

Storage stability of SBS modified bitumen based on mixed-level orthogonal array design

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Abstract: The styrene-butadiene-styrene (SBS) modified bitumens with different contents of SBS modifiers are stored in different conditions to study the storage stability of SBS modified bitumen. Mixed-level orthogonal array design (OAD) is used and factors such as SBS modifier content, storage time, storage temperature and container size are chosen in a mixed-level OAD with an $OA_{16} (3^1 \times 4^4)$ matrix. Parameters like the separation softening point difference (the separation difference of the ring and ball softening point of the top and bottom samples) and the average softening point (the arithmetic mean of the softening points of the top and bottom samples) are proposed to evaluate the separation and the ageing of modified bitumen during storage in this experiment, respectively. The results reveal that the separation and the ageing during storage exhibit a complicated variation for storage temperature and time. The separation softening point difference decreases with the storage temperature rising from 20 to 120 °C and increases with the temperature exceeding 120 °C, and the average softening point drops with the storage time being prolonged. Different storage conditions have various effects on the storage stability of SBS modified bitumen.

Key words: storage stability; mixed-level orthogonal array design; separation softening point difference; average softening point

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Polymer modified bitumen has been widely used in the construction of flexible pavements. Pavement distresses, such as rutting at high temperatures and cracking in the low temperature region, can be reduced by using modified binders^[1]. However, the formation of a functional modified binder system is based on the dissolution and/or fine dispersion of polymer in bitumen and on the compatibility of the polymer/bitumen system^[2], which makes it possible that polymer modified bitumen tends towards separation and ageing. Polymer-modified asphalt must have superior stability during storage and transportation in order to guarantee homogeneous mechan-

ical properties during application and road service^[3]. Therefore, research on the storage stability of polymer modified bitumen has become a hot issue.

Some studies have already been carried out by researchers to explore the separation mechanism of SBS modified bitumen. Bitumen is regarded as a colloidal system consisting of a suspension of high molecular weight asphaltene micelles dispersed in a lower molecular weight oily medium (maltenes)^[4]. The introduction of polymers with a high molecular weight will disturb the dynamic equilibrium and reduce the homogeneity of the bitumen system^[5]. There is a polymeric network structure in the base bitumen^[6] when the SBS polymer is dissolved/dispersed in the bitumen^[7], so the bitumen system is unstable. Due to the influence of thermodynamic and gravitational fields, phase separation will take place in SBS-modified asphalts during storage at elevated temperatures according to Stock's law^[8].

Regarding the variation of properties of modified bitumen, there are few studies that have been published on this aspect. During hot storage, the performance of SBS modified asphalt deteriorates, although phase separation does not occur^[9]. Even though the results meet the requirement that the difference of the ring and ball softening point of the top and bottom samples is less than 2.5 °C according to China's current specifications, i. e., "Polymers Modified Asphalt Separation Test", the storage stability in the field cannot be guaranteed^[10]. On account of the defects in the present standard "Polymers Modified asphalt Separation Test", Ji et al.^[11] developed the modified laboratory asphalt stability test (MLAST) to simulate the storage of modified asphalt, and the ratio of separation was proposed to evaluate the storage stability of SBS modified asphalt. However, the instrument was complicated and difficult to promote. Huang et al.^[12-13] studied the interactions among SBS, asphalt and maltenes, and found that the swelling and dispersion of SBS in asphalt is a dynamic balance procedure. The softening point of the unstable SBS modified asphalt may decrease even during room temperature storage and the separation phenomenon may be more serious.

In addition, few researchers have considered the ageing of modified bitumen during long-term storage. Most of them have concentrated on the storage stability of modified bitumen during hot storage for a 48 h period. The soften-

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ing point of SBS modified bitumen is found to decline in the process of hot storage^[14]. Chen et al.^[15] discussed the interaction between SBS polymer and base bitumen during production, construction and operation. However, the interaction during storage was not considered. The SBS polymer modified bitumen aged by means of the thin film oven test (TFOT) and the rolling thin film oven test (RTFOT) is studied and it is observed that ageing results in polymer degradation and bitumen oxidation^[16]. Ageing of the SBS PMBs tends to result in a reduction of the molecular size of the SBS copolymer with a decrease in the elastic response of the modified road bitumen^[17]. However, the modified bitumen stored in the sealed container is under non-oxidative conditions, so the ageing of modified bitumen during storage is non-oxidative.

