

AGING OF ASPHALT BINDERS AND ASPHALT MIXTURES. SUMMARY PART I: EFFECT ON PHYSICAL-CHEMICAL PROPERTIES

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ABSTRACT

The phenomenon of aging of asphalt binders and asphalt mixtures impacts directly in the physical-mechanical response and durability of asphalt pavements. Because of such reason, multiple research efforts have been carried out in relation to this topic. By conducting a deep bibliographical review, the most important aspects that the pavement engineer needs to be aware of in relation to the aging of asphalt binders and asphalt mixtures were organized and summarized. This information was summarized and described in two articles. This first one (Titled Part I), introduces and describes the changes that physical-chemical properties of asphalt binders and asphalt mixtures undergo when they age. Additionally, commonplace tests for attempting to evaluate said properties in these materials are introduced.

Keywords: aging, chemical properties, physical properties, asphalt binder, asphalt mixture.

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1. INTRODUCTION

The aging of asphalt binder is an irreversible process which reduces the durability of pavement and causes early damages and an increase in repair and maintenance costs. [1-2]. Aged asphalt binder generates cracking under either load or thermal stress, and other damage

mechanisms such as stripping, pothole, ravelling of aggregates, among others [3-5]. The formation of these types of discontinuities in asphalt mixtures undermines its integrity and provides additional pathways for the infiltration of water and oxygen into the asphalt-aggregate interface and accelerates damage with time [6].

Asphalt binder aging is mainly caused by thermal oxidation, which occurs thanks to the relationship with oxygen. Said oxidation is mainly produced during manufacturing operations in asphalt production plants (including in storage), transporting and placement of asphalt mixture (extension and compaction), accompanied by the volatilization of light compounds and the reorganization of asphalt microstructure [7]. This process is known as short-term aging [8-9]. Another important source of oxidative aging is the photo-oxidation of asphalt binder caused by ultraviolet (UV) rays in sunlight [5, 10]. This type of aging occurs within an in-situ mix during its service lifetime in pavement and is accompanied by oxidation that is generated by climate effects (temperature shifts, humidity, water evaporation, among others). This process is called long-term aging and occurs due to oxidation, polymerization, photo-oxidation, thixotropy, syneresis and separation [7, 11-13]. Vallergera et al. [14] describes the mentioned phenomena in the following manner:

- Oxidation, defined as the reaction of oxygen with asphalt binder.
- Volatilization, defined as the evaporation of asphalt's light components. It is primarily defined in function of temperature.
- Polymerization, defined as the combination of molecules in order to form larger ones.
- Thixotropy or steric hardening, defined as the progressive hardening that asphalt binder undergoes due to the formation of an internal structure in the colloidal system, as a product of reorientation of molecules and crystallization of certain chemical components. This process may be reversed up to a certain degree with asphalt reheating and manipulation.
- Syneresis, defined as an exudation reaction of oils or lighter components, which are exuded to the surface of the asphalt layer. With the loss of these oils, asphalt binder begins to harden.
- Separation, defined as the removal of oily components, resins or asphaltenes from the binder, caused by a selective absorption of pores in the aggregate.

Traxler [15], suggested additional factors, including diverse effects of light, water, chemical reactions with aggregates, microbiological deterioration and absorption of heavy asphalt compounds in the surface of aggregates. Petersen [16], reported three important factors related to the composition of asphalt which affect asphalt hardening: molecular structuring - which produces thixotropic effects -, changes in the chemical composition of asphalt molecules and the loss of oily components due to volatility or absorption of pores within the aggregate. According to [17], the mechanisms for in situ oxidation depend on: i) temperature increase, which increases the degree of oxygen diffusion and accelerates chemical reactions; ii) exposure time; iii) the reduction of thickness in the asphalt layer that covers aggregates, which increases the binder's surface exposed to oxygen; and iv) increase of air flow (mainly in porous mixes).

The changes in micro-mechanical, adhesion and chemical properties as a product of aging are more significant in long term than in short term [18-19].

