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# Evaluation of the temperature effect on Rolling Thin Film Oven aging for polymer modified asphalt



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### HIGHLIGHTS

• A drastic carbonyl jump at certain aging temperature levels was observed for PMA.

• Equal aging effect between RTFOT and MRTFOT was detected after carbonyl jump.

Rheology master curves were constructed to validate the findings of FTIR test.

• To guarantee fluidity, the aging temperature should be set above 178 °C for PMA.

#### ARTICLE INFO

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### ABSTRACT

Rolling Thin Film Oven Test (RTFOT) was found to be inadequate for polymer modified asphalt (PMA) since high viscosity PMA will not roll inside the glass bottle during the test. Many suggested that the aging temperature of RTFOT should be promoted for PMA; however the degree of promotion remains to be decided. To investigate the impact of temperature on RTFOT aging and determine a suitable PMA aging temperature, an innovative approach was proposed by comparing aging effect of RTFOT and Modified Rolling Thin Film Oven Test (MRTFOT) at different aging temperatures. Infrared spectroscopy and rheology characteristic were employed to evaluate aging.

Carbonyl change suggested that with aging temperature lifted, a drastic increase in carbonyl combined with an equal aging effect between MRTFOT and RTFOT occurred at certain aging temperature, and was inferred to be an indication of PMA obtaining fluidity. PMA rheology master curves exhibited consistent results with carbonyl change. Based on above findings, a commonly used PMA with 4.5% styrene–buta diene–styrene should be aged at 178 °C in RTFOT to guarantee fluidity and equal aging effect compared with neat asphalt

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#### 1. Introduction

Rolling Thin Film Oven Test (RTFOT) is one of the most common used test methods to simulate aging of asphalt plant mixing. It has been applied for many years and was included in AASHTO and ASTM standard. However RTFOT has been reported not suitable for Polymer Modified Asphalt (PMA) [1]. Researches showed that as a result of the rather lower aging temperature of 163 °C, PMA with high viscosity will not melt completely and spread into uniform thin film [2]. Absence of fluidity in RTFOT leads to insufficient aging and causes serious problems, one of which was the inapplicability of  $G^*/\sin\delta$  in evaluating PMA [3]. According to the survey facing contractors, suppliers and academia conducted by NCHRP Project 9-10, PMA short-term aging was considered as one of the most problematic areas [4].

Due to the drawbacks of RTFOT, researches from two directions merged in order to find an appropriate way to simulate short-term aging for PMA.

Some tried to develop whole new aging methods to replace RTFOT. New equipment such as Modified German Rotating Flask (MGRF) [5], Stirred Air Flow Test [6] and Rotating Cylinder Aging Test [7] were proposed in recent years as alternatives to RTFOT and the latest outcomes were reported in NCHRP Project 09-36 [8]. The report concluded that MGRF was an acceptable alternative to RTFOT for both neat and modified asphalt binders. However, due to the inconvenience and financial issue of renewing equipment, new aging methods like MGRF did not gain popularity [9].

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On the other hand, a number of researchers attempted to modify RTFOT for PMA, so one can age both neat and modified asphalt without replacing the whole equipment.

The foundation of the RTFOT method is to spread 35 g asphalt binder into homogeneously thin film with a thickness of 5– 10  $\mu$ m. Hence the binder will be aged efficiently and uniformly [10]. Neat asphalt can easily obtain fluidity in aging bottle while PMA with high viscosity may be too stiff to roll. Thereby, if any possible measure is taken to force PMA roll in the aging bottle as neat binder does, the use of RTFOT for PMA will be suitable.

A typically modification is the Modified Rolling Thin Film Oven Test (MRTFOT) proposed by Bahia [11]. In the MRTFOT, a steel rod or a number of steel spheres were put into the glass bottle to create extra shearing forces to force the spreading of thin film. Evaluations indicated that the steel rod was more practical, and was easier to use and clean after aging. Steel rod of 127 mm long and by 6.4 mm in diameter was recommended in NCHRP Project 9-10 [4]. However, some investigations reported that the increase in aging effect was not satisfying and the MRTFOT may not age PMA fully as expected [12].

Besides the modification in equipment, a number of researches tended to upgrade the RTFOT by revising the test parameters [15–17]. And aging temperature and aging period are the most influential parameters and gain most concerns.

According to AASHTO standard, in the RTFOT, asphalt binder will be aged in a glass bottle for 85 min at the temperature of 163 °C. The proposer of the RTFOT, California highways, determined the 85 min test period from actual field calibration on a series of asphalt binders. The suitability of this test period has been confirmed by a correlation study with the calibrated Ottawa sand mixers [10]. There was rare debate about the 85 min aging period. Most controversies were raised by the aging temperature. Many pointed out that the temperature of 163 °C was established on neat asphalt field investigations and apparently lower than the actual PMA mixing and planting temperature [1].

