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Research on rapid separation technology of asphalt components based on large column chromatography

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Abstract. The four component determination method is widely used among the many methods of measuring the chemical composition of asphalt. However, the specification of asphalt four-component test data accuracy is low, the separation time is long, and the amount of the asphalt component is small so that it is not enough to be used for other experiments. Based on this, this paper presents a rapid separation technology based on large column chromatography, through the different amount of asphalt components separation test, and the results are compared with the standard test results. The results show that the error of the separation of the asphalt component is less than 1.2%, and the other three components are less than 1.6%, which meets the requirement of the specification. The results obtained from this method are similar to the standard test results, which can be used for the rapid separation of asphalt components, and the content of the components is increased, which is convenient for the follow-up study of the components.

1. Introduction

Asphalt, as a very important geotechnical material, has been widely used in road engineering and building waterproofing works. From the beginning of nineteenth Century, people began to use the asphalt road. Today, asphalt pavement has become the main road pavement structure [1-3]. As an important road building material of asphalt pavements, the performance index and the quality of asphalt mixture directly affect the pavement performance and service life of asphalt pavement. The performance of petroleum asphalt depends on the difference of oil source and production method, especially the difference of chemical composition. Any changes in composition, chemical structure and binding form will directly affect the physical and chemical properties of asphalt [4-7]. In order to find out the difference and correlation, it is very important to study the relationship between the chemical composition of asphalt and the road performance.

The four component determination method is widely used among the many methods of measuring the chemical composition of asphalt. The existing JTG E-20-2011 "Highway Engineering Asphalt and Asphalt Mixture Test Procedures" in the four components of the asphalt test is the classical column chromatography. However, in the actual analysis work, it is often found that the results the accuracy is not high, the reproducibility of the experiment is not good [4, 8-10]. In the specification, the existing components analysis method can only be used to analyze 0.5-1.0g asphalt sample, the amount of the single component of each test is often less than 0.1g. And in the process of testing, the separation speed is slow, and the operation time is too long. This will not only lead to large test errors, but also is



not conducive to the analysis and testing of single component and long-term performance. Based on this, some scholar [14-15] has been proposed a double hammer decompression type asphalt four points separating device and aliquots of solution sampling method for improving the original method and increases the accuracy of the results. But it does not solve the problem of small amount of separated asphalt.

Therefore, it is necessary to develop a high efficiency and fast component analysis method that can provide sufficient quantities of a single sample for the follow-up quality evaluation and long-term performance research. Therefore, based on the existing component analysis technology, the fast component analysis method was put forward by developing a large column, and the effect for different types of asphalt in the component analysis test was investigated. Its advantages include increasing the accuracy and repeatability of the test results, achieving rapid separation, and the increased content four component of the asphalt so that easy to do other analysis.

