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# Effects of pore structure on oxidative aging and related mechanical properties of asphalt concrete



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#### HIGHLIGHTS

• Effects of different type of pores on changes in sulfoxides and ketones.

• Effects of different type of pores on modulus and hardness of asphalt binder.

• The necessity of pore structure evaluation for aging study.

#### ARTICLE INFO

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#### ABSTRACT

Aging in asphalt concrete (AC) is an important issue that has been well studied by researchers for decades. Although some works have been conducted to evaluate the effect of total pore on aging, there is lack of study on how different type of pores or pore structure affects the aging process and related mechanical properties. This study attempted to evaluate the effects of pore structure on oxidative aging and related mechanical properties of AC. Slab samples were prepared in the laboratory using a linear kneading compactor. Air voids of the samples range from 7% to 20% to cover dense graded to highly open graded pavements. Samples were kept inside the laboratory for three years to age by action of air only. Pore structure was evaluated using tracer test. Aging was quantified using Fourier Transform Infrared Spectroscopy (FTIR). Mechanical properties were determined by binder fatigue test using a Dynamic Shear Rheometer (DSR) and by nanoindentation. It was observed that aging increases with the increase of total pore, effective pore, or permeable pore. Moduli from both nanoindentation and DSR increase with the increase of all type of pores. Fatigue life decreases linearly with the increase of all type of pores. It was also observed that the use of permeable or effective pores doesn't improve the accuracy of the pore versus aging study.

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#### 1. Introduction

Aging in asphalt is defined as the changes of chemical composition over time that affects its physical properties (in this study, aging refers to oxidative aging). Aging occurs in three different ways: evaporation of volatile compounds, oxidation and steric hardening. Evaporation of volatile materials occurs mostly during the production, storage, transport and lying of asphalt, thus, mostly before construction. Oxidation occurs throughout the lifetime, although the rate decreases with time. Steric hardening occurs due to internal reactions over the time [1]. Oxidative aging is mostly responsible for hardening of asphalt; therefore, it is the main focus of research [2–4]. Oxidation mainly converts sulfides into sulfoxide, and benzyl carbon to ketones [5–7]. Changes in aromatics also occur but are very negligible. Saturates act as an inert group during oxidation. Therefore, tracking the changes in sulfoxides and ketones leads to the understanding of the degree of oxidative aging. Fourier Transformed Infrared Spectroscopy (FTIR) is widely used to detect sulfoxides and ketones.

Aging increases the viscosity, hardness, and modulus of asphalt and decreases fatigue life and ductility [8–12]. Oxidative aging is a mostly irreversible process although sulfoxides concentration sometimes decreases at elevated temperatures. Aging increases stiffness as much as 30% and dynamic modulus as much as 60% [13]. As asphalt becomes hard, its penetration capacity decreases



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with the increase of aging. Aging also reduces the healing capacity of asphalt binder [14].

Aging of asphalt concrete (AC) generally represents oxidation of asphalt binder. Binder should have adequate access to oxygen, therefore, access to air. Not all the pores in AC are accessible to air. Isolated air voids are inaccessible to air and therefore should have little to no oxidation. On the other hand, permeable pores are accessible to air and binders near the permeable pores are expected to be oxidized more. At lower air voids, pores are mostly discontinuous and isolated. With the increase of total pores, connectivity of pores increases exponentially. Only a few studies are available correlating aging with different type of pores [2,15].

To evaluate permeable pores, X-ray CT scans could be used [16]. However, it is a costly and time consuming process and does not measure different types of pores directly. On the other hand, tracer test is very popular by soil scientists for evaluating permeable porosity [17]. Tarefder and Ahmad showed that tracer test is a very cost effective, quick and direct procedure to determine pore structure of AC [18], although, it has a limitation that the total pore should be more than 6%. To evaluate stiffness and fatigue life of binder, Dynamic Shear Rheometer (DSR) is generally used. As asphalt is used as a thin layer around the aggregate, it is more logical to use nanoindentation to evaluate hardness of asphalt binder [19].