2. PHYSICAL-CHEMICAL PROPERTIES

The first technical document on aging of asphalts was probably reported by [20]. Ever since that time, several researchers have studied the influence of this phenomenon on the physical-mechanical, rheological and chemical properties of asphalt binders and asphalt mixtures. In order to more clearly understand the phenomenon of aging of asphalt binders and asphalt

mixtures, it is necessary to initially know and describe how their chemical composition changes, in order to explain through said changes, how these impact on physical properties.

2.1. Chemical Changes

According to [21], asphalt binder is a material that can be obtained in natural reservoirs or as a derivate of petroleum (either as a residue or a product of extraction through solvents), and its chemical composition mainly depends on its raw source. It is a material that is mainly comprised of carbon (typically, 80–88%) and hydrogen atoms (10–12%), which provide a content of hydrocarbons of around 90%. The other 10% is comprised of heteroatoms (nitrogen: 0-2%; oxygen: 0-2%; sulphur: 0-9%) and metals (vanadium, nickel and iron, present in typical quantities lower than 1%) [22]. Asphalt binder is a colloidal dispersion of asphaltenes (A) in an oily matrix comprised by saturated agents (S), aromatic naphthenes (Ar) and resins (R). S+Ar+R comprise what is called the maltene (M) fraction [3, 23]. A, increases asphalt viscosity, provides it with stiffness and decreases its thermal susceptibility, R provides cementing and adherence characteristics, and oils (S and Ar) provide manageability and aging protection.

During the asphalt aging process, some oily components of the asphalt binder are lost due to volatility or absorption in porous aggregates [13], changes are generated in the chemical composition of asphalt molecules due to the reaction with atmospheric oxygen, where a molecular re-structuring takes place [16, 24-25]. As asphalt binder ages, M decrease and A increase [26-31]. In other words, during the aging process, the ratio of M/A is reduced, which finally results in a more stiff and brittle binder [12, 32]. Additionally, A are the most reactive fraction, with a greater tendency to interact with oxygen [33]. Oxygen eliminates hydrogen atoms from carbon atoms, which causes an increase in the ratio of A and R, as well as a reduction in the quantity of Ar compounds. [34-36]. These processes also lead to an increase in the polarity, acidity and condensation of molecules [23]. In general, it is acknowledged that aromatics transform into resins which in turn transform into A during the aging process, while the S agents remain essentially unchanged, as can be concluded from their low chemical reactivity [24, 37-40]. The relationship between A and R is important because if the first are the components that most grow, R would be in deficit, which would facilitate a greater condensation of A and would accelerate the kinetics of aging [41-42]. According to [26], ratios of A do not considerably change during the mixing process (short term) and increase during the long-term aging process. The ratio of S agents decreases during mixture and changes very little during long term aging. Ar-naphthenes decrease during mixing, but do not change much after this process. They also conclude that anhydrides, carboxylic acids and ketones become A during aging.

Oxidation makes aged asphalts contain a greater quantity of polar molecules and functional groups [3, 23-24, 26, 43]. This generates molecules to lose the capacity to move between each other under thermal or mechanical stress [44], which as a result increases viscosity [23], and brittle, making it more susceptible to cracking [45]. The fractions that comprise asphalt binder may be classified in order of increasing polarity in the following manner: S, Ar (of naphthenes and polars) and A. According to [46], as the asphalt binder reacts with oxygen, the polar functional groups in asphalt molecules increase. Ketones, anhydrides, dicarboxylic and carboxylic acids, which are called carbonyl compounds, and sulfoxide, are the main polar functional groups formed during asphalt oxidation [7, 18, 47-58]. A and R have hetero atoms that are strongly associated to polar functional groups. Therefore, these molecules have interaction and/or association forces, and are highly reactive to electronegative oxygen [45]. During asphalt oxidation, the less polar fractions convert into polar fractions, as functional groups that contain oxygen in asphalt molecules are formed [39,

44]. According to [59], adhesion is directly correlated to non-polar fractions of asphalt binder, while stiffness is correlated to polar fractions.

