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Full Length Article

Identification of asphalt aging characterization by spectrophotometry technique

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ABSTRACT

During the construction and service life of asphalt pavement, the bituminous material is inevitably exposed to sunlight, oxygen, water, ambient temperature and other climate conditions, which lead to aging or oxidation of asphalt binder and thus accelerate the deterioration of asphalt pavement. In order to characterize the aging impact of asphalt materials, conventional methods have been used including rheological test, Fourier transform infrared spectroscopy (FTIR), Gel Permeation Chromatography (GPC), etc. However, some drawbacks limited their application in practice such as the complexity of test procedures and the expensive test instruments. A novel method, spectrophotometry, was introduced in this study to analyze the aging characteristics of asphalt binder. The sample preparation method and test procedures were developed and the correlation analysis was used to assess the feasibility of this method. The experiments illustrated that a 2 g/L "toluene-heptane" asphalt suspension (0.2 g/mL in toluene, 2 g/L in heptane) and a test of absorbance area from 700 nm to 900 nm could be effectively used to characterize the aging of asphalt. Furthermore, the test and statistical results also demonstrated that the absorbance change of asphalt during the aging procedure was from the increased asphaltene. Moreover, the FTIR test indicated that the feasibility of using spectrophotometry to characterize the aging of asphalt. In addition, the research indicated that the sources and penetration grades could not be used as mutual indices to characterize the aging resistance of asphalt.

1. Introduction

As a kind of organic material, asphalt is prone to be aged with the exposure to oxygen, water, sunlight and other climate conditions during its service life. Aging is associated with many physical and chemical reaction processes, such as volatilization, oxidation, condensation [1]. These physical and chemical reaction processes can harden the asphalt materials and accelerate many pavement distresses like raveling, potholes, cracking. Thus, the study of asphalt aging impact is conducive to better understanding of the aging mechanism as well as providing a decision-making basis for pavement management and maintenance.

Aging preponderantly occurs in two stages in the life cycle of asphalt pavement. The first is the pavement construction stage, including the process of mixing, transportation, laying and compaction, and the second is the pavement service stage. In the laboratory, rolling thin film oven (RTFO) and pressurized aging vessel (PAV) are commonly used to simulate these two aging stages. The aging of asphalt is characterized by the increase of viscosity and brittleness or the decrease of flexibility in a macroscopic point of view. Therefore, characterization of the aging effect was mostly based on the rheological indices such as penetration, soft point, phase angle (δ), $G^*/\sin\delta$ [2]. Meanwhile, some new techniques such as FTIR, GPC, etc. were applied to characterize the aging properties of asphalt from different perspectives [3].

Generally, it is feasible to characterize the asphalt aging impact or aging degree by rheological test, FTIR, GPC, etc., but these methods have some drawbacks. For example, all of these instruments are expensive. They are mostly equipped in scientific research institutions and universities whereas rarely used by construction contractors. Meanwhile, the testing process is very tedious, such as a long time of heat preservation to ensure the accuracy as well as a complex sample preparation process [4]. In addition, these test setups are complex and require harsh indoor conditions. All of these methods exhibit a low test efficiency, nor can they perform a real-time detection of the asphalt aging degree in the field.

Spectrophotometry is mainly used in environmental monitoring and petrochemical industry. For example, many researchers used UV–Vis spectrophotometer to determine the oil content in wastewater [5]. In 1984, Miershaya conducted a test to compare the absorbance

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differences of asphalt before and after removing asphaltene in order to determine the content of asphaltene in asphalt [6]. This method needs to extract the asphaltene first, which is time consuming. After that, Fukui designed a double-wavelength method to test the asphaltene content in vacuum residuum by spectrophotometry [7]. This method could perform a rapid determination of the asphaltene content in vacuum residuum. However, when applied on testing asphalt, the accuracy decreases because of a high asphaltene content in asphalt. After that, Liu et al. carried out some relevant research and optimized some experimental parameters to improve the accuracy [8].

In recent years, spectrophotometry has gradually been introduced to road-engineering field to the study asphalt and additives. Some researchers used spectrophotometry to analyze the volatile and dissolved organic matters during the heating process of asphalt in order to develop the flame retardant materials [9]. Soenen et al. found that the absorbance of asphalt increased between 200 ~ 600 nm after aging, attributed this phenomenon to the increase of large conjugated aromatic rings during the aging procedure [10]. In addition, the absorbance between 200 and 610 nm was used to characterize the elasticity of asphalt due to the relationship between these large conjugated aromatic rings and asphalt performance [11]. However, there are no systematic studies related to the application of spectrophotometry on asphalt aging characterization.