Generally, storage stability means the stability of modified bitumen during the hot storage. In the present study, storage stability refers to the ability to maintain the original performance of the modified bitumen under a certain storage condition, and it is represented by separation and ageing. The separation softening point difference and the average softening point are used to evaluate separation and ageing, respectively. In this research, apart from identifying the significant factors which influence the long-term storage stability of SBS modified bitumen stored at different temperatures by a mixed-level orthogonal array design (OAD), the variation regularity of storage stability is examined and the rational storage temperature and time are given to provide a reference for guiding industrial storage of SBS modified bitumens.

1 Materials

Bitumen AH-70 obtained from China is used for the experiment. The physical properties are illustrated in Tab. 1. The SBS polymer used in this research is KTR101, supplied by Korea Kumho Petrochemical Company. KTR101 is a linear SBS polymer containing 30% styrene. The stabilizer is purchased from Shanghai Doctor Asphalt Technology Co., Ltd. The stabilizer content is 0.45% by weight of base bitumen.

Tab. 1 Physical properties of bitumen AH-70

Property	Value
Penetration (25 °C, 100 g, 5 s)/0.1 mm	63.4
Softening point/°C	55.5
Viscosity (135 °C)/(mPa · s)	485.1

2 Experimental Program

The technological procedure for producing the modified bitumen is as follows: The base bitumen is heated to fluid condition and poured into a metal container. When the temperature is above 160 °C, a prescribed amount of polymer is added to the base bitumen and swelling is done with a high shear mixer for 30 min until the modifier is swollen completely. After swelling, the modified bitu-

men is mixed with the high shear mixer for 30 min. The maturity for the modified bitumen is then performed using a high speed mechanical blender. Upon reaching 170 °C, the stabilizer is added to the modified bitumen, and the maturity lasts for 2 h. The temperature for the preparation is controlled at 160 to 180 °C. After completion, the modified bitumen is removed from the metal container and housed in the containers for storage.

2.1 Separation test

The separation test is a polymer modified asphalt separation test (PMAST) according to Chinese code T0661—2000^[18]. The test consists of keeping the polymer modified asphalt specimen in a test tube at (163 ± 5) °C for a period of (48 ± 1) h and leaving the tube in the refrigerator for a minimum of 4 h and then taking away samples from the top and from the bottom of that test tube. The separation softening point difference of the modified asphalt during storage is evaluated by comparing the ring and ball softening point of the top and bottom portions. If the difference is lower than 2.5 °C, the material is classified as a stable polymer modified asphalt. This is a typical test used in China asphalt industries to determine the storage stability of polymer modified asphalt. However, in order to investigate the storage stability of SBS modified bitumen for a certain period of storage time, it is unnecessary to maintain the specimen in the (163 ± 5) °C oven for (48 ± 1) h. In this case, the separation test in the present study is carried out by improving the polymer modified asphalt separation test. All the tests are carried out in accordance with JTJ 052—2000^[18].

According to the PMAST, the test procedure is as follows: The tube with a 25 mm diameter by a 200 mm length is placed down in the rack. The sample is heated until it is sufficiently fluid to pour. After thoroughly stirring, the sample is poured into the vertically held tube. The rack containing the sealed tubes is placed in a (163 ± 5) °C oven. The tubes are allowed to stand undisturbed for a period of (48 ± 1) h. At the end of the heating period, the tubes are placed in the refrigerator for a minimum of 4 h. The top and bottom samples should be tested at the same time in the softening point test.

