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Hardening of asphalt in uncompacted bituminous mixes

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HARDENING OF ASPHALT IN UNCOMPACTED
BITUMINOUS MIXES

by

Bachitter Singh Bassi

A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of
The Requirements for the Degree of
DOCTOR OF PHILOSOPHY

Major Subject: Highway Engineering

Approved:

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1960

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INTRODUCTION

Bitumen, according to the Association Internationale Permanente des Congres de la route in Paris, is a mixture of hydrocarbons either of natural or of pyrogenous origin or a combination of both (frequently accompanied by their non-metallic derivatives) which can be gaseous, liquid, semi-solid or solid and which are completely soluble in carbon disulphide.

Bituminous materials cover a large number of paving and non-paving materials. These materials are distributed in nature in various forms such as natural gas, petroleum, maltha, asphalt, coal, and rock asphalts. Asphalts from petroleum crudes, natural asphalts from Bermudez and Trinidad lakes and tars from coal are useful for highway paving purposes. Petroleum asphalts, however, are used more extensively than all other bituminous materials in highway construction.

Although bituminous materials have been known to man from very early times, their use in road construction is comparatively recent. It was in 1838 that these materials were used for the first time in the United States for sidewalks. In 1870 an experimental road was constructed in Newark, New Jersey. Success of this road resulted in the granting of a

patent to N. B. Abbott for laying asphalt pavement in 1871. Trinidad asphalt used in Washington, D. C., in 1876 was authorized by an act of Congress passed the same year.

However some bituminous materials called "rock asphalt mastics" were sold in France in the 1790's and used for surfacing floors, bridges and sidewalks, also to a limited extent for waterproofing purposes.

In the early part of this century, petroleum asphalts for road construction were obtained from very few established sources. The boom in the motor vehicle industry affected both the use as well as manufacture of asphalts. Thus asphalts which once were obtained from few sources are now available from many sources and by a variety of refining methods.

Development of motor vehicle, demanded good all-weather roads connecting almost all cities and rural areas. This resulted in the development of an extensive rural and urban highway system. Asphalt therefore, which once was used almost exclusively in high type pavements for city streets prior to the motor vehicle age, was developed for the construction of both rural and urban highways. In recent years the consumption of asphaltic materials for paving purposes have increased greatly in the United States. In 1919, only

half a million tons were used, whereas in 1958 asphalt consumption rose to 15 million tons and is showing a steady increase every year (9).

Availability of funds and equipment for paving modern highways, the low cost of construction and the smooth riding surface characteristics provided by asphalt roads, have made imperative the development of better means and methods of testing the quality of asphalt and asphaltic mixtures.

Asphalt roads have been looked upon with favor by many authorities in this country and abroad, due to their low cost of construction and maintenance. The asphalt industry, many asphalt technologists, and road or highway departments are therefore engaged in improving the quality of asphalt pavements every day.

Since asphalt is a chemically complex material, which varies with source of the petroleum crude and the refining process employed, its constituent and chemical composition is as yet not clearly nor completely understood. Petroleum crudes from different sources differ considerably in composition. Consequently, the asphalts secured from these crudes will also differ in many respects. Since the chemical composition of asphalts is as yet not fully understood, the engineer must rely entirely upon the physical tests for asphalts.

Asphalt when mixed with mineral aggregates under specified conditions yields a paving mixture. Since the properties of the ingredients of paving mixtures cannot be determined accurately, the physical tests of asphalt, mineral aggregate and paving mixture, along with the service behavior of existing pavements, are the only guides for paving engineer.

Laboratory investigations are made on asphalt, mineral aggregate and asphaltic mixtures before the actual pavement is laid. Due to the unlimited number of variables found in the field, pavements laid with similar laboratory approved asphaltic concrete mixes might react differently to natural field conditions. In other words, natural weathering plays a vital role in the service behavior of a highway. Many asphalt pavements have shown cracking after a few years of service, but others constructed with almost similar materials stand up perfectly even after many years of service, under different field conditions.

Studies of the asphalt recovered from pavements show hardening of asphalt in pavements. This hardening of asphalt is probably one of the causes of cracking or of other pavement failures.

To learn a little more about what occurs to the asphalt, the aggregate and to the asphalt-aggregate mixtures when the latter is subjected to severe laboratory treatments simulating field conditions, this investigation was undertaken.

REVIEW OF PREVIOUS WORK

Types of Asphalt Hardening

Asphalt generally increases in consistency with time, even at moderate temperatures. This increase in consistency is commonly referred to as "hardening". Asphalt hardening may either be reversible or irreversible or a combination of both (14, 49).

