of bitumen, determine the temperature when bitumen softens and deforms. This is one of the factors in predicting the meltability of bitumen materials when temperatures rise. The softening temperature of bitumen is determined by the Ring and Ball Apparatus as shown in Fig. 5. The softening point of reference bitemen and SBB was investigated at 0, 7, 14 and 30 days.

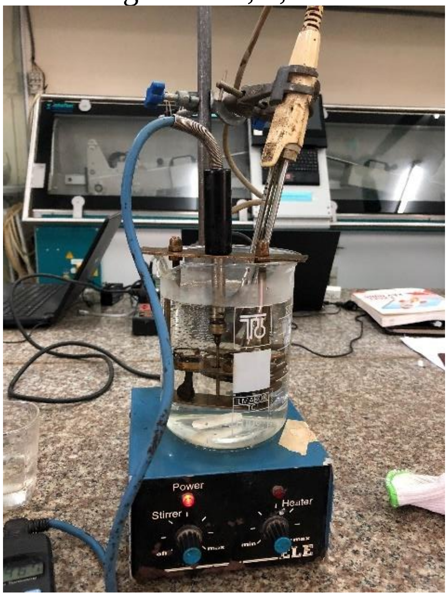


Figure 6. Softening Point testing apparatus.

2.2.3. Marshall testing

The Marshall test according to ASTM D6927 with Daiwa apparatus (Fig.6) determines Marshall stability and Flow.

Figure 7. Marshall testing apparatus.

In study, three asphalt mixtures were evaluated in the laboratory experiment, one HMA used bitumen 60/70 (Penetration-Graded of 60/70) and two HMAS (HMAS_30/70; HMAS_40/60) containing 30% and 40% sulfur (by the weight of the total binder). To investigate the effect of time on Marshall properties, HMAS samples were prepared and curing at room temperature. After periods of 1,7,14,21 and 28 days, samples were tested to determine Marshall stability and Flow.

3. Results and discussion

3.1. Penetration of SBB

The results on average depth of 06 samples per penetration test of SBB binder samples are shown in Table 3 and Fig. 8. Experimental results were evaluated for precision according to standards ASTM D5 /D5M - 19a.

Table 3. Results of tests on penetration depth of SBB samples by date of age.

No.	Penetration afer 0 day				Penetration afer 7 day				Penetration afer 14 day				Penetration afer 30 day			
	SBB 10/90	SBB 20/80	SBB 30/70	SBB 40/60	SBB 10/90	SBB 20/80	SBB 30/70	SBB 40/60	SBB 10/90	SBB 20/80	SBB 30/70	SBB 40/60	SBB 10/90	SBB 20/80	SBB 30/70	SBB 40/60
1	95,6	105,4	108,9	108,5	63,5	51,4	53,3	50,2	51,3	46,6	40,3	42,7	53,5	41,2	35,5	34,6
2	100,3	105,1	102,2	109,6	66,3	54,6	50,2	53,3	53,1	43,7	42,8	39,8	56,1	39,5	38,5	36,4
3	109,6	105	105,4	110,6	63,1	52,4	51,3	50,2	51,5	44,7	43	41,3	53,8	43,2	36	37,7
4	101,7	106,3	105,7	104,3	66,2	55,7	52,5	53,3	53,6	42,2	42	38,9	55,6	40,6	38,8	34,1
5	101,4	95,6	100,7	101,5	66,5	52,1	50,7	50,2	52,7	42,6	44,1	38,5	55,7	41,3	36,5	35,2
6	103,4	94,8	101,8	100,7	63,1	55,2	53,6	52,5	50,8	41,5	41,5	41,5	52,5	40,7	36,9	35,8
Ave.	102,0	102,0	104,1	105,9	64,8	53,6	51,9	51,6	52,2	43,6	42,3	40,5	54,5	41,1	37,0	35,6
Range R= max - min	14,0	11,5	8,2	9,9	3,4	4,3	3,4	3,1	2,8	5,1	3,8	4,2	3,6	3,7	3,3	3,6
Standard deviation, 1s (pen)	2,06	2,061	2,124	2,176	0,944	0,8	0,8	0,8	0,8	0,8	0,8	0,8	0,8	0,8	0,8	0,8
Standard deviation value: 4x1s	8,24	8,244	8,494	8,704	3,774	3,2	3,2	3,2	3,2	3,2	3,2	3,2	3,2	3,2	3,2	3,2
Evaluate Precision	Fail	Fail	Pass	Fail	Pass	Fail	Fail	Pass	Pass	Fail	Fail	Fail	Fail	Fail	Fail	Fail