Kumar and Goetz [60] and Bell and Kliwer [61] mention, that the aging of asphalt mixtures is strongly dependent on the interaction between the asphalt binder and aggregate. Additionally, they point out that the chemical bond that surrounds the aggregate has an influence in aging resistance. With regards to aggregates, the type and content of their minerals, their properties of absorption and the orientation of polar molecules of asphalt in the vicinity of the asphalt-aggregate interface impact the aging process [7]. According to [3, 62], the mineral components on the surface of aggregates can catalyze asphalt oxidation. Curtis et al. [63] mentions, that the surface of aggregates may absorb some of the polar functional groups within the asphalt, which would help to reduce aging. However, the aggregates also absorb oily components in asphalt, which increase the oxidation rate. Barbour et al. [64], carried out electroscopic and infrared tests that showed that the fractions in asphalt binders that contain functional groups with greater polarity display a stronger interaction with the aggregate's surface. Additionally, they reported that the type of mineral in the aggregate impacts the aging process. For example, they reported that limestone interacts in a stronger manner than quartzite with asphalt fractions, and quartzite was a more effective oxidation catalyzer for less polar fractions. Additionally, they reported that carboxylic acids are a main component formed in oxidation, and that this component is strongly absorbed in the surface of limestone. Anderson et al. [34] confirmed that the aggregates with less capacity to absorb polar fractions (such as quartzite and granite) are those that most accelerate asphalt oxidation in comparison to others with greater absorption capacity, such as limestone. A similar conclusion is reported by [65], mentioning that as limestone absorbs polar fractions in greater proportions, it can protect mixes from oxidation. According to [66], the aggregates with most absorption generate greater aging in mixes.

On the other hand, oxidation increases LMS (large molecular size) parameter of asphalt molecules [7, 18, 24, 67-70], and reduces the content of small molecules, increasing molecular weight. [3].

2.1.1. Tests for Evaluating Chemical Properties

In order to evaluate the change that chemical properties of asphalts undergo when they age, several techniques, equipment, and/or tests are utilized. The GPC method (Gel Permeation Chromatography) for example, evaluates the variation of molecular weight distribution of asphalt throughout the aging process [3]. With GPC, LMS (%) is measured. FTIR (Fourier Transform Infrared Spectroscopy) test, is a technique that is generally used in order to detect functional chemical groups present in polymer chains or asphalts. With this test, it is possible to measure carbonyl and sulfoxide functional groups. With EDX (Energy Dispersive X-ray spectroscopy) technique, using a Scanning Electron Microscope (SEM), it is possible to measure the percentage of oxygen in weight, which may be used as an aging indicator of asphalt binders [10]. On the other hand, spectrophotometry is a quantitative and qualitative method for analyzing the content of organic components and is developed based on the colorimetric method. Asphalt binder is a complex organic material and its aging produces chemical changes, therefore, it is possible to characterize the changes of asphalt components through the technique of spectrophotometry and to further describe the effect of aging or the degree of aging in asphalt [30].

2.2. Physical Properties

In general terms, when an asphalt binder ages, it increases its viscosity and stiffness, making it more brittle [10, 13, 18, 26, 40, 53, 70-72], accompanied by the loss of adhesion and cohesion within the asphalt mixture [23, 54, 67, 73-78], which may impact the reduction of its

useful life (decreases fatigue and thermal cracking resistance, moisture damage resistance and tire wear resistance [51, 72, 79-84]). Additionally, as asphalt binder becomes brittle, it is more prone to cracking in contact points between vehicle tire loads and the roadways surface, generating a type of cracking named TDC (Top-Down Cracking) that initiates on the pavement's surface and is propagated within the asphalt layer [85-86].

Generally, in conventional physical characterization tests of asphalt binders, penetration and ductility are diminished, and softening points and viscosity points increase with aging [24, 40, 49, 53, 87-88].