Reversible hardening

Reversible hardening may occur isothermally, but it does so without material loss and chemical change. This results from internal physical re-orientation and re-organization at the atomic, molecular and micelle levels of the components of asphalt. Reversible hardening could further be subdivided into two classes (14);

- (a) Strain hardening
- (b) Steric hardening.

Strain hardening results from internal re-orientation caused by continuing deformation or strain. When the straining is stopped, the material reverts to its original consistency by relaxation.

Steric hardening also results from re-orientation but takes place without dimensional deformation. This takes place at constant temperature in high consistency asphalts, which

have been quickly chilled from a heated condition and it increases with time to a limiting value.

Irreversible hardening

Irreversible hardening involves changes such as loss of material and chemical changes. Examples of hardening by loss of material are distillation of crude to bottoms for increasing softening points and evaporation of diluent from cut-back asphalts on a road bed. Air blowing of residuum and the oxidation of asphalts when weathering are examples of chemical changes.

Factors Affecting Asphalt Hardening

The following factors could contribute to the hardening of paving asphalts (1, 18, 20, 68, 69, 70):

- (a) Oxidation
- (b) Volatilization
- (c) Polymerization
- (d) Thixotropy
- (e) Syneresis
- (f) Separation
- (g) Carbonization
- (h) Effect of moisture.

Oxidation In asphalt technology, oxidation means the reaction of oxygen with asphalt. The rate of oxidation depends upon the character of the asphalt and such reaction conditions as temperature, among others. According to Labout (35) at high temperatures, such as exist in a hot mix plant, oxygen may react readily with asphalt and bring about dehy-

drogenation of asphalt, resulting in the formation of asphaltenes. The rate at which oxygen reacts with asphalt at low temperatures is slow and the oxygen is primarily absorbed by the asphalt. Both of these processes result in the hardening of asphalt but to different degrees. Some researchers believe that oxidation is the primary cause of hardening of asphalt during mixing and laying processes and in the pavement thereafter.

Volatilization Asphalt is a mixture of hydrocarbons with a wide range in molecular weight. Volatilization is the evaporation of lighter constituents of asphalt (18, 66). The degree to which volatilization progresses in any given asphalt depends to a certain extent on its grade, source, refining process, and other atmospheric conditions. Higher temperature, that is when heated alone or during mixing of the asphalt with heated aggregates, accelerate volatilization (18, 28).

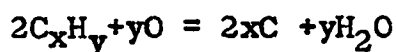
Polymerization Polymerization refers to the process in which two or more molecules of the same kind combine with one another, resulting in the formation of higher molecular weight compounds. In asphalt the lower molecular weight hydrocarbons combine to form larger molecular weight hydrocarbons. This might result in progressive hardening, as the lighter molecular weight constituents give asphalts their fluidity.

Thixotropy Thixotropy is considered as the progressive hardening due to the formation of a structure within the asphalt over a period of time. This being a temporary hardening it can be eliminated to a certain degree by re-heating the asphalt. The most likely place for hardening of asphalt by thixotropy is areas where the pavement is subjected to little or no traffic and thereby enabling the structure to form within the asphalt (1, 12).

Syneresis According to Oliensis, (48) syneresis is an exudation reaction taking place in asphalt in which, due to the formation of the structure within the asphalt, a thin oily liquid containing either dispersed or dissolved intermediate and heavier bodies is exuded to the surface. The loss of this oily liquid results in the hardening of asphalt. This phenomenon is more common with blown asphalts, but it may occur in paving asphalts.

Separation Separation is described as either the removal of asphaltenes or petroleues or resins from the asphalt. This generally results by the selective absorptions of some porous aggregates, on which an asphalt film has been placed (37). Such an action may result in the hardening or softening of the asphalt, depending which of the constituents is removed from the asphalt film.

Carbonization Carbonization is the formation of free carbon in the bituminous materials and is induced by an extensive elimination of hydrogen (1). This can be represented by a general reaction;



This reaction progresses most rapidly in sun light, but will similarly take place when bituminous substances are subjected to higher temperatures.

Effect of moisture All bituminous substances are more or less affected upon exposure to moisture, which manifests itself in two ways, namely by the actual absorption of water and by the gradual leaching out of soluble constituents. These actions become intensified when they are in oxidized form, as oxygenated substances seem to have a greater affinity for moisture than the hydrocarbons themselves. It has been found that asphaltic materials in the presence of light and oxygen are gradually converted into water soluble products containing acid and ketone bodies. Presence of mineral matter, having great affinity for water, serves to hasten the destruction of bituminous substances (1).