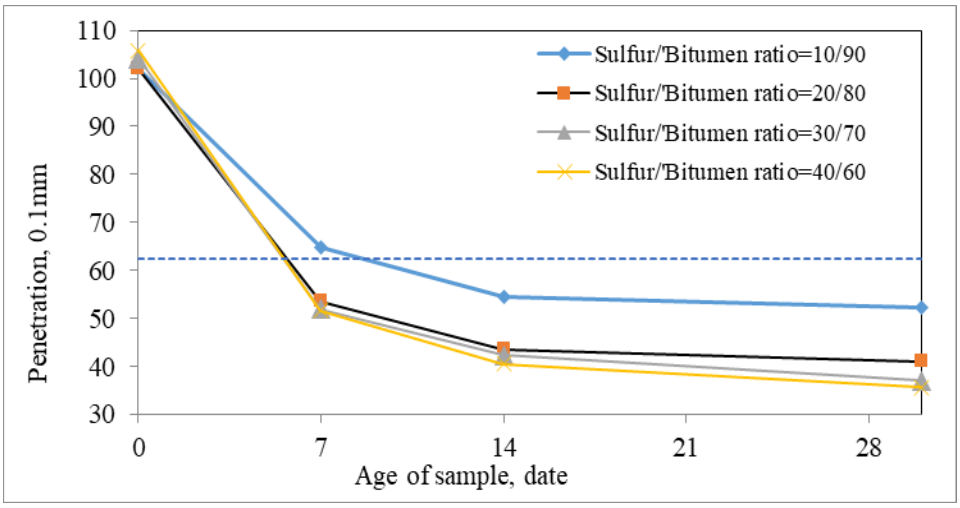


Figure 8. Variation in penetration of SBB samples with age.

The test results show that at 0 day old, the penetration of SBB samples with different S/B ratios increase substantially (from 1.64 to 1.70 times) compared to the original 60/70 bitum sample. However, after periods of 1,7,14,21 and 28 days, the penetration of SBB samples decreased significantly, especially SBB 30/70 and SBB 40/60 sample. The penetration of

SBB10/90, SBB20/80 decreased from 1,5 to 1,9 times after 7, 14 day. The penetration of SBB 30/70, SBB40/60 decreased from 2 to 3 times after 7, 14, 30 day and decreased from 1,1 to 1,7 compared to the original 60/70 bitum sample. Results of the penetration test show that the penetration of SBB samples were lower than the reference sample. Results of evaluating the precision of the penetration test show that not all results are pass.This might be because when the bitumen and sulfur was combined, a part of the sulphur is either dissolved or chemically combined with the bitumen. The un-dissolved quantity of sulfur in the bitumen remains predominantly as free sulfur and it crystaled during cooling, settled, unevenly distributed in the sample, this created an inhomogeneous binder. In that case, the measured penetration value normally was not reproducible and depended on the existence of sulfur crystals at or near the penetration needle.

The analysis of ANOVA variance with 95% confidence level, Tukey's family error rate 5% shows a significant effect of time to the penetration value of SEA samples at 25°C. For samples SBB 10/90 and SBB 20/80, sharp decrease in penetration occurred at times of 0, 7 and 14 days. After 30 days, penetration of SBB 10/90 and SBB 20/80 samples did not differ statistically compared to 14 days of age. For the SBB 30/70 and SBB 40/60 samples, however, the penetration still decreased significantly after 14 days.

The rule of varying the penetration of SBB samples with different sulfur content over the days is explained by the crystallization of sulfur by the time [9, 10, 11]. The penetration of SBB decreased show that the stiffness of the SBB mixture was seen to increase with time. These studies to explain the increase stiffness of asphalt pavements containing SBB.

3.2. Softening point of SBB

Experimental results on average softening temperatures of 06 samples of SBB binder are shown in Table 4 and Fig 9. Experimental results were evaluated for precision according to standards ASTM D36.

The results show that the softening temperature of SBB samples with different S/B ratios at 0 day of age decreases in comparison with the original 60/70 asphalt sample. But the softening temperature tends to increase with sulfur ratio and age of sample. The softening temperature increased most significantly after 14 days.

Table 4. The softening temperatures of SBB samples by date of age.