The specific test in the present study is slightly different from the current specifications. The tubes of different sizes, and the ovens with different temperatures and different storage times are used.

2.2 Average softening point test

The arithmetic mean of the ring and ball softening point of the top and bottom samples is defined as the average softening point. The average softening point is calculated by

$$S_{ASP} = \frac{S_T + S_B}{2} \tag{1}$$

where S_{ASP} is the average softening point; S_T is the ring and ball softening point of the top sample; S_B is the ring and ball softening point of the bottom sample.

2.3 Matrix construction method

In the present study, four factors selected in this study are as follows: 1) SBS modifier content (3.0%, 3.5%, 4.0% and 4.5% by weight of base bitumen); 2) Storage time (1, 3, 8 and 15 d); 3) Storage temperature (20, 80, 120 and 160 °C); 4) Container size (16 mm × 150 mm, 25 mm × 200 mm, 60 mm × 75 mm. 16 mm × 150 mm means a 16 mm diameter by a 150 mm length glass tube for container size).

In order to construct a mixed-level $OA_{16}(3^1 \times 4^4)$ matrix, an original four-level $OA_{16}(4^5)$ matrix and its assignment table must be constructed initially^[19]. $3^1 \times 4^4$ indicates that one factor (D) has only three levels whereas the others have four levels. A mixed-level OAD table is presented to arrange the testing plan. 16 kinds of SBS modified bitumens with three parallel samples from each kind are prepared in the laboratory using the same base

bitumen. The arranged mixed-level OAD table is shown in Tab. 2 and one column in the three-level design provides only two degrees of freedom whereas the four-level column has three degrees of freedom. The letters A , B , C , D and E are used to represent modifier content, storage time, storage temperature, container size and error line, respectively.

Tab.2 Values of factors and levels for mixed-level OAD

Levels	SBS modifier content/% (A)	Storage time/d (B)	Storage temperature/°C (C)	Container size/(mm × mm) (D)
1	3.0	1	20	16 × 150
2	3.5	3	80	25 × 200
3	4.0	8	120	60 × 75
4	4.5	15	160	(25 × 200)

3 Results and Discussion

3.1 Mixed-level OAD test results

The mixed-level OAD test program and results are shown in Tab. 3.

Tab.3 Mixed-level OAD test results

No.	Factors					Results			
	A	B	C	D	E	Separation softening point difference/°C	Softening point of the top sample/°C	Softening point of the bottom sample/°C	Average softening point/°C
1	1	1	1	1	1	2.6	67.9	65.3	66.6
2	1	2	2	2	2	1.3	70.4	69.1	69.8
3	1	3	3	3	3	0.8	76.7	75.9	76.3
4	1	4	4	4(2)	4	5.4	80.0	74.6	77.3
5	2	1	2	3	4	1.6	77.5	75.9	76.7
6	2	2	1	4(2)	3	1.9	73.0	71.1	72.1
7	2	3	4	1	2	4.2	81.8	78.6	80.2
8	2	4	3	2	1	2.7	73.3	70.6	72.0
9	3	1	3	4(2)	2	1.1	87.4	86.3	86.9
10	3	2	4	3	1	3.5	86.1	82.6	84.4
11	3	3	1	2	4	5.7	71.0	65.3	68.2
12	3	4	2	1	3	3.2	73.1	69.9	71.5
13	4	1	4	2	3	2.3	88.6	86.3	89.5
14	4	2	3	1	4	1.3	85.8	84.5	85.2
15	4	3	2	4(2)	1	2.4	74.6	72.2	73.4
16	4	4	1	3	2	7.6	71.4	63.8	67.6

3.2 Analysis of variance

The sum of squares of deviations for factor A , S_A , is calculated, and x_{ij} represents the j -th observation taken under factor level i ^[20].