Service Behavior of Asphaltic Materials in Pavement

The service behavior of bituminous materials in pavement is not fully understood, mainly because there is no direct way

of evaluating this property. Information about service behavior of asphaltic materials in pavements is obtained by:

- (1) Frequent visual inspection of the pavements.
- (2) Obtaining pavement cores after certain periods of service and testing for density and stability.
- (3) Recovering asphalt from cores taken from severely cracked, slightly cracked and pavements in excellent condition and studying the changes in physical and chemical characteristics of these asphalts.

It can be improved by:

- (1) Developing new physical and chemical tests which will predict within reasonable accuracy, the durability of bituminous materials in pavements.
- (2) Development and better understanding of laboratory accelerated weathering tests which will more or less simulate field conditions.

In general, asphalt pavements can fail by cracking. Causes of pavement failures have been studied by many investigators in the past (13, 25, 29, 33, 42, 54). Severe hardening of asphalt is concluded as one of the causes of pavement failure. Asphalts recovered from pavements laid many, many years ago, show that penetration value approaches 30 asymptotically on penetration versus pavement life curve. It is concluded therefore, that pavement will start cracking and

consequently fail when the penetration value will reach 30 or below (49). On the contrary some pavements having asphalt penetration as low as 20, are found in fairly good condition (62). The total percent loss and the rate of loss of penetration in pavements depends upon the type of asphalt and aggregates used and many other field conditions. In general all the asphalts recovered from the cracked pavements show a loss of 60 to 70 percent in penetration (41). Hardening of binder in pavements and their relation to service behavior have also been investigated by many other investigators (36, 43, 55, 71, 72).

Methods of Asphalt Extraction and Recovery from Asphalt-Aggregate Mixtures

In evaluating the service behavior of paving asphalt, the test sample must be retrieved from asphalt-aggregate mixtures. In order to achieve this purpose, however, it is important that the properties of the asphalt be changed as little as possible, during the extraction and recovery processes.

Extraction methods

The binder (asphalt in this case) can be extracted from the asphalt-aggregate mixture by dissolving it in any suitable solvent. The most commonly used solvents are benzene, carbon disulphide, carbon tetrachloride, chloroform and cyclohexane.

Asphalt from very small samples of sheet asphalt or fine graded mixtures is extracted by soxhlet type apparatus. Materials to be extracted are held in the paper or alundum thimbles (7). Comparatively larger samples can be extracted in New York extractor and Colorado extractors, which employ similar procedure. These methods though simple are most time consuming. During development of methods for extracting larger samples of sheet asphalt and asphaltic concrete, the mixtures were soaked in large quantities of solvent for a few hours. The asphalt solution was separated from aggregate by decantation. This solution was allowed to stand undisturbed for a few days and decanted, thereby removing the sedimented fine mineral matter (11). Such a procedure was used until the A.A.S.H.O. approved the centrifugal method. Similar or modified extraction techniques as described by Siegman and Chalk using carbon disulphide, Suida and Hoffmann using benzene and by Kamptner using a variety of solvents are reviewed by Ford and Arabian (24). In A.A.S.H.O. a centrifugal extractor method materials up to 1000 grams can be easily used. In this method mineral aggregate is retained inside the centrifuge and the asphalt solution is collected outside the centrifuge assembly (6). A.A.S.H.O. method is good when materials up to 1000 grams are used, for large quantities (5000-6000 grams). Wood (74) has described a method which gives uniform results. Many other modified methods are used by various

highway departments and research laboratories.

Almost all methods other than A.A.S.H.O. method, for the extraction of binder from the asphalt-aggregate mixture use a similar operation to that of soxhlet extraction or other reflux type extraction apparatus. The heated solvent vaporizes, and then condenses and falls back upon the mixture and then percolates through it dissolving out the asphalt.

The asphalt solution extracted from the mixture by such methods contains a small percentage of colloidal mineral matter. Presence of mineral matter in asphalt recovered from such a solution, materially effects the asphalt properties. The solution is therefore, further centrifuged to remove the colloidal mineral matter.

Recovery methods

Many methods have been tried for recovering asphalts, from products in which they are used, in the hope of recovering the asphalts without appreciable alteration in their physical and chemical properties. Almost all the methods use simple distillation procedure to remove the solvent from asphalt solution. Most of the methods used earlier for asphalt recovery were not satisfactory as the solvent was not completely removed at the specified distillation temperatures. In order that a recovery method be accepted universally it should have:

(a) Application over a wide range of asphaltic products, since in most cases no prior knowledge is available concerning the type or grade of materials originally used.