Through literature review [17], it was found that there was no as much as field investigation in the establishment of 163 °C aging temperature compared with aging period. As the replacement of the last generation aging method Thin Film Oven Test (TFOT), RTFOT directly followed the 163 °C aging temperature from TFOT in 1963. Similarly, TFOT inherited this temperature in 1940 from the older aging method [18]. The 163 °C aging temperature was found originating in 1903 when Dow heated asphalt binder and measured the change in weight and penetration [19]. And back then, PMA was far from being invented or applied. Hence it is reasonable to doubt the suitability of 163 °C for PMA.

Numerous of researches agreed with increase in aging temperature to enhance the aging effect for PMA. However there was no clear conclusion about how much aging temperature should be raised. The level of increase is arbitrary and varies from different researches in a range of 10–30 °C [15–17]. Some tried to find the suitable aging temperature for modified asphalt by using viscosity-temperature curve [20]. It is well-known that based on a great amount of field investigation, a correlation was found between mixing temperature and asphalt viscosity, and the optimum neat asphalt viscosity for mixing was 0.17 Pa.s [21]. It seems reasonable to determine the aging temperature according to the optimum viscosity. However, the estimated PMA aging temperature acquired from the optimum viscosity of 0.17 Pa.s often tends to be higher than expected. At some cases, the estimated temperatures can reach and even exceed 200 °C. Thus the reasonability of 0.17 Pa.s regard to PMA is debatable, and the optimum PMA viscosity and aging temperature for the RTFOT remain to be decided.

It should be noticed that rise in temperature improved aging from two aspects. First, heating-up lowers the viscosity of PMA and makes binder fluid enough to spread into thin film as expected. Second, higher temperature leads to more molecular activities in asphalt and subsequently more chemical reaction with oxygen. While both sides have significant effects on aging, one cannot tell which factor governed the enhancement in aging when adjusting aging temperature, and cannot tell at what temperature PMA can obtain fluidity in RTFOT.

This study tried to decide whether the PMA in the aging bottle has obtained fluidity at a certain temperature by comparing the aging effect between RTFOT and MRTFOT. As mentioned before, MRTFOT is one of the most famous modifications of RTFOT. And one important precondition of MRTFOT is the introduction of steel rod will not affect the aging of neat asphalt. It is because that neat asphalt has already obtained fluidity at 163 °C without steel rod, thus extra shearing force has little effect [4]. In other words, 163 °C is suitable for neat binder while PMA requires higher aging temperature. Based on this assumption. If MRTFOT shows more serious aging effect than RTFOT at a certain temperature, then the PMA is still too stiff and the aging temperature should be further raised. When the MRTFOT and RTFOT finally show an equal aging effect, one can consider that at this temperature, PMA has obtained fluidity and fully aged by RTFOT. Hence, by comparing the aging effect of RTFOT and MRTFOT, a suitable PMA aging temperature can be determined.

#### 2. Objectives

This study aimed to assess the aging temperature impact on RTFOT aging regard to PMA, and to propose a method to determine the suitable RTFOT aging temperature for PMA.

PMAs of different styrene–butadiene–styrene (SBS) content were aged at different temperatures through RTFOT and MRTFOT respectively. After aging, infrared spectroscopy and rheology characteristic of residue were analyzed to evaluate the aging effect.

It should be noted that the suitable temperature mentioned in this study is the certain temperature at which PMA obtains fluidity and can be fully aged by RTFOT. The author holds the opinion that one should first guarantee the applicability of RTFOT for PMA, and then it will be appropriate and possible to consider revising the test parameters from the perspective of practical relevance.

#### 3. Experimental

#### 3.1. Materials

In this study, one neat base binder, one SBS modifier and elemental sulfur were selected to prepare PMA in laboratory. Base binder was ESSO asphalt (PG64-16). SBS T161B was produced by DuShanZi Petroleum and Chemical Corporation, China. SBS T161B was a kind of radial polymer with average molecule weight of 230,000 g/mol, containing 30 wt% of styrene. The amount of SBS modifier was set at 4.5%, 6.0% and 7.5% by weight of base binder. In industry production, elemental sulfur and organic sulfide were commonly used as cross-linking agent to produce storage stable PMA. In this study, elemental sulfur was used as a cross-linking agent with 0.15 wt% of base binder.

Following procedure was taken to prepare the SBS modified asphalt according to the method disclosed in the patent [22]. Firstly, SBS was added to base binder and sheared for 30 min at 180 °C with high shear mixer at the shear speed of 4000 r/min. Secondly, the blend was stirred for 60 min using mechanical stirrer at 180 °C. Thirdly, cross-linking agent was added to the blend and stirred for another 240 min at 180 °C. The resulted binders satisfied the requirements of storage stability test, and because PMAs were

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