In some cases, a slight aging of asphalt binder may be good, given that it could increase resistance to permanent deformation in high temperature climates [1, 72, 88-89]. The initial increase of viscosity during the first aging stages of asphalt binder is rapid, then said increase is stabilized at a constant speed [47]. This effect has been attributed to the fact that the more volatile parts of asphalt react first and the less reactive groups undergo a longer oxidation reaction [23, 38]. In rheological characterization tests, when asphalt binders ages, the increase of stiffness is coherent with the increments reported in the complex shear modulus (G^*), zero shear viscosity (ZSV), and the reduction of J_{nr} (recoverable creep compliance) and the phase angle (δ) [5, 7, 24, 55-56, 90]. Some authors use the relationship between aged and unaged asphalt as an indicator of evaluating asphalt aging. For example, the CMAI (Complex Modulus Aging Index) parameter, which relates the aged asphalt's G^* to the unaged asphalt's G^* in rheology tests [91]. The higher the CMAI becomes, the greater is the asphalt's aging. For the case of asphalt mixtures, the relationship between resilient modulus of aged and unaged mixes is also usually used as an indicator [66]. During oxidation, the elastic response of asphalt binder increases more rapidly than the viscous response because of the molecule size increase that occurs through the presence of oxygen. In this state, molecules and molecular agglomerates lose enough mobility for flowing [44]. In consequence, the δ decreases and viscosity increases, as depicted in the results of dynamic shear rheometers (DSR).

The effects of aging in the performance of asphalt mixtures varies depending on the type of asphalt binder and aggregate [7, 13, 23, 65], the duration of manufacturing processes, and the mix placement and compaction procedures in construction, along with environmental conditions [92].

As the content of air voids (V_a) in the mix is higher and the thickness of the surface covering of aggregates, the percentage of air voids between mineral aggregates (VMA), and voids filled with asphalt (VFA) is lower, there is a greater probability of aging [12-13, 23, 39, 60, 65, 79, 93-98]. In order to improve aging resistance of asphalt mixtures, Campen et al. [93] recommended a higher thickness in the surface covering of aggregates with asphalt between 6 to 8 μm , Goode and Lufsey [94] of 6 μm , and Kandhal & Chakraborty [95] between 9 to 10 μm . Kemp and Predoehl [79] evidenced that the effect of air voids and porosity of aggregates on the aging of asphalt mixtures is greater when the area temperature increases. According to [6], the effects of aging are more severe in the surface of pavement and diminish exponentially with the depth of the asphalt layer. Li et al. [99], based on field tests where samples of a roadway with 4 years of service were extracted at different depths, concluded that the depth of aging in the asphalt layer is within the first centimeter.

Kemp and Predoehl [79] mention, that the most important factor that affects the rate of hardening of asphalts is high service temperatures. The asphalt's temperature within the mix is usually greater than environment temperature (mainly in summer), given that this material absorbs the heat of solar radiation mainly because of its black color [100]. This inherent nature of the exterior black color of asphalt binder will cause pavement to absorb a considerable amount of solar radiation during the day, which would accelerate the asphalt's

aging rate and would worsen the environmental problem of the heat island effect [55]. In order to diminish the effects of this phenomenon, some studies have used thermal-chromatic materials to place on the pavement, with the purpose of increasing its reflection capacity for solar radiation, and in order to reduce the thermal conductivity of asphalt [55, 100-105]. Additionally, discovered that by using this technology, asphalts may keep pavement relatively warmer during winter. On the other hand, in very cold climates, asphalt binders with high wax content present in the maltene phase may generate stiffness and cracking that can be confused with false asphalt aging [105]. This type of false aging is named physical or steric hardening, there are no changes in chemical properties and its effect is reversible (for example, by applying heat).