(b) The solvent should be completely removed without causing any hardening or polymerization by high temperature or prolonged time of heating, and that no other significant or material alteration in physical and chemical properties take place during the recovery operation.

Abson (2) in 1933 proposed a method for the recovery of asphalt from asphalt solution. Benzene was used as a solvent. This method employs simple distillation procedure at 300-325°F to remove a large part of the solvent. The remaining solvent is removed by bubbling CO₂ through the asphalt. Reproducible and uniform results were obtained with this method for many asphalts. The validity of this method was further investigated by Abson and many others (3, 4, 5, 54, 55, 59, 65). The revised procedure and apparatus was accepted by ASTM (7). Bussow (15) proposed another method in which last traces of solvent (benzene) are removed by raising the distillation temperature from 325°F to 572°F in a specified time, thereby eliminating the use of any inert gas. Pfrengle's method (53) employs chloroform as a solvent, and removes the solvent by passing CO₂ over the constantly stirred solution. Many other investigators have proposed methods for asphalt recovery using heat, vacuum, or some inert gas or their combination to

remove last traces of the solvent (4, 11, 24, 27).

It is very seldom that any two asphalts from different sources will react in a like manner to the same heat treatment or be affected by the solvent used to the same extent. Certainly therefore, absolute accuracy of any method for all asphalts is not possible. The details of the method used for both extracting and recovering asphalt from the asphaltic concrete mixtures for this study are given in the Materials and Method section.

Role of Aggregate in Asphalt Hardening

The asphalt, theoretically, should not undergo any change (physical or chemical) when mixed with aggregate provided the aggregate behaves as an inert material. Mineral aggregates used for highway construction show a wide range in physical and chemical properties. Aggregates, therefore, can not be considered as inert matter. Based upon their affinity for water they are classified as hydrophobic (water hating) and hydrophilic (water loving). Absorption of water by aggregates was considered as a guide for estimating binder absorption by aggregates. Goshorn and William (26) and Nevitt and Krchma (46) made aggregate absorption studies using SC-6, 85-100 penetration grade and SC-4 asphalts respectively. Further studies on aggregate absorption show that water absorption is no criteria for binder absorption (37).

The physical mechanism by which absorption takes place has been shown by microscopy, to be a passage of liquid through the intercrystalline channels of the aggregate. More-absorption is related to the nature of these intercrystalline channels.

Aggregate absorption using kerosene and other heavy oils have also been investigated (22, 31). Hveem (31) developed a method for determining the kerosene oil absorption by aggregate. This method is commonly known as centrifuge kerosene equivalent (CKE). CKE along with few other physical tests on aggregates has been used by California Highway Commission for determining optimum asphalt content for asphaltic concrete mixes.

Certain aggregates absorb asphalt rapidly under mixing conditions. A phenomenon generally referred to as "Burning" is often observed when a highly absorptive aggregate is mixed with asphalt (37). This results in the dull brown appearance or dryness of the mixture. However, other non-absorptive aggregates with same asphalt content when mixed under similar conditions result in much better mixtures. Burning is caused by the absorption of the binder during mixing.

It is believed that this absorption of asphalt by aggregate is not complete asphalt absorption but it is more or less selective type absorption. In this type of absorption,

some asphalt constituents (asphaltenes or petrolenes or resins) are absorbed more preferentially than the others. The removal of some constituents particularly the lighter ones from asphalt results in hardening of asphalt, and is very severe during mixing time in hot mix plant. It is considered that loss in penetration that occurs during mixing and laying operation may be greater than the loss occurring in the pavement over a period of as much as ten years after construction (49).

Tests for Asphalt, Aggregate and Asphaltic Mixtures

A large number of tests for studying the asphalts, aggregates and their mixtures have been proposed to AASHTO and ASTM. The accepted tests by these organizations are used as standard tests (6, 7). Specification limits for asphalt paving materials are based upon these tests. The objective of these standard tests vary from classification to durability of these materials and their mixtures.

Routine tests for asphalts

Routine tests for asphalt cement are specific gravity, penetration, flash and fire points, softening point, loss on heat, total bitumen content, ductility, spot test, viscosity test and chemical composition (per cent asphaltenes). These tests aid in the classification of asphalts, predicting their durability in pavements and furnish precautionary information

needed for their handling.

