

Physical Properties as Related to Asphalt Durability: State of the Art

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ABSTRACT

Asphalt pavements were first constructed in the United States about 1876 with asphalt mined from a lake of native asphalt in Trinidad and fluxed with a petroleum residuum to make an asphalt cement of the desired consistency. As early as 1892, observers became aware that the durability of the asphalt pavements often depended on the asphalt cement in that it must be capable of cementing together the aggregate and it must be elastic and in no way brittle. Domestic asphalt became available around 1900. The engineers and chemists of the time realized that test methods were needed to measure the properties of asphalt and specifications were necessary to prevent the use of inferior or untried materials. Test and evaluation methods that have been used include the following: (a) oven volatilization in thick films, air blowing, and laboratory aging of mixes (tests for evaluating the aged asphalt included penetration, ductility, and softening point); (b) microfilm aging and microviscosity tests; and (c) field aging of pavements and penetration, ductility, and viscosity tests on the recovered asphalt. Of the various methods for testing and evaluating the durability of asphalts, only the thin-film and the rolling thin-film test methods have been adopted as standard by AASHTO and ASTM. Nearly all the states have adopted one of the methods as a means of measuring potential hardening of asphalt during hot-plant mixing. There is a real need for the development of a rational method to predict asphalt durability during service in pavements. The use of tensile and resilient tests should be investigated for this purpose.

The purpose of this paper is to prepare a state-of-the-art report on the use of physical properties of asphalt to evaluate asphalt durability. The literature shows that the first asphalt pavements in the United States were constructed during the 1870s. One of the earliest references concerning asphalt durability was in 1892. The report stated (1,p.25):

The durability of asphalt pavements depends wholly on the suitability of the asphalt for the purpose. It must be of such a nature as to permanently and thoroughly cement together the sand and limestone powder forming the body of the pavement. It must be elastic, independent of the residuum oil required in making the paving cement and in no degree brittle.

The major source of asphalt available at that time was a native asphalt from a natural lake of

asphalt in Trinidad. The asphalt occurs as a solid to semisolid material surface mined from the lake. Excess organic and inorganic material and water were removed and the asphalt was loaded and shipped to points of use. The asphalt was then softened to the desired consistency by adding a fluxing material, which was a residual petroleum product, remaining after the lighter distillate fractions and some heavier distillates had been removed. Early reports showed that the percentage of volatile material in the residuums ranged from 1.4 to 12.3 when the asphalt was heated to 400°F (204°C). Their use as fluxes often resulted in some asphalts that had high losses on heating and rapid and excessive hardening in service.

EARLIEST TESTS

To determine the amount of loss and hardening, A.W. Dow in 1903 proposed two tests to measure the stability (resistance to hardening) of asphalt exposed to high mixing temperatures or during service in pavements (2).

Dow Method 1

A 20-gm sample of asphalt was weighed into a glass retort in an air bath and heated to 400°F (204°C) for 30 hr. The loss in weight was determined and the consistency of the residue measured by the penetration machine, which had been developed by H.C. Bowen in 1888. One specification at that time required that the loss on heating should be less than 8 percent and the penetration of the residue more than 75 percent.

Dow Method 2

A portion of a sand-asphalt mix, in the proportions to be used on the job, was heated in an oven at 300°F (149°C) for 30 min. The asphalt was then extracted from portions of the aged and unaged mixes with carbon disulfide and recovered by distillation. The change in asphalt consistency from heating or aging the mix was determined by the penetrometer. There was no evidence found that Method 2 was used in specifications, but it gave some indication of the relative durability of asphalts supplied at that time. The interesting fact is that the overall procedures developed by Dow before 1900 are similar to extraction and asphalt recovery test methods used today.

The early use of asphalt in pavements was essentially a matter of rule-of-thumb practices. Some mixes resulted in excellent pavements and some were partial or total failures. The chemists and engineers soon realized that specification requirements for asphalts, mixtures, and construction practices were essential to high quality and durability. Thus, during the early 1900s numerous laboratory-accelerated aging tests were used or proposed to assure more uniform and more durable asphalt pavements. The

test conditions for heating varied from 212 to 400°F (100 to 204°C) and the time of heating varied from 30 min to 30 hr. The loss in weight and penetration of the residues were usually determined to measure the effect of aging.