Spectrophotometry is a quantitative and qualitative method for analyzing the content of organic components and is developed based on colorimetric method. As a complex organic material, the aging of asphalt is essentially the chemical change of each component. Therefore, it is potential to characterize the component change of asphalt by spectrophotometry technique and further depict the aging effect or aging degree of asphalt binder. Due to the simplicity of the equipment and test procedure, a rapid description can be achieved.

The objective of this study is to provide a new method to analyze the aging effect on asphalt and assess its feasibility in characterization of asphalt aging. In this study, spectrophotometry was introduced firstly to detect the aging effect on asphalt binder by analyzing the relationship between asphalt components and their absorbance and conducting the correlations of these factors. Meanwhile, FTIR test results were used to demonstrate the feasibility and accuracy of this spectrophotometry application.

2. Materials and test methods

2.1. Materials

To make the spectrophotometry universally applicable, eight asphalts from five sources ((Liaoning, Qinhuangdao, Zhonghaiyou, SK and Qilu Petrochemical, referred from A to E.) were used in this study. Moreover, there were two penetration grades (referred to # 70 and # 90) for each source of asphalt except for D and E. The main rheological properties of these binders are shown in Fig. 1.

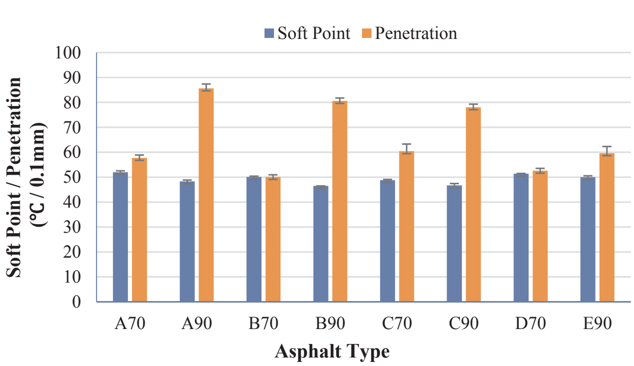

Fig. 1. Rheological properties of eight asphalts.

To prove the feasibility of spectrophotometry to characterize the asphalt aging, three kinds of tests were performed including spectrophotometric test, two-component (asphaltene and malthene) separation test and FTIR test. The separation of asphaltene and malthene allows clarifying the component change of asphalt, especially the change of asphaltene because of its susceptibility to aging. Given the relationship between the component content and the absorbance, the quantitative study of asphalt component is propitious for the further analysis. The spectrophotometric tests include the absorbance of asphalt, asphaltene and malthene in three aging situations. The objective is to clarify the fundamental cause of asphalt absorbance changing in aging procedure. FTIR is a basic chemical analysis method and it has been demonstrated that the quantitative analysis of functional groups can describe the aging of asphalt. It is introduced in this study to analogically explain the feasibility of spectrophotometry in asphalt aging characterization.

These three kinds of tests are proved mutually, but only a qualitative evidence is not sufficient to demonstrate a new method. Therefore, the Pearson correlation analysis is conducted to quantitatively assess the consistency of the results obtained from those three kinds of test and a high consistency is concrete to prove the feasibility. The experiment design for this study is shown in Fig. 2.

2.2. Aging method

Rolling thin film oven (RTFO) and pressurized aging vessel (PAV) methods were used in laboratory to simulate the aging process in construction and operation stage of asphalt pavement, according to AASHTO T 240 and AASHTO R 28 respectively, and meanwhile the aged asphalt samples were obtained.

2.3. Component separation

Asphalt was separated into two components (referred to asphaltene and malthene) according to their solubility in heptane solution. During the separation procedure, asphalt was solved in heptane and then asphaltene was filtered. In addition, an extractor was used to remove the residual malthene on the filter paper. The percentage of each component could be measured after the solution was evaporated.

2.4. Principle of spectrophotometry

Spectrophotometry is an optical test method, developed based on colorimetric method. Colorimetry is used to determine a specific component content by comparing the depth of the solution color. It includes visual colorimetry and photoelectric colorimetry. Both are based on the "Lambert-Bill" law, which is shown in formula (1). When a monochromatic light passes through a uniform absorbing material, the absorbance "A" is proportional to the concentration "c" and the thickness of the absorption layer "b".

$$A = \log\frac{1}{T} = kbc \tag{1}$$

With the development of optical instruments, especially the application of dispersion units such as grating and prism, the wavelength of the monochromatic light became unique. Thus, the spectrophotometric method is considered to effectively test the sample in every unique wavelength. In general, the development from colorimetry to spectrophotometry is a process of approaching "Lambert-Bill" law. It is also the basis of improving the accuracy. Fig. 3 presents the schematic diagram of the spectrophotometer used in this study.

2.5. Sample preparation and test procedure for spectrophotometry

The spectrophotometric test mainly follows two procedures, the sample preparation and the absorbance test. As a key procedure in spectrophotometry, the sample preparation method directly affects the