During the production of modified asphalts and asphalt mixtures, generally high temperatures are used in mixing and compaction processes. This induces a higher energy consumption and the aging of asphalt binder [106]. Additionally, the aging of modified asphalts is comprised by two relatively independent parts: asphalt binder hardening and polymer degradation [23, 52, 58, 107]. The hardening of asphalt binder could be restored through the adding of rejuvenating agents, while polymer degradation may not be restored. In other words, the aging of modified asphalts ruins the polymer network and deteriorates its engineering properties [108]. In general terms, the oxidation rate is doubled with each 10 °C increase of temperature above 100 °C [109-110]. Storing asphalt mixtures for long periods of time before placement and compaction at the construction may generate binder oxidation. Brown y Scholz [111] for example, report that the storing of asphalt mixtures in a loose state at 135 °C significantly affects the stiffness modulus of compacted mixes. According to them, one hour of storage results in an increase of stiffness between 9% and 24%, compared to mixes that were not stored. Additionally, they conclude that aging caused as a product of storing mixes for around two hours in the laboratory without compaction and at 135 °C, which is representative of aging occurred during mixing, storing and transporting in a real case.

2.2.1. Tests for Evaluating Physical Properties

Just as for the case of evaluation of chemical properties, in order to evaluate the change that the physical properties of asphalts undergo when these age, there are several techniques, equipment and/or tests used. Said techniques generally compare the response of the asphalt material before and after simulating the aging condition. Generally, they use physical aging indexes calculated through traditional tests such as softening point (ASTM D36), penetration (ASTM D5) and ductility (ASTM D-113), among others [73], which are of an empirical nature. These empirical tests have the following main limitations: i) They do not effectively describe the visco-elastic characteristics of asphalt binder; ii) they do not properly correlate with the asphalt mixture performance; iii) they are insufficient in order to characterize the properties of modified asphalts [5]. In addition to these tests, they also carried out viscosity tests by using rotational viscometers (AASHTO T 316 and ASTM D 4402) or DSR. These last ones are also used in order to determine rheological parameters such as G^* and δ , with the purpose of evaluating the performance of asphalt at high and intermediate service temperatures. With DSR it is also possible to determine the resistance of asphalt binder to permanent deformation and fatigue cracking, by carrying out MSCR (Multiple Stress Creep and Recovery, AASHTO T 350) and LAS (Linear Amplitude Sweep, AASHTO TP101) tests, respectively. By carrying out an MSCR test it is possible to determine J_{nr} rheological parameters (nonrecoverable creep compliance, 1/Pa) and R (elastic recovery, %), and LAS determines the number of failure cycles (N_f) that asphalt is able to resist when subjected to a level of shear deformation ($\gamma_{\text{máx}}$, %). Additionally, LAS determines parameters A and B , which are the coefficients that depend on the material's characteristics (the value of A

represents the variation of material integrity in function of accumulated damage and B represents the sensitiveness of material to the level of deformation). They also use Bending Bear Rheometer (BBR) and Direct Tension Test (DDT) with the purpose of characterizing rheological properties of asphalts when they are subjected to low service temperature (mainly evaluates resistance to cracking due to low temperatures). The BBR test determines the static stiffness in time (S), also known as creep-stiffness, and the logarithm of the relaxation modulus (m). DDT determines rupture tension and deformation of asphalt binder when stretched at low temperatures.

Boiling Test (ASTM D3625) is a test for evaluating adherence. In this test, the aggregate is placed in an oven at 105 °C during one hour. Then, the aggregate is submerged in preheated asphalt during 45 s, in order to then be suspended at room temperature for 15 minutes with the purpose of allowing excess asphalt to flow. Finally, the aggregate is submerged in the center of a beaker that contains boiling water. After boiling during 3 minutes, the asphalt film is separated from the aggregate's surface, and it is possible to evaluate the degree of adhesion between the asphalt and aggregate [54].