$$\sum_{i=1}^{n_a} \sum_{j=1}^a x_{ij} = \sum_{k=1}^n x_k \tag{2}$$

$$S_A = \frac{1}{a} \sum_{i=1}^{n_a} \left(\sum_{j=1}^a x_{ij} \right)^2 - \frac{1}{n} \left(\sum_{i=1}^{n_a} \sum_{j=1}^a x_{ij} \right)^2 = \frac{1}{a} \sum_{i=1}^{n_a} K_i^2 - \frac{1}{n} \left(\sum_{k=1}^n x_k \right)^2 \tag{3}$$

The degree of freedom for factor A is $n_a - 1$, where n_a is the level for factor A . The total degree of freedom is $n - 1$, where n is the total number of observations. The error degree of freedom is $n - 1$ minus the degree of freedom for all the factors. In these experiments, a blank column is set in the mixed-level OAD table for error estimation. The sum of squares of deviations (SS), the degree of freedom(DF), and the mean squared deviation (MS) of separation and average softening points are determined and summarized in Tab. 4. The F value of a factor is the ratio of the MS value of the factor to that of error line. Through comparing the obtained F value with the theoret-

Tab. 4 Analysis of variance results

Indicator	Factor	SS	DF	MS	F	Critical value of F	Significance
Separation softening point difference	A	2. 735	3	0. 911 7	0. 592 9	4. 19	
	B	21. 035	3	7. 011 7	4. 560 4	4. 19	*
	C	23. 655	3	7. 885 0	5. 128 5	4. 19	*
	D	0. 855	2	0. 427 5	0. 278 0	4. 32	
	E	6. 150	4	1. 537 5			
Average softening point	A	97. 542	3	32. 514 0	3. 467 5	4. 19	
	B	145. 047	3	48. 349 0	5. 156 3	4. 19	*
	C	512. 002	3	170. 667 3	18. 201 2	4. 19	*
	D	0. 312	2	0. 155 9	0. 016 6	4. 32	
	E	37. 507	4	9. 376 7			

Note: $F_{0.10}(3,4) = 4.19, F_{0.10}(2,4) = 4.32$; “*” stands for significance under the level of 0.10.

ical one of specific level and DF, the significance level can be determined for each factor.

The analyses of variances for separation softening point differences and average softening points are carried out. For factor B (storage time) and factor C (storage temperature), the F values are greater than the critical values of F.

Therefore, it can be concluded that the storage time and temperature have shown significant influences on the separation softening point difference and the average softening point.

3.3 Analysis of range

The analysis of range is done by calculating the difference between the maximum and the minimum of the averages for each factor^[20]. The results are illustrated in Tab. 5.

Tab. 5 Analysis of range results

Indicator	Factor	A	B	C	D
Separation softening point difference	K_1	290. 0	319. 7	274. 5	303. 5
	K_2	301. 0	311. 5	291. 4	609. 2
	K_3	311. 0	298. 1	320. 4	305. 0
	K_4	315. 7	288. 4	331. 4	
	\bar{K}_1	72. 500	79. 925	68. 625	75. 875
	\bar{K}_2	75. 250	77. 875	72. 850	76. 150
	\bar{K}_3	77. 750	74. 525	80. 100	76. 250
	\bar{K}_4	78. 925	72. 100	82. 850	
	R	6. 425	7. 825	14. 225	0. 375
	K_1	10. 1	7. 6	17. 8	11. 3
	K_2	10. 4	8. 0	8. 5	22. 8
	K_3	13. 5	13. 1	5. 9	13. 5
Average softening point	K_4	13. 6	18. 9	15. 4	
	\bar{K}_1	2. 525	1. 900	4. 450	2. 825
	\bar{K}_2	2. 600	2. 000	2. 125	2. 850
	\bar{K}_3	3. 375	3. 275	1. 475	3. 375
	\bar{K}_4	3. 400	4. 725	3. 850	
	R	0. 875	2. 825	2. 975	0. 550

For the separation softening point difference, the range of storage temperature is close to that of storage time, and is far more than that of modifier content and container size. The order of factors is as follows; storage tempera-

ture = storage time>>modifier content > storage size.