Office of Public Roads and Rural Engineering Method

In 1911 the Office of Public Roads and Rural Engineering of the Department of Agriculture published Bulletin 38, Methods for the Examination of Bituminous Road Materials. Its objective was to present a description of methods used by the Office for the examination of bituminous road materials and in such a form that "with a little practice and proper equipment such examinations may be made by any intelligent person. The various tests have, therefore, been described rather more in detail than would be necessary if they were intended for the use of chemists only, and the illustrations of practically all of the apparatus required have been included." For the record, the authors of the methods were Prevost Hubbard, chemical engineer, and Charles S. Reeve, chemist. Bulletin 38 was revised to show improvements in methods and was published as Bulletin 314 in 1915 (3).

ASTM Methods

In 1903 an ASTM Committee on Road and Paving Materials was formed to develop test methods and specifications for highway materials. Test methods for volatilization, penetration, and bitumen were developed by the Office of Public Roads and were adopted by ASTM in 1911. In the volatilization test a 20-gm sample was heated for 5 hr at 325°F (163°C) in a tin box 6 cm (2.4 in.) in diameter and 2 cm (0.8 in.) deep. The loss in weight was determined and the consistency of the residue was an optional requirement of that time. To provide a greater depth of sample the method was revised to use a 50-gm sample and a tin box 5.5 cm (2.2 in.) in diameter and 3.5 cm (1.4 in.) deep.

For the asphalt cements produced at that time the adoption of the volatilization and penetration tests provided some degree of control of excessive changes during plant mixing that might be reflected in more durable asphalts. The adoption of the method for bitumen was intended to provide a means for identifying Trinidad asphalt by observing the amount and color of the insoluble ash.

Specifications

The early specifications were concerned with only a few sources of asphalt, and requirements often were used just to identify or restrict the use of materials from new sources. With the increase in the use of asphalt in highway construction and with the introduction of asphalts from new unproven sources and refining methods, specifications in force could not be depended on to restrict the use of unsatisfactory materials. Standard specifications that were believed to adequately control the quality and serviceability of the early asphalts were subject to considerable questioning. The consumer was confronted with the problem of revising or adding new requirements, often based on limited data, or developing new methods that would assure improved asphalt durability.

During the 1920s the following three national specifications for asphalt cements were published:

1. Federal specifications, adopted in 1925;
2. AASHTO specifications, adopted in 1924, revised in 1926; and
3. ASTM, specifications, adopted in 1922 to 1926, withdrawn in 1939, and reissued in 1947.

Some of the general requirements of asphalt that were related to use or performance were as follows:

1. The federal specifications stipulated that only those asphalts that had been demonstrated by service tests as satisfactory for the intended use would be accepted. The specifications also indicated the type and location of construction and the relative amount of traffic for each of the grades.
2. The AASHTO specifications indicated that the use of each grade depended on the type of road, climate, and traffic.
3. ASTM suggested the type of construction for which each grade would be used.

With minor exceptions the requirements for physical and chemical properties were essentially the same for the three national specifications. The specification tests included:

- Penetration [77°F (25°C)],
- Flash point [°F (°C)],
- Ductility [77°F (25°C)], 5 cm per min,
- Loss at 325°F (163°C), percent,
- Penetration of residue, percent of original,
- Bitumen, solubility in CS₂,
- Bitumen, solubility in CCL₄.

The federal specifications included requirements for specific gravity, 77°/77°F (25°/25°C) and softening point, °F (°C).

The only requirements in the three specifications that might be indicative of durability were the percentage of loss on heating and penetration of the residue. However, based on a survey of asphalt production in the United States in 1935 the results of percentage of loss and of original penetration were of little value to predict the resistance to change in heating during plant mixing or during service in the pavement.

STATE OF THE ART IN ASPHALT DURABILITY

The primary objective of this paper is to develop a state-of-the-art report of the use of physical methods to evaluate asphalt durability. The scope of the paper will be limited to test methods and properties of asphalt that are directly or indirectly related to durability and will essentially disregard the influence of aggregates and mix properties, construction practices, environment, and quality assurance. For example, the literature is replete with data showing the influence of air void and asphalt content on asphalt aging in service and their effect on durability.

During the 1930s the then Highway Research Board organized a committee to investigate the relation of asphalt properties to pavement durability. One of the objectives was to acquire information on which to base the need for more rational specifications that were related to pavement performance. To define the problem, the committee collected and studied information on the causes of failures and the rea-

sons for good service behavior. A summary of 91 replies to a questionnaire brought out these facts:

1. Failure or unsatisfactory performance from the use of poor-quality asphalt occurred in all sections of the country.
2. Cracked asphalts appeared to be more troublesome than uncracked asphalts. (Cracked asphalts were produced from petroleum by high-temperature and high-pressure refining methods.)
3. Among the asphalt cements used, the harder grades appeared to be the chief source of unsatisfactory pavement performance.
4. Age hardening and increased brittleness of the asphalts were found to be the changes that most frequently produced the most unfavorable performance.
5. Cracking and raveling appeared to be the principal types of distress attributed to poor-quality asphalts.