Over and above these tests, many indices, such as penetration index, deterioration index, and factors like the penetration temperature susceptibility factor and characterizing factor etc. have been developed (34, 39, 50, 51, 52). They are useful in understanding the changes that take place in asphalts. Pfeiffer (52) has even used penetration index for classifying asphalts.

Routine tests for aggregate

In almost all highway laboratories, the tests conducted on aggregates are gradation, specific gravity, unit weight, percent voids, Los Angeles abrasion test, absorption of water or other oils (kerosene) and chemical composition. Similar tests are run on the aggregate recovered from test cores taken from pavements, to study the actual de-gradation that has taken place under traffic during a certain period.

Tests for asphaltic concrete mixtures

Tests conducted on specimens compacted from asphaltic concrete mixtures are specific gravity, density, per cent voids, stability and cohesion. There are many methods of testing stability. Neppe (45) and Stevens (67) have tabulated all the tests for measuring the stability of compacted samples. Asphaltic concrete mixtures are tested by preparing specimens of a standard size and compacted in a manner pre-

scribed by the specific test procedure. Although many uncertainties exist in all these tests and their interpretation, these tests along with the service behavior of the old highways are the best guide a construction engineer has. Vokac (71), while emphasizing the sensitivity of the compression test, its relation to service behavior and its usefulness as a means for mixture control and design, explained the lack of significance of certain other physical characteristics measured by many other stability tests. Opinions differ from person to person, regarding the validity of stability tests, as a measure of actual service behavior of asphalt pavements.

Accelerated Laboratory Weathering Tests

Hardening of asphalt continues with time even at normal temperatures in pavements. Asphalts recovered from pavements laid many years ago have shown this effect. The specifications for asphalts used in highway construction appear to be too flexible. Moreover none of the standard tests for asphalts and asphaltic mixtures represent any well established relationship with service behavior (10, 19, 71). Many people have tried one way or the other to study asphalts and asphaltic mixtures in the laboratory by subjecting them to conditions which might simulate field conditions. Although it is impossible to develop natural field (traffic and weather) conditions in laboratory, certain efforts are being made to ap-

proach such conditions. Following are some of the accelerated laboratory weathering tests run on asphalt and asphaltic concrete mixtures.

Thin film oven test for asphalts

The standard loss on heat test does not furnish adequate information concerning the probable behavior of asphalt in asphaltic concrete pavements. The Bureau of Public Roads has modified this test and named it "Thin Film Oven Test" (40). In this test, asphalt is spread in a standard container in 1/8 inch thick film and heated in a specified oven at 325°F for five hours. Routine tests are run (specially ductility and penetration) on the residue from this test. The Bureau of Public Roads has run Thin Film Oven tests for almost all grades of asphalts, supplied by almost all the producers, to different highway construction agencies in this country (38, 40, 73). The film thickness can be changed to any other thickness if any modification of this method is considered necessary just by using a larger pan or using a smaller quantity of material. Changes that take place during this test are comparable to the changes that may be expected to occur in bitumen recovered from mixtures prepared in hot mix plants. Vallergera, Monismith and Granthem (70) have further modified this test for studying the effect of infra-red and ultra-violet radiation on asphalts.

The ability of asphalts to retain their original characteristics as measured by this test, offers a mean of evaluating their relative durability. The allowable loss in the physical properties during this test is specified by the Bureau of Public Roads:

(a) The loss in weight after this treatment, should not be greater than one per cent for all penetration grades of asphalt from 50-60 through 120-150 grades.

(b) Residue from Thin Film Oven test shall have a penetration at 77°F (100 grams, 5 seconds) of at least 50% of that of the original sample. This appears to be equally suitable for all penetration grades of asphalt from 50-60 through the 120-150 grades.

(c) Residue from Thin Film Oven test shall have a ductility at 77°F (5 cm per minute) of not less than 40 cm for 50-60 and 60-70 penetration grades and not less than 100 for 85-100, 100-120 and 120-150 penetration grades.

Asphalt oxidation

It is claimed by many researchers that oxidation is the prime cause of asphalt hardening (8, 35, 47, 56, 64). Nicholson (47) used a modified Abson apparatus, to oxidize asphalts by blowing air through samples heated at various temperatures. Since asphalts from different crudes and different refining processes react differently to oxidation, he

concluded that the mixing temperatures and their temperature susceptibility should be given primary consideration. Blowing air for one hour at a specified rate and at mixing temperature, reduced the penetration of many asphalts to one half of their original value. Anderson, Stross and Ellings (8) subjected the asphalt solution in benzene to oxygen at different pressures and at constant temperature. Changes in pressure led them to develop a deterioration index for the evaluation of asphalt behavior when subjected to oxygen.