For the average softening point, the range of the storage temperature is far greater than that of the storage time and the modifier content. The order of factors is as follows; storage temperature>>storage time = modifier content > storage size.

3.4 Separation

The analysis of variance results in Tab. 4 shows that the storage temperature and time are confirmed as the significant factors. The relationship curves between the separation softening point difference and the significant factors are plotted in Fig. 1.

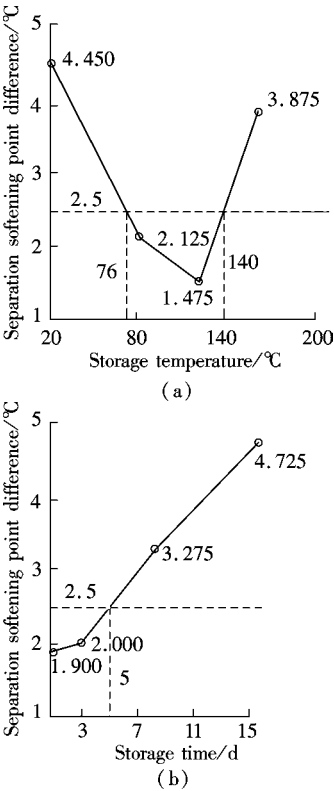


Fig. 1 Separation softening point difference of samples vs. storage temperature and time. (a) Storage temperature; (b) Storage time

From Fig. 1 (a), the separation softening point difference drops gradually with the increase in storage temperature and rises abruptly from around 120 °C. The lowest value of the separation softening point difference is observed at 120 °C. According to the specifications^[21], the modified bitumen is considered stable when the separation softening point difference is below 2.5 °C. The storage stability of the modified bitumen stored at 76 to 140 °C can be classified as good storage stability as shown in the dashed line of Fig. 1 (a) according to the current specifications^[21]. The reasons that this phenomenon appears are ascribed to the fact that the SBS modified bitumen possesses the characteristic of swelling at a high temperature and precipitation at a low temperature^[12]. SBS polymers absorb many maltenes from the bitumen at a high temperature during preparation and swelling^[22]. When the storage temperature drops to 20 °C, the SBS polymers are unable to absorb maltenes and will precipitate some maltenes. With the rising temperature, precipitation reduces and swelling takes place, and separation decreases. The separation softening point difference reaches the minimum at about 120 °C. On the other hand, the maltenes fraction of bitumen absorb the elastic fraction of SBS, so there is a competition between SBS and asphaltenes for the absorption of maltenes. As SBS polymers absorb maltenes sufficiently upon exceeding 120 °C, the polymers which consist of SBS polymers and maltenes are usually accumulated and float at a high storage temperature, and asphaltenes precipitate and settle to the bottom. As a result, the softening point of the top section of modified asphalts becomes higher than that of the bottom section.

From Fig. 1 (b), it can be observed that the separation softening point difference increases with the increasing storage time. The modified bitumen for a period of 15 d has reached a maximum value in separation softening point difference compared with other bitumens, followed by 8, 3 and 1 d of the storage time. The results in Fig. 1 (b) indicate that the limit is at 3 d of storage time as the average softening point plot shows a subsequent “sudden rise”. The average softening point at 8 d of the storage time increases by 63.8% compared with that at 3 d. When the storage time is more than 5 d, the separation cannot meet the specifications. It can be interpreted that the droplets of the SBS melt dispersed in bitumen are usually accumulated and float on the top of the bitumen, and the softening point of the top section of modified bitumens becomes higher than that of the bottom section. Consequently, phase separation between SBS polymer and base bitumen takes place. Therefore, the longer the storage time, the larger the separation softening point difference will be and hence, sharper separation.

3.5 Ageing

The analysis of variance in Tab. 4 presents the factors

which influence the average softening point, and indicates that storage temperature and storage time are the significant factors. Fig. 2 illustrates the average softening point of samples vs. the storage temperature and time.