The HRB activity as well as the general concern that more information was needed to develop performance-type specifications prompted an accelerated research and development program. Beginning in the 1930s a large number of studies were initiated to develop methods to measure or predict the durability of asphalt cements. One of the most important breakthroughs was the development of a method by Gene Abson in 1933 for recovering the asphalt from mixtures immediately after plant mixing or after periods of extended service in the pavement. Since its development, the recovery test has been used extensively in studies to determine change in asphalts and to relate the changes to asphalt durability. In the following part of this paper the information on a number of research studies or investigations that were conducted to develop physical tests to measure and evaluate durability characteristics of asphalt cements will be summarized.

Numerous attempts were made during the period between the 1900s and the 1980s to devise or develop test methods or procedures that would measure and predict asphalt durability. A review of the literature shows that the studies could be classified as follows.

Laboratory-Accelerated Tests and Evaluation Methods

In these studies asphalt aging was determined by oven volatilization, by air blowing at high temperatures, or by aging asphalt mixtures and testing the recovered asphalt. In most cases the effects of the aging procedures were evaluated by penetration and ductility. In some instances the changes in asphalt were measured by physical tests on compacted mix specimens. Table 1 gives the data reported, the investigator, the test, and the evaluation method used. The time and temperature of the various tests varied appreciably. Most of the evaluation methods compare the rate of change in properties.

Of the tests listed in Table 1 only the 1/8-in. thin-film oven (TFO) test has been adopted as a standard method for evaluating asphalt durability, particularly from the standpoint of hardening during hot-plant mixing. Extensive studies were made to select the test and specifications before its adoption. Some of the studies using mixtures and recovered asphalt were found to be too complex for routine control.

Laboratory Microfilm Tests and Evaluation Methods

During the development of tests to measure and predict durability there was considerable concern with

TABLE 1 Laboratory Accelerated Test and Evaluation Methods to Determine Asphalt Durability

Date	Investigator	Test Method	Evaluation Method
1903	Dow (2)	30 hr, 400°F	Change in weight penetration of residue
1903	Dow (2)	Mixture aged for 30 min, 300°F	Recovered asphalt, change in penetration
1937	Nicholson (4)	Air blowing, 15 min 425°F	Penetration, ductility
1937	Rashig and Doyle (5)	Air blowing, 15 min, 400°F	Change in penetration
1937	Hubbard and Gollumb (6)	Mixture, time, and temperature varied	Recovered asphalt, change in penetration
1939	Lang and Thomas (7)	Mixture, oven aging, outdoor exposure	Change in mix properties, abrasion, strength, etc.
1940	Shattuck (8)	Mixture, oven aging 30 min, 325°F	Recovered asphalt, penetration, ductility, soft point
1940	Lewis and Welborn (9)	1/8-in. film oven test 5 hr, 325°F (TFOT)	Change in weight, penetration, ductility
1946	Lewis and Halstead (10)	1/8-in. film oven test, 5 hr, 325°F (TFOT)	Change in weight, penetration, ductility
1952	Pauls and Welborn (11)	Mixture, oven aging, 325°F (TFOT)	Compressive strength, recovered asphalt, TFOT residues

Note: $t^{\circ}\text{F} = (t^{\circ}\text{C} + 0.55) + 32$. TFOT = thin-film oven test.

testing the asphalts in relatively thick films such as those used in the TFO test and air-blowing tests listed in Table 1. Thus, beginning in the 1950s several studies were made to develop testing apparatus and to age and test asphalts in microfilm thicknesses.

Several modifications of the microfilm test are shown in Table 2. In general the studies have indicated that the microfilm tests can be used to measure and predict hardening of asphalts under laboratory and field conditions.

TABLE 2 Laboratory Microfilm and Evaluation Methods to Determine Asphalt Durability

Date	Investigator	Test Method	Evaluation Method
1955	Griffin et al. (12)	Shell microfilm test, 5-micron film, 2 hr, 225°F	Viscosity before and after aging, aging index
1958	Heithaus and Johnson (13)	Road tests, laboratory aging, microfilm test	Recovered asphalts, microfilm aging index
1961	Traxler (14)	TFOT and microfilm, 15-micron film, 2 hr, 225°F	Microviscosity at 77°F compared
1961	Halstead and Zenewitz (15)	TFOT and 15-micron film, 2 hr, 225°F	Microviscosity at 77°F compared
1963	Hveem et al. (16)	Shell microfilm test, modified; 20-micron film, 24 hr at 210°F	Microviscosity at 77°F before and after aging
1963	Hveem et al. (17)	RTFOT and TFOT	Viscosities of RTFOT, TFOT, and recovered asphalts compared
1969	Schmidt and Santucci (18)	RTFOT, 20-micron film, 210°F	Microviscosity of residue
1981	Kemp and Predoehl (19)	Tilt-oven durability test, 168 hr, 235°F	Penetration, 77°F, ductility, 77°F

Note: $t^{\circ}\text{F} = (t^{\circ}\text{C} + 0.55) + 32$. TFOT = thin-film oven test, RTFOT = rolling thin-film oven test.