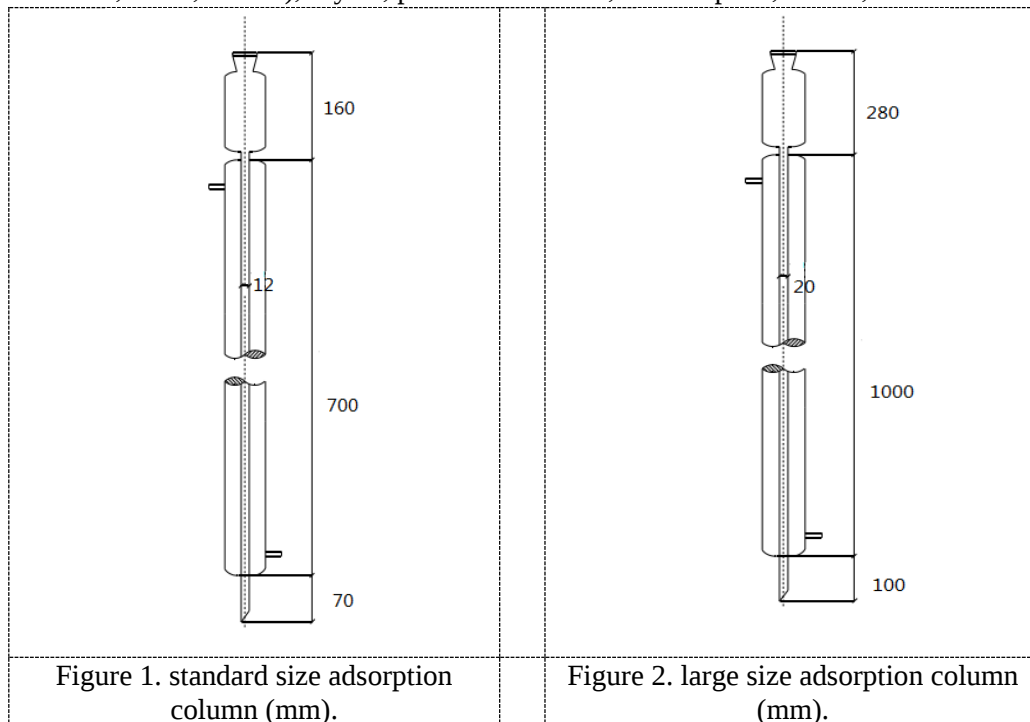
2. Experimental

2.1 Reagent

The asphalt are Shell -90 matrix, Shell -70 and SK-90 asphalt. The reagents are neutral Al_2O_3 (100 to 200 mesh, specific surface area is greater than $150 \text{ m}^2/\text{g}$, empty volume $250 \text{ mm}^3/\text{g}$), heptanes (A.R.), toluene (A.R.) and ethanol (A.R.).

2.2 Experimental Instruments

The experimental instruments include bitumen extractor and condenser, glass adsorption column (shown in Figure 1), large glass adsorption column (shown in Figure 2), 50°C thermostat water tank (HH.W21, temperature control accuracy of 1°C), vacuum drying box (DZF type), muffle furnace (SX-4-10), rotary evaporation apparatus (RE-52AA), digital display intelligent temperature controlled magnetic stirring apparatus, analytical balance (FA1104 and JA5003), conical flask (250mL, 500mL), cylinder (20mL, 50mL, 100mL), dryers, porcelain crucible, enamel plate, funnel, etc.



2.3 Test Content and Methods

According to the requirements of E-20-2011 JTG "Highway Engineering Asphalt and Asphalt Mixture Test Procedures", the four components of different types of asphalt were tested, and the data were obtained as the control group. Then specification standard adsorption column for large adsorption column chromatography and increase the asphalt dosage, asphalt of different dosage of four component test and the experimental results were compared with that of the control group, and to compare the separating time length. The vacuum pump was adopted by connecting the bottom of adsorption column instead of double ball on the top of the adsorption column in order to increase the pressure and improve the separation speed. Each asphalt sample was tested three times, and the average value of four component of the asphalt was taken.

3. Experimental results and analysis

3.1 The test results of different asphalt sample content

According to the E-20-2011 JTG "Highway Engineering Asphalt and Asphalt Mixture Test Procedures", 1g asphalt sample was carried out, and the component content of three kinds of asphalt was determined, and the test results are shown in table 1.

Table 1. The test results of four components of 1g asphalt (%)

Asphalt type	Test times	Saturates	Aromatics	Resins	Asphaltene	Rate of Recovery
Shell -70	1	12.1	40.6	31.7	15.6	91.8
	2	12.9	41.2	30.1	15.8	93.2
	3	13.1	39.9	30.8	16.2	89.8
	mean value	12.7	40.6	30.9	15.9	91.6
Shell -90	1	13.4	46.7	32.1	7.8	96.1
	2	12.6	46.1	33.4	7.9	92.9
	3	13.1	47.1	31.8	8.0	91.7
	mean value	13.0	46.6	32.4	7.9	93.6
SK-90	1	12.7	38.0	33.4	15.9	88.9
	2	13.2	37.3	33.2	16.3	90.2
	3	12.1	38.5	32.6	16.8	93.3
	mean value	12.7	37.9	33.1	16.3	90.8

It could be known from the analysis table data, the maximum difference of the saturates content of shell-70 asphalt was 1%, the maximum deviation of the aromatic content was 1.3%, the maximum difference of the resins content was 1.6% and the maximum difference of the asphaltene content was 0.6%. The maximum difference of the four component content of shell-90 asphalt and SK-90 asphalt was also limited. In the above three kinds of asphalt, saturates content deviation was less than 1.2%, and the error of the other three components were less than 1.6%. In accordance with the specification error requirements, the test separation method of the four components of the asphalt was accurate and good repeatability.