#### 2. Objective

The main objective of this study is to evaluate the effects of pore structure on oxidative aging and related physical changes in AC. The objective could be accomplished by evaluating

- (i) Effects of different type of pores on changes in sulfoxides and ketones.
- (ii) Effects of different type of pores on modulus and hardness of asphalt binder.
- (iii) The necessity of pore structure evaluation for aging study.

#### 3. Methodology

Asphalt concrete mix was collected from a construction site on Interstate 40 (I-40) in New Mexico. Three laboratory slabs were compacted using linear kneading compactor. The target air voids for the three slabs were 7%, 14% and 20% to cover all ranges from dense graded to highly open graded pavement [20]. It was assumed that each slab is homogeneous and material properties determined for any part of a slab represents the whole slab. The slabs were kept under open air inside the laboratory for three years. The three years' period was chosen expecting that the binders would undergo significant amounts of oxidation by this time.

Following the natural aging process by air only for the three years, the slabs were ready for testing. One 6 in. diameter core was collected from the center of each of the slabs. The bulk specific gravity and percent absorption of the cores were determined as per ASTM standards. Cores were tested for permeable porosity using the tracer test device. As petroleum jelly was used during tracer test, the cores were no longer usable for further analysis.

The remaining part of each slab after coring was cut down into four pieces. A randomly chosen piece was kept in an oven at 150 °C for one hour to make a loose mix. Asphalt binders were extracted from each of the three samples using a coarse extractor, centrifuge and rota-vapor. The extracted binders were tested for chemical composition by FTIR. Elastic modulus and hardness were determined using nanoindentation. DSR was used to determine complex modulus and fatigue life. Once all the tests were completed, analysis were made to observe how aging and related mechanical properties are affected by pore structure of AC.

#### 4. Materials and sample preparation

The mix was collected during construction of a new overlay on I-40, mile marker 142. The mix was a dense graded Superpave, type SP-III with the nominal maximum aggregate size of 19.00 mm. The mix contained 35% Reclaimed Asphalt Pavement (RAP) materials and a PG 70-22 binder. The asphalt content was 4.4% by the weight of the mixture. The properties of the mix are listed in Table 1. Three slab samples of 30 cm by 45 cm were prepared using a linear kneading compactor.

#### 5. Laboratory testing

#### 5.1. Test for total pore and effective pore

Bulk specific gravity of each cylindrical sample was determined as per ASTM D6752 [21] which uses vacuum seal method. The dry weight of a sample was measured. The sample was placed inside a plastic bag of known specific gravity. It was sealed using a Corelok<sup>M</sup> device. The sealed sample was placed under water. The weight was measured before and after opening the plastic bag. Bulk and apparent specific gravity of the sample were calculated from Eqs. (1) and (2) respectively;

$$G_{mb} = \frac{A}{A + D - B - D/F} \tag{1}$$

$$G_{ma} = \frac{A}{A + D - C - D/F} \tag{2}$$

where A = sample dry weight in air; D = polybag weight in air; B = weight of sealed sample in water; C = weight of sealed sample and polybag cut under water; F = specific gravity of polybag at 25 °C.

As the maximum specific gravity is already known from the mix design, the total pores were calculated using Eq. (3). Total pores and air voids are used interchangeably in this paper;

$$n = \frac{G_{mm} - G_{mb}}{G_{mm}} \tag{3}$$

Effective pores are all the pores that are accessible to water. It is the sum of dead-end and permeable pore [22]. Effective pore was determined using Eq. (4).

$$n_e = \frac{G_{ma} - G_{mb}}{G_{ma}} \tag{4}$$

#### 5.2. Test for permeable pore

Permeable pores are defined as the pores that are capable to transmit water from one side to other side of the pavement. It can be determined easily using a tracer test. Tracer test set up consists of a salt-meter attached to a permeameter as shown in Fig. 1

 Table 1

 Material Properties of the AC.

Property	Value	Property	Value
RAP content	35%	Total Asphalt Content	4.4%
Recycle Binder Ratio	0.75	Air void	7-20%
G <sub>mm</sub>	2.573	Compaction Temperature	156 ± 2 °C
Versa bind	1%	Final blend grade	76-22
Binder grade	PG 70-22	-	

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