No.	Soften point afer 0 day				Soften point afer 7 day				Soften point afer 14 day				Soften point afer 30 day			
	SBB 10/90	SBB 20/80	SBB 30/70	SBB 40/60	SBB 10/90	SBB 20/80	SBB 30/70	SBB 40/60	SBB 10/90	SBB 20/80	SBB 30/70	SBB 40/60	SBB 10/90	SBB 20/80	SBB 30/70	SBB 40/60
1	47	46	45	44	48,7	51	51,8	53,5	48,2	52,8	53,5	55,1	48,6	52,7	53,8	56
2	47,5	46,6	46,5	44,5	48,5	51,3	52	53,8	48,6	53,1	53,6	55,6	48,9	53,2	54,1	56,3
3	47	46,5	45	45,5	47,3	50,5	50,3	53,3	48,7	52,3	53,6	54,7	47,9	53,7	54	56,2
4	47,7	46	45,3	44	49,2	50,8	50,8	53,7	49,1	52,8	53,6	54,9	48,5	53,8	53,9	56,5
5	47,2	46,2	45	44	48,5	50,2	50,1	53	47,6	52,1	53	54,6	48,7	52,8	53,7	55,8
6	47	46,5	45,5	44,6	48,2	51,5	51,3	54	48,5	53,6	54	55,7	48,9	53,7	54,1	56,7
Ave.	47,2	46,3	45,4	44,4	48,4	50,9	51,1	53,6	48,5	52,8	53,6	55,1	48,6	53,3	53,9	56,3
Range R= max - min	0,7	0,6	1,5	1,5	1,9	1,3	1,9	1	1,5	1,5	1	1,1	1	1,1	0,4	0,9
Standard deviation , 1s (°C)	1,2	1,2	1,2	1,2	1,2	1,2	1,2	1,2	1,2	1,2	1,2	1,2	1,2	1,2	1,2	1,2
Standard deviation value: 4x1s	4,8	4,8	4,8	4,8	4,8	4,8	4,8	4,8	4,8	4,8	4,8	4,8	4,8	4,8	4,8	4,8
Evaluate Precision	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass

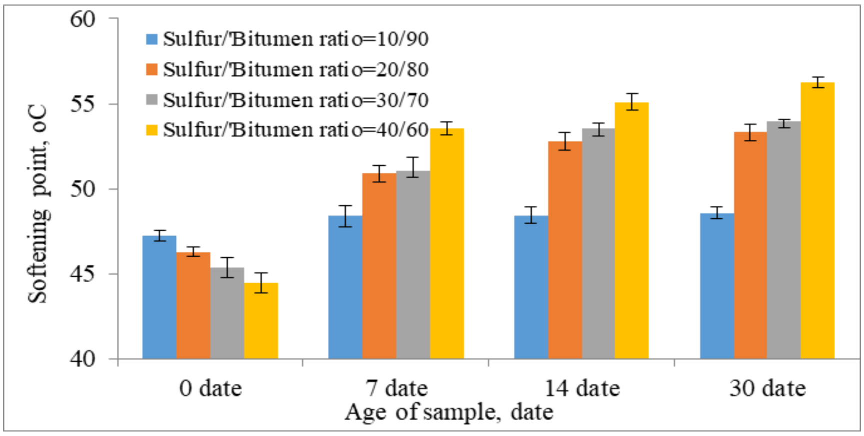


Figure 9. The softening temperatures of SBB samples by date of age.

The analysis of ANOVA variance on softening temperatures of SBB samples with the confidence level 95% and Tukey's family error rate 5% shows a significant influence of time on the softening temperature values of SBB samples. For SBB samples 10/90, after 7 days , there was insignificant increase in softening temperature and there was no change in softening temperature at 7, 14, 30 days of age. The softening temperature of the SBB sample 10/90 after 30 days is equivalent to the 60/70 bitumen sample. For SBB samples of 20/80 and 30/70 softening temperatures increased after 7, 14 days of curing, higher than bitumen 60/70. The analysis also shows that there was no statistical difference between 30-day and 14-day samples in the softening temperature of SBB 20/80 and SBB 30/70 samples. In particular, for 40/60 SBB samples, the softening temperature increased substantially after 7, 14 and 30 days. After 14 days, the SBB 20/80, 30/70, and 40/60 samples have the softening temperatures increased 1.11-1.16 time (4.5^{o}C-6.8^o C) compared to the 60/70 bitumen sample.

The tendency to increase the softening temperature of SBB samples signifies the ability to increase the Marshall stability and to reduce the rutting in asphalt samples using SBB. Specifically, as increasing the softening temperature of bitumen by 5^{o}C the Marshall stability would increase by more than 1.3kN, and the incremental rut depth would decrease by half [12].

3.2.3. Marshall Testing

Experimental results to determine the optimal binder content for the HMA and HMAS are shown in Table 5.

Table 5. Optimal binder content in asphalt concrete mixtures.

Asphalt Concrete	**Binder Type**	**Optimal binder content, %**		
			Original bitumen	**Sulfur**
HMA	Bitumen 60/70	4,5	4,5	0
HMAS_30/70	SBB_30/70	5,62	3,94	1,68
HMAS_40/60	SBB_40/60	6,08	3,65	2,43

These results show that optimum binder content increased with increasing sulfur content in the mixture. The test results of the Marshall properties shown in Fig.10; Fig. 11.