According to the E-20-2011 JTG, 4gasphalt sample was carried out, and the component content of three kinds of asphalt was determined, and the test results are shown in table 2.

Table 2. The test results of four components of 4g asphalt (%)

Asphalt type	Test times	Saturates	Aromatics	Resins	Asphaltene	Rate of Recovery
Shell -70	1	12.5	40.3	31.9	15.3	96.3
	2	13.4	39.6	31.1	15.9	93.2
	3	12.1	41.0	30.6	16.3	95.9
	mean value	12.7	40.3	31.2	15.8	95.1
Shell -90	1	13.2	46.3	32.5	7.7	94.8
	2	13.0	46.9	32.2	7.6	97.2

	3	12.1	46.1	33.4	8.4	93.5
	mean value	12.8	46.4	32.7	7.9	95.2
SK-90	1	12.3	38.9	33.6	15.2	91.9
	2	12.5	38.4	33.2	15.9	95.8
	3	13.4	37.5	32.8	16.3	92.1
	mean value	12.7	38.3	33.2	15.8	93.3

The maximum difference of the saturates content of shell-70 asphalt used in asphalt content of 4g was 1.3%, the maximum deviation of the aromatic content was 1.4%, the maximum difference of the resins content was 1.3%, the maximum difference of the asphaltene content was 1.0%. The maximum difference of the four component content of shell-90 asphalt and SK-90 asphalt was similar with that of shell-70 asphalt. In the above three kinds of asphalt, saturates content deviation was less than 1.2%, and the error of the other three components were less than 1.6%. The test separation of the four components of the asphalt was accurate and good repeatability.

According to the E-20-2011 JTG, 8g asphalt sample was carried out. The component content of three kinds of asphalt was determined, and the test results are shown in table 3.

Table 3. The test results of four components of 8g asphalt (%)

Asphalt type	Test times	Saturates	Aromatics	Resins	Asphaltene	Rate of Recovery
Shell -70	1	13.2	39.1	32.3	15.4	94.3
	2	12.1	40.7	32.6	14.6	97.7
	3	13.1	40.2	31.6	15.1	91.9
	mean value	12.8	40.0	32.2	15.0	94.6
Shell -90	1	12.6	46.1	33.4	7.9	96.1
	2	12.3	46.1	33.4	8.2	92.1
	3	12.6	45.7	33.4	8.5	88.4
	mean value	12.5	46.0	33.4	8.2	92.2
SK-90	1	13.3	37.2	33.1	16.4	95.0
	2	12.1	38.6	33.8	15.4	96.8
	3	12.7	37.9	33.6	15.8	91.3
	mean value	12.7	37.9	33.5	15.9	94.4

The maximum difference of the saturates content of shell-70 asphalt used in asphalt content of 8g was 1.1%, the maximum deviation of the aromatic content was 1.6%, the maximum difference of the resins content was 1.0%, the maximum difference of the asphaltene content was 0.8%. The maximum difference in the saturation content of shell-90 asphalt and SK-90 asphalt was also similar with that of shell-70 asphalt. In the above three kinds of asphalt, saturates content deviation was less than 1.2%, and the error of the other three components were less than 1.6%. In accordance with the specification error requirements, the separation test of the four components of the asphalt was accurate and good repeatability.

3.2 Test results of three kinds of different asphalt content

Figures 3 to 5 are the four components of Shell-70, Shell -90, and SK-90 asphalt at 1g, 4g and 8g. It can be known from the data, the maximum difference of saturates content of shell-70 asphalt with different asphalt content is 0.1%, the maximum difference of the aromatic content is 0.6%, the maximum difference of the resins content is 1.3%, the maximum difference of the asphaltene content is 0.9%. The maximum deviation of four components content of shell-90 asphalt and SK-90 asphalt with different asphalt content was also very small.