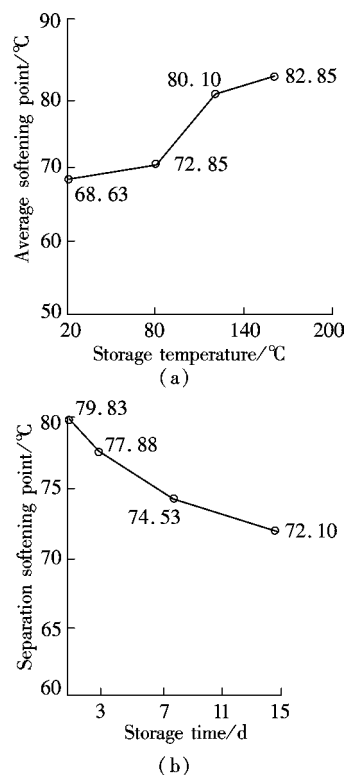


Fig. 2 Average softening point of samples vs. storage temperature and time. (a) Storage temperature; (b) Storage time

It can be observed from Fig. 2 (a) that the average softening point increases with the increase in storage temperature. The highest average softening point is registered at the temperature of 160 °C, while the lowest value is observed at 20 °C. The rising temperature for modified bitumens during storage has increased the average softening point by 20.7%. Therefore, we can see that as the storage temperature of modified bitumen increases, the average softening point increases. The increase in the average softening point is due to the continuation of the ageing of modified bitumens on the bottom and SBS accumulation and floating on the top of the modified bitumens during storage.

The results in Fig. 2 (b) illustrate a gradual decrease in the average softening point with the storage time. The average softening point at 15 d of the storage time has dropped by 9.9% compared to that at 1 d. Therefore, we can also conclude that storage time has modest influences on the ageing of modified bitumens during storage since there is no significant reduction in the average softening point. This conclusion is similar to the study by Huang et al.^[12–13] who have studied the morphological structure of modified bitumens before and after tube storage at 163 °C, and found that during the long-term storage at room and high temperatures, the softening point of modified bitumens drops.

4 Conclusions

1) The analysis of variance and the analysis of range have obtained very similar results.

2) Based on the analysis of variance and the analysis of range, the significant factors of the separation softening point difference and the average softening point are obtained. The order of factors for the separation softening point difference is as follows: storage temperature = storage time \gg modifier content > storage size. The order of factors for the average softening point is as follows: storage temperature \gg storage time = modifier content > storage size.

3) As the storage temperature rises from 20 to 120 °C, the separation softening point difference decreases gradually, and then increases with the temperature exceeding 120 °C. When the modified bitumens are stored at 76 to 140 °C, the separation softening point difference is less than 2.5 °C. The separation softening point difference increases with the increasing storage time. When the storage time is more than 5 d, the separation softening point difference will not meet the current specifications.

4) The average softening point increases with the increase in the storage temperature. With the increasing storage time, the average softening point decreases.

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基于混合水平正交设计的 SBS 改性沥青储存稳定性研究

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摘要: 为了研究 SBS 改性沥青的储存稳定性, 将不同 SBS 改性剂掺量的 SBS 改性沥青储存于不同条件下. 采用混合水平正交设计, 选取 SBS 改性剂掺量、储存时间、储存温度和容器尺寸作为混合水平正交设计 $OA_{16}(3^1 \times 4^4)$ 中的因素. 提出用离析软化点差(试样的顶部和底部环与球法软化点差)和平均软化点(试样的顶部和底部环与球法软化点的算术平均值)分别评价在储存期间改性沥青的离析和老化. 结果显示, 改性沥青的离析和老化随储存温度和储存时间呈现复杂的变化规律. 当储存温度从 20 ℃ 上升到 120 ℃ 时, 离析软化点差变小, 当超过 120 ℃ 时增大, 而平均软化点随储存时间的延长而降低. 不同储存条件对 SBS 改性沥青的储存稳定性的影响各有差异.

关键词: 储存稳定性; 混合水平正交设计; 离析软化点差; 平均软化点

中图分类号: U414