Atomic Force Microscope (AFM) is another technique used to evaluate materials on a Nano scale. It is a high-resolution scan probe microscope used with a tip that is joined to the free edge of a cantilever that has laser tracking in order to scan the sample's surface. This process results in the interaction of forces between the sample's tip and surfaces. AFM can precisely measure forces on a Nano scale, including mechanical forces, chemical union forces, Van der Waals forces, magnetic and electrostatic forces, and capillary forces [18]. Adhesion properties of asphalt can be evaluated through the total energy required for separating the tip from the asphalt sample [75, 112]. In a nano-indentation test, a penetration tip of an acknowledged elasticity and geometry module for penetrating the asphalt's surface is used and then discharged (generally obtaining a curve of load-displacement). This technique has been recently used for understanding the behavior of time depending creep of visco-elastic materials such as asphalt binders [113].

Thermo Gravimetric Analysis (TGA) is a technique used for evaluating changes of mass in function of temperature. Differential Thermo-Gravimetric (DTG) specifies an obvious point of weight loss. Generally, it is used for describing several characteristics of polymeric materials, such as degradation temperatures, the content of moisture absorbed by materials and the level of organic and inorganic fragments of materials. Differential Scanning Calorimetry (DSC), characterizes the change of ductile-brittle behavior undergo by asphalt binders when aged through the determination of glass transition temperature (T_g).

Optical Polarization Microscope (OPM) is used in order to analyze the morphology of asphalt binders and polymers, and it allows to characterize the nature (continuous phase, discontinuous or disperse phase) of the polymer within the asphalt matrix [97, 114].

According to the surface energy theory, each solid surface has an unsaturated force field which makes its surface to spontaneously attract other materials in order to reduce its free energy [115]. When the asphalt binder and aggregate come into contact with each other, the aggregate spontaneously attracts asphalt molecules in order to reduce the free energy between the aggregate-asphalt, with the purpose of attaining energetic balance of the whole system. This energy exchange allows for asphalt binder to display an adhesion effect with the aggregate [54]. Surface energy theory can be used to calculate the change of adhesion energy, cohesion energy and free Gibbs energy by measuring the contact angle and the surface tension that are then used in order to evaluate adhesion between the asphalt binder and aggregate [116]. The results of Surface Free Energy (SFE) parameters show that aging in asphalt binder causes an increase in the absolute value of free adhesion energy and free cohesion energy, leading to the reduction of adhesion [2].

On some occasions, asphalt mixtures are aged in laboratories, or asphalt cores are extracted from service roadways, and asphalt binder extraction is carried out on these mixes in order to evaluate their properties. There are several methods for extracting asphalt recommended by AASHTO T164 and AASHTO T170. The first one uses a centrifuge for the extraction of asphalt binder in the mix by using trichloroethylene of a reactive degree, or methyl chloride of reactive degree and the latter, uses a set of Abson distillation recovery for the recovery of asphalt binder with properties similar to those present in the asphalt mixture. Another standardized method is EN 12697-3, which uses toluene as a solvent ($C_6H_5-CH_3$) [97].

3. CONCLUSIONS

Based on the reviewed literature, it is possible to conclude the following from a chemical standpoint: processes that cause asphalt binder aging (oxidation, volatilization, photo-oxidation, among others), generate i) a reduction of the maltene fraction and an increase of asphaltenes and resins; ii) aromatics transform into resins, which in turn transform into asphaltenes; iii) polar functional groups increase (mainly the carbonyl and sulfoxide groups), as well as the asphalt's molecular weight. On the other hand, the chemical bonds that surround the aggregate, as well as the type of mineral that comprise it, have an influence in aging resistance.

From a physical standpoint it is possible to conclude that: aged asphalt binder i) increases its viscosity and stiffness; ii) changes its behavior from ductile to brittle; iii) loses cohesion and adhesion with aggregates; iv) reduces fatigue resistance, moisture damage resistance, low temperature cracking resistance and tire wear resistance. On the other hand, mixes are more prone to aging the as Va becomes greater and VMA and VFA parameters decrease, given that under these conditions, generally, the asphalt binder that coats aggregates is thin and more prone for oxidation. Both the asphalt binder and asphalt mixtures are more prone to aging as temperature increases (environment temperature or production process temperature) which it is exposed.

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