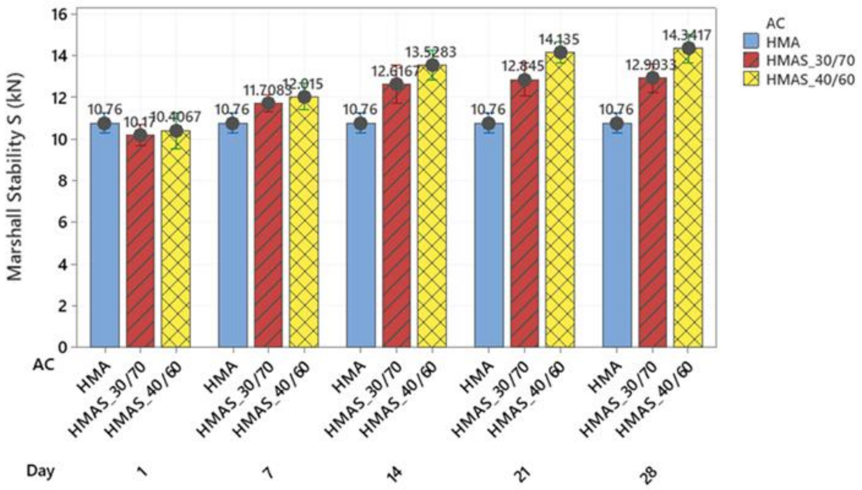


Figure 10. Marshall Stability of HMAS by date of age.

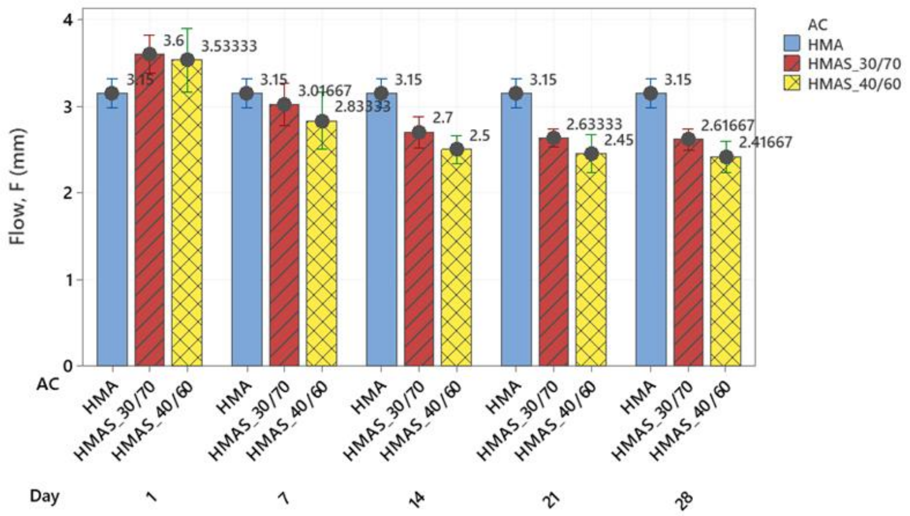


Figure 11. Marshall Flow of HMAS by date of age.

Marshall stability of HMAS_30/70 and HMAS_40/60 samples after 28 days was 20% to 33% higher than HMA. The Marshall flow of HMAS after 28 days was 17% to 23% lower than that of HMA.

Investigation of changes in the Marshall properties of HMAS by time showed that Marshall stability of HMAS samples increased significantly during the first 14 days after preparation and increased slightly from 14 to 28 days. The law of reduction in Marshall Flow over time is similar: a rapid decrease during the first 14 days and gradually stabilize from 14 to 28 days.

Analysis of ANOVA variance and post-hoc analysis (Tukey's HSD method) comparing the differences in Marshall stability values, Marshall Flow of HMAS samples according to time showed Marshall stability of sample HMAS increased with time and stabilized after 14 days. After 14 days, the Marshall stability value continued to have an increase, but was not statistically significant. Marshall Flow of sample HMAS decreased gradually by the time and stabilized after 14 days. By the time, the crystallization of sulfur in the structure of HMA leading to increased stiffness and reduced deformation under the effect of external forces.

4. CONCLUSION

- Sulfur is a byproduct of petrochemical plants in Vietnam can be used in combination with bitumen to produce asphalt concrete.

- HMAS has a lower mixing and compaction temperature than conventional HMA from 20^oC to 25^oC (mix at 135^oC and compact at 125^oC).

- By the time, sulfur crystals formed in the HMAS structure significantly improved Marshall stability. Marshall stability of HMAS is higher than conventional HMA from 20% to 33% (depending on the amount of sulfur used), the Marshall Flow of HMAS is lower than conventional HMA from 17% to 23%). Marshall parameters did not change significantly at 21, 28 days of age, showing that the crystallization of sulfur took place rapidly at the first 14 days after compaction and stabilization.

- Experimental program indicated that SBB concrete samples showed the significant performance of Marshall stability, which recommended that the age for evaluation of Marshall stability of SBB concrete mixtures should be after 14 days.

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