In line with the effort to develop a test that would age asphalts in a film thinner than that in the 1/8-in. TFO test, California developed a rolling thin-film oven (RTFO) test. The test procedure involves heating a 35-gm (± 0.5 -gm) sample of asphalt at 325°F (163°C) in a special bottle that rotates, causing the asphalt to flow and expose a new surface during rotation. Tests have shown that the hardening effects are comparable to the hardening in the TFO

test. The RTFO test has been incorporated into the California specifications and has been adopted by ASTM as a standard.

There have been several modifications of the RTFO test using the thin-film bottle. Some studies have been made to give a thinner film of asphalt in the bottle by first dissolving a specified amount of asphalt in benzene or other solvents, coating the inside of the bottle, and evaporating the solvent during rotation of the bottle. The microfilm is then exposed in the oven at 210°F (99°C) for 48 hr and the change in consistency is measured by viscosity.

More recently, the RTFO test has been modified to heat the bottle with the oven slightly tilted to prevent asphalt build-up. Studies indicate that the tilt-oven asphalt durability test could be used to predict asphalt hardening in hot climates.

Studies by Traxler (14), and by Halstead and Zenewitz (15) compared the aging in 15-micron (0.0006-in.) films heated for 2 hr at 225°F (107°C) with aging by the TFO test. Traxler concluded that for six asphalts, the hardening in a 15-micron film for 2 hr at 225°F (107°C) and the hardening measured by viscosity at 77°F (25°C) on the residue from the TFO test were of the same order.

Halstead and Zenewitz also reported on the hardening measured by an aging index for the 15-micron film and for the residue from the TFO test. All viscosities were determined by a sliding plate viscometer, which is a standard ASTM method, at 77°F (25°C) at a shear rate of 0.05 per sec. For low aging indexes (low hardening rate), there was no significant difference in the results of the two tests. As the resistance to hardening decreased, the increase in aging index for the microfilm residue increased much more rapidly than for the TFO test residue. The authors showed that there was a straight-line correlation between the aging index of

the TFO test residue and the percentage of original penetration.

Field Test and Evaluation Methods to Determine Asphalt Durability

Another source of information for evaluating the durability of asphalt is the correlation of pavement condition with physical properties of the recovered asphalt. The development of the Abson method for recovering asphalt from paving mixtures prompted many of the studies.

Table 3 presents the source of information, the number of pavements or test sections included in each study, and critical properties of the recovered asphalt for most of the studies reported from 1937 to 1975.

The literature search shows that early asphalt pavement construction often used asphalts in the 40 to 60 penetration range. The initial asphalt hardness together with the hardening during hot-plant mixing and in service probably contributed to the cracking problems shown in Table 3.

In 1979 an NCHRP study, Relationship of Asphalt Cement Properties to Pavement Durability (31), concluded that the hardness of asphalt in the pavement is the one property most closely associated with pavement performance and depends on

1. The initial consistency of the asphalt,
2. The susceptibility of the asphalt to hardening during plant mixing, and
3. The rate of hardening of the asphalt in the pavement.

To develop a performance-related specification for asphalt pavement construction, the durability factor of the asphalt must be considered.