Laboratory natural weathering

It has been observed that when asphalt in thin film is subjected to natural weathering conditions (rain, light, etc.) it develops cracks. Zapata (76) subjected the asphalt in thin film to the action of light, darkness, freezing temperatures, water sprays, etc. to simulate a natural field condition. The failure of thin films was checked by spark and stain tests. The cycle he used for his study was:

Freezing at 20-25°F	-----1.5	hours
Water spray	-----1	hour
Light without spray	-----2	hours
Water spray	-----2	hours
Light without spray	-----6	hours
Water spray	-----1	hour
Light without spray	-----2	hours
Water spray	-----2	hours
Light without spray	-----6	hours

ASTM has a tentative method for accelerated weathering of bituminous materials (7). For this test asphalt samples of

6" x 3" x 1/16" dimensions are placed on aluminum plates and subjected to light, rain, dry weather etc. inside the laboratory for certain periods of time. Tested samples are compared with the original ones. The durability of the material is expressed as the number of cycles of weathering that are required to bring about definite changes in pliability, breaking strength, solubility in standard solvents and appearance of the material tested.

Shaw (63), interested in the 50-60 penetration grade asphalts used for water proofing canals, studied the effect of natural weathering on thin films of asphalts. He evaluated his results based on penetration, ductility, softening point, cavitometer, ball pressure, strain and film hardness tests. The last four tests are not standard tests. From his work he developed an aging index for evaluation of the changes that take place in asphalts during thin film natural weathering.

Weathering of asphaltic mixtures

Sheet asphalt mixtures prepared with Venezuelan asphalt at 325°F were maintained at the same temperature for 30 minutes to one hour. Asphalt recovered by Abson method was compared for penetration and ductility with the original and also that recovered from mixes just after mixing (29). Com-

parison of such heat treatment was also made with serviceability of pavements. Similar heat treatment studies were made on 2" by 2" samples prepared from Ottawa sand-asphalt mixes. Heat treated samples were tested for compressive strength at 77°F (49, 62, 75). It was found that the weathering of Ottawa sand was very severe, due to high per cent of voids which result in greater oxidation.

Aggregates and asphalt go through a severe change in temperature before the asphaltic concrete mixture is obtained. Asphaltic concrete mixture may remain at high temperatures for a period of time in an uncompacted state prior to laying. Though undesirable, many times in colder climate areas during winter months, the asphaltic concrete mixture is supplied on jobs up to a temperature of 375°F (61).

Selected asphaltic concrete mixtures were subjected to heat, oxidation and ultra-violet radiation treatments in this investigation. The details of the work are presented in different parts of this thesis.

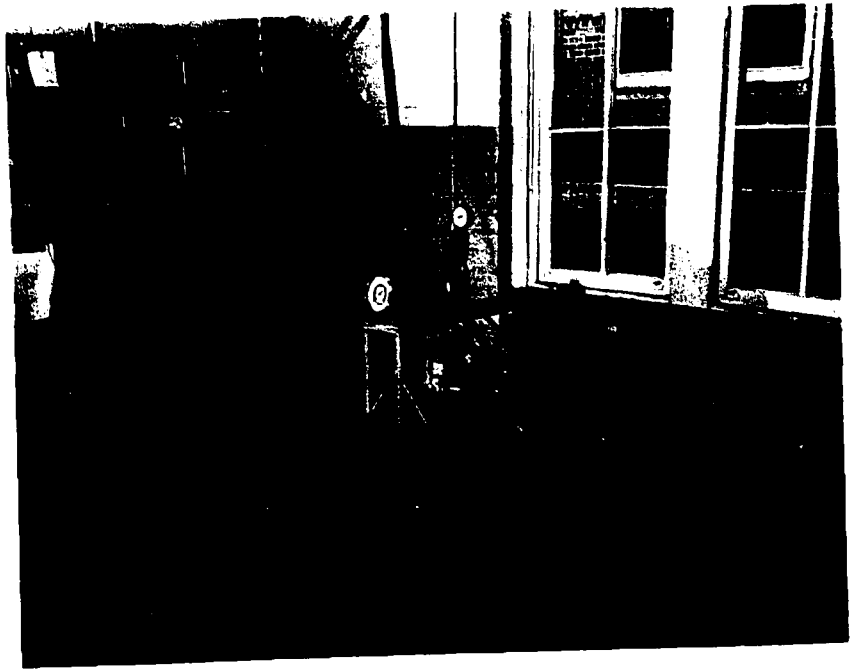
Effect of laboratory traffic on asphaltic concrete mixtures