In the above three kinds of asphalt content, the deviation of saturates was less than 1.2%, the error of the other three components were less than 1.6%. And from the graph, it also can be found that the separation accuracy of the components from the large column separation and the requirement of the 1g

asphalt sample is roughly similar. So it is concluded that the separation test result of the asphalt component by the large column method is reliable.

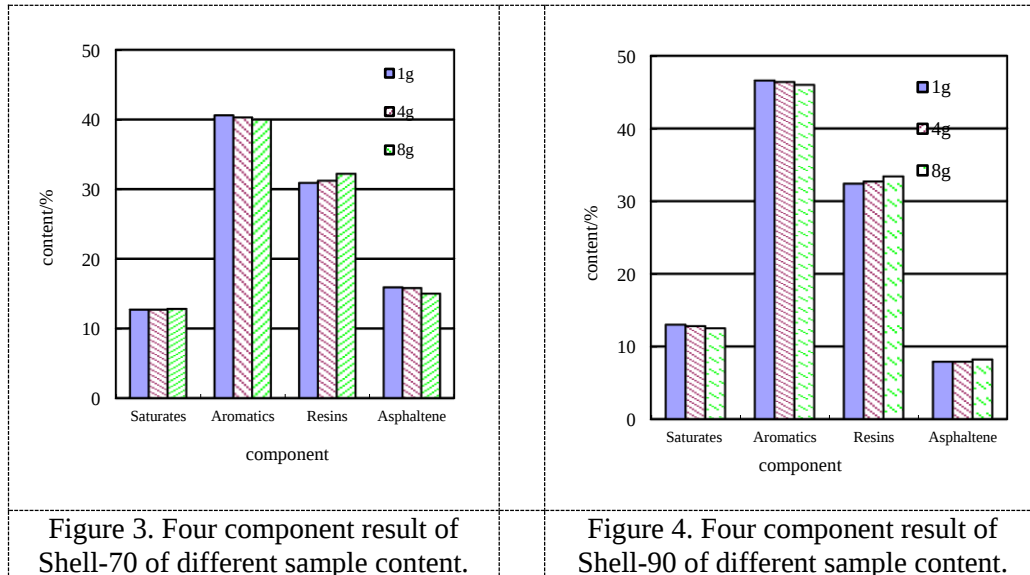


Figure 3. Four component result of Shell-70 of different sample content.

Figure 4. Four component result of Shell-90 of different sample content.

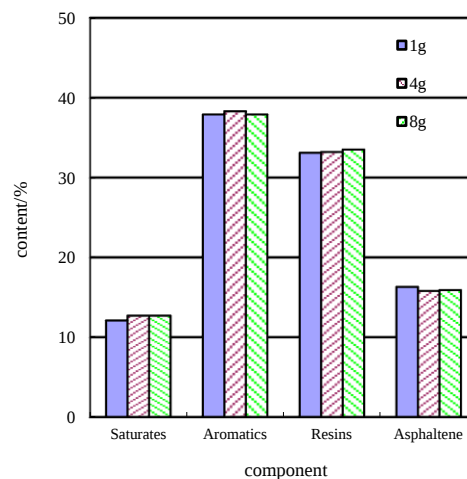


Figure 5. Four component result of SK-90 of different sample content

4. Conclusion

The experimental results show that the deviation of the three kinds of asphalt is less than 1.2%, the deviation of the other three components are less than 1.6%, which are in line with the requirements of the specification error. The separation results obtained from the large column method and the standard test results are similar and it can be used for the experimental analysis. When the column diameter is increased, the length of the column is increased, and the amount of the washing agent is increased, and the flow rate is faster than that of the standard column. At the same time, the amount of the separated asphalt increased, the amount of the component also increased, which is convenient for the subsequent properties and chemical analysis of each component of asphalt.

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