TABLE 3 Field Test and Evaluation Methods To Determine Asphalt Durability

Date	Investigator	No. and Location of Pavement	Condition	Recovered Asphalt	
				Penetration at 77°F	Ductility at 77°F
1937	Radar (20)	8, Michigan	Good	34	15
			Poor	13	3.2
1937	Powers (21)	12, Arizona	Good	10+	10+
			Cracked	10-	10-
1937	Rashig and Doyle (5)	30, various states	Good	25+	15+
			Poor	25-	15-
1937	Hubbard and Gollumb (6)	19, five states	Sound	30+	—
			Prone to crack	30-	—
			Cracked	20-	—
1939	Miller et al. (22)	40, various cities	Sound	25+	24+
			Prone to crack	18-25	4-24
			Cracked	18-	4-
1940	Shattuck (8)	33, Michigan	Very good	20+	50+
			Good	20+	25+
			Cracked	20-	25-
1959	Parr and Serafin (23)	6 sections, Michigan	No difference in performance after 4 years		
1967	Serafin et al. (24)	Same as above	No difference in performance after 12 years		
1959	Simpson et al. (25)	20 sections, Zaca Wigmore, California	Fatigue cracking	Low ^a	Low
1971	Vallerga and Halstead (26)	53, 19 states	Variable distress	Variable	Variable
1975	Zenewitz and Welborn (27)	54, 19 states	Variable distress	Variable	Variable
1967	Chipperfield and Welch (28)	8 asphalts, France; 8 asphalts, Germany; 124 test sections each	No difference in performance		
1970	Chipperfield et al. (29)	Same as above	No significant difference in pavement performance attributed to asphalt		
1968	Cisco and Brunstrum (30)	12, 12 states	Some pavements cracked	Increase in complex modulus	

^aLow viscosity.

SUMMARY

This development of the state of the art of physical properties as related to asphalt durability shows that there has been great concern with asphalt durability almost since the beginning of asphalt pavement construction. The engineers and chemists have searched for test methods that would predict asphalt durability with a high probability of correlation with pavement performance. They usually were aware that an evaluation method for asphalt durability per se can be negated by external variables resulting from mixture design, including asphalt grade and aggregate type, construction practices, environment, traffic, and quality assurance.

More than 40 years ago the TFO test and later the RTFO test were developed and subsequently adopted by AASHTO, ASTM, and almost all state highway departments and other governmental agencies as specification tests. The significance section of the AASHTO and ASTM standards states, "This method indicates approximate change in properties of asphalt during conventional hot-mixing at about 302 degrees F. (150 degrees C) as indicated by viscosity, penetration or ductility measurements. It yields a residue which approximates the asphalt condition as incorporated in the pavement. If the mixing temperature differs appreciably from the 302 degrees F level, more or less effect on properties will occur."

The TFO tests have served to screen out or predict the behavior of those asphalts that showed excessive change in physical properties when subjected to the high temperatures encountered in hot-plant mix construction. Although the tests predict the properties of the asphalt at the time of construction, they do not provide adequate information on change in properties during service in the pavement. In order to develop performance-related specifications for asphalt pavement, such information is essential. To accomplish this, the following approaches should be investigated:

1. Evaluation of the present TFO tests to determine whether modifications are necessary to conform with current asphalts and construction practices,
2. Investigation of current or new methods that can be used to predict long-term durability of asphalts in pavements, and
3. Investigation of the use of tensile strength and resilient-modulus properties of asphalt paving mixtures to predict asphalt durability during long-term exposure in pavements.

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Publication of this paper sponsored by Committee on Characteristics of Bituminous Materials.

Significant Studies on Asphalt Durability: California Experience

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ABSTRACT

Some of the key durability studies conducted by the California Department of Transportation since the late 1930s, the conclusions reached, and how the results have been implemented in asphalt specifications are reviewed. A description is given of a recent study in which carefully prepared laboratory briquettes of three divergent types of asphalt and absorptive nonabsorptive aggregates were used. The mixes were compacted using three different void contents and weathered in the field in four different climatic areas. New asphalt tests and specification requirements that will be used in obtaining asphalt for test roads in hot desert areas are described. A summary of the major findings from these studies is also included.

PREVIOUS ASPHALT DURABILITY STUDIES

Durability studies were under way in the late 1930s. Using the criterion that asphalts hardened under field conditions at the slowest rate were the most durable, an attempt was made to evaluate asphalt

products available in California (1). This required new and different test methods because the specifications then in use did not separate hardening during construction operations from the natural rate of hardening during the service life of the pavement under traffic. A test investigated during this early period was the asphalt classification by the Benson method of thin-film studies on microscope slides (2). This study led to the belief that thin films were essential for a durability determination. However, the Benson test did not lend itself well to specification purposes.

In 1939 Lang and Thomas of Minnesota (3) published their durability work using mixes of Ottawa sand and asphalt. They explored three methods of testing these samples:

1. Elongation of a test briquette,
2. Destruction by impact, and
3. An abrasion method that tumbled the briquettes in the Devel testing machine.

From these studies, an abrasion test evolved--the shot abrasion test--that used Ottawa sand and 2 percent asphalt mixtures prepared at 230°F and compacted into the lids of 3-oz ointment tins. The compacted specimens were cured under drying lamps in a special weathering machine in which the temperature was maintained at 140°F. The briquettes were then abraded by a stream of free-falling shot dropped from a prescribed height. The weight loss