Cantrill (17) and many other investigators, developed test tracks for accelerated testing of bituminous paving mixtures. Various aggregates and asphalts mixtures were studied. Correlation between test track results and actual pavement behavior can be established. Ekse and LaCrosse (23) have

developed another type test track to study the influence of bituminous mat thickness upon surface deflections, sub-grade soil pressures and base course requirements. A similar test track to study the effect of traffic on sheet asphalt and asphaltic concrete compacted specimens by various compaction devices is underway in the Bituminous Research Laboratory at Iowa State University at Ames, Iowa. Plate 1 gives a general view of this test track.

Plate 1. A general view of the test track.

Plate 2. Hetherington-Berner twin shaft pug mill mixer.



PURPOSE OF STUDY

Many researchers have shown that the asphalt recovered from asphalt-aggregate mixtures by the available methods represent the true condition of the asphalt in mixtures. It has been concluded that the asphalt hardens with age in pavements even at moderate temperatures. This hardening of asphalt is considered one of the fundamental causes for the failure of asphalt pavements.

The object of this study is therefore threefold:

1. To determine the changes that take place in the physical and chemical properties of asphalt when heat treated at high temperatures and for long periods of time.

2. To determine the changes in the physical and chemical properties of asphalts recovered from the asphalt-aggregate mixtures subjected to heat, ultra-violet and oxidation treatments in uncompacted state.

3. To determine the changes in the physical characteristics of the asphalt-aggregate mixtures when subjected to heat, ultra-violet and oxidation treatments in uncompacted state.

MATERIALS AND METHODS

Asphalts

Asphalts used in this study are designated as A, B, C and D. They are of 30-40, 85-100, 85-100 and 150-200 penetration grades respectively. The physical and chemical characteristics of these asphalts are given in Table 1. Asphalts B and C were obtained from the plants during the construction of two Iowa highways. Asphalts A and D were obtained from the producers directly. The 85-100 penetration grade is commonly used in the state of Iowa. The other two penetration grades namely 30-40 and 150-200 were selected to check the beneficial or detrimental effect of harder and softer grades over 85-100 penetration grade.

Aggregates

Aggregates from two sources were selected for this study. Combined aggregates are designated as X and Y. The physical and chemical characteristics of these aggregates are given in Table 2. Four types of aggregates were obtained from each source, i.e. coarse aggregate, intermediate aggregate, fine aggregate and dust. These aggregates were collected from hot bins at the plants during the construction of two Iowa highways. Gradations of individual and combined X and Y aggregates are given in Tables 3 and 4.

Table 1. Physical properties of asphalts used^a

As-phalt	Pene- tration 77°F/100 gms/5 sec	% loss on heat at 325°F for 5 hours	Speci- fic gravity at 77°F	Soft- ening point °F	Solu- bility in cc1 4%	Flash point °F	Fire point °F	Duc- til- ity	Oli- ensis spot test	% as- phalt- enes ^b
A	34	.05	1.004	144	99.64	650	695	100+	Neg.	19.94
B	76	.07	1.005	120	99.93	640	720	150+	Neg.	16.60
C	86	.08	1.015	115	99.92	635	705	150+	Neg.	18.20
D	140	.09	0.991	108	99.70	625	685	150+	Neg.	13.40
Test Method	ASTM D-5	ASTM D-6	ASTM D-70	ASTM D-36	ASTM D-165	ASTM D-92	ASTM D-92	ASTM D-113	AASHO T-102	-

^aAll tests except % asphaltenes follow as mentioned, AASHO and ASTM Standards (6 and 7).

^bCsanyi and Fung (21).

Table 2. Physical properties of aggregates used

Comb. agg. designation	Agg. type	X-ray diffraction components		Appar. spec. grav.	Bulk spec. grav.	Pwdr. spec. grav. ^a	Pore size dist. u ^b	Eff. porosity % ^b	Centri. kerosene equiv. %	Water absorp. %
		Major	Minor							
X	Coarse aggregate	Calcite	Quartz Traces of Dol.	2.69	2.56	2.70	1.20	4.3	-	1.87
	Inter. aggregate	Calcite	Quartz Dolomite	2.67	2.48	-	-	-	4.0	2.15
	Fine aggregate	Quartz	Calcite	2.66	2.45	-	-	-	4.5	3.29
	Dust	-	-	2.69	-	-	-	-	8.7	-
Y	Coarse aggregate	Dolomite	Calcite Quartz	2.73	2.36	2.72	4.40	14.2	-	6.64
	Inter. aggregate	Dolomite	Quartz Calcite	2.66	2.43	-	-	-	6.25	-
	Fine aggregate	Dolomite	Calcite Quartz	2.67	2.49	-	-	-	4.2	2.72
	Dust	-	-	-	-	-	-	-	-	-

^aRush (58).^bRitter and Drake (57).^cBased on microscopic measurement of binder penetration in coarse a

used

a	Pore size dist. u ^b	Eff. porosity % ^b	Centri. kero-sene equiv. %	Water absorp. %	Absorp. with SAE No. 10 oil %	Ratio oil to water absorp.	Esti-mated binder absorp. ^c
1)	1.20	4.3	-	1.87	3.2	1.71	Low
	-	-	4.0	2.15	3.0	1.40	-
	-	-	4.5	3.29	-	-	-
	-	-	8.7	-	-	-	-
2)	4.40	14.2	-	6.64	5.6	0.84	High
	-	-	6.25	-	5.0	-	-
	-	-	4.2	2.72	-	-	-
	-	-	-	-	-	-	-

binder penetration in coarse aggregates.

Table 3. Gradations of combined aggregates X, design mix and Iowa Highway Commission specification limits for type A asphaltic concrete mixtures

U. S. sieve number	Total percent passing				De-sign mix	Iowa Highway Commission specification limits
	Coarse aggregate	Intermediate aggregate	Fine aggregate	Filler (dust)		
3/4"	99.4	100	100	100	99.8	98-100
1/2"	67.9	100	100	100	94.7	85-100
3/8"	20.8	99.9	100	100	80.2	67-87
4	1.1	29.9	98.8	100	57.4	47-68
8	0.8	2.1	88.1	100	45.9	37-55
30	0.7	1.4	51.9	99.9	31.3	19-34
50	0.0	1.4	30.4	96.5	22.0	13-26
100	0.0	1.2	11.9	65.7	11.6	6-18
200	0.0	0.9	7.3	43.3	7.4	3-10

Table 4. Gradations of combined aggregates Y, design mix and Iowa Highway Commission specification limits for type A asphaltic concrete mixtures

U. S. sieve number	Coarse aggregate	Total percent passing		De-sign mix	Iowa Highway Commission specification limits
		Intermediate aggregate	Fine aggregate		
3/4"	99.7	100	100	100	98-100
1/2"	59.6	100	100	89.9	85-100
3/8"	14.3	99.2	100	78.4	67-87
4	0.9	28.5	100	61.0	47-68
8	0.8	1.6	90.3	50.5	37-55
30	0.7	1.3	48.6	27.2	19-34
50	0.0	1.3	27.7	15.5	13-26
100	0.0	1.2	13.6	7.7	6-18
200	0.0	1.1	8.7	5.0	3-10

Mix Design

Laboratory mixes for asphaltic concrete were designed from the gradations given in Tables 3 and 4 to meet the Iowa Highway Commission specifications for type A asphaltic concrete mixes (32). The designed mix for aggregate X consists of 25 per cent coarse aggregate, 25 per cent intermediate aggregate, 43 per cent fine aggregate and 7 per cent dust and for aggregate Y, 25 per cent coarse aggregate, 20 per cent intermediate aggregate and 55 per cent fine aggregate. The gradation of designed mixes and the Iowa Highway Commission specifications for such mixes are given in Tables 3 and 4 and shown graphically in Figures 1 and 2.

Mixing Procedure

The laboratory samples of the asphaltic concrete mixes were made in a Hetherington-Berner twin shaft laboratory pug mill mixer of 50 pound capacity. Plate 2 gives a general view of the mixer. The paddle tips of the mixer are so arranged as to give a figure eight type mixing action. All the mixing was done in batches of twenty-five pounds. The mixer is equipped with an electrical heating system, hence the body of the mixer was heated prior to any mixing.

The designed mix aggregate was heated up to $350 \pm 10^{\circ}\text{F}$ and asphalt heated to $300 \pm 10^{\circ}\text{F}$ before mixing. Twenty-five

pounds of aggregate was mixed dry for fifteen seconds and then 1.56 pounds of asphalt (6.25 per cent by weight) was poured over the mixing aggregate manually, the wet mix was continued for another 30 seconds. The temperature of the resulting mixture in all batches varied from 250°F to 280°F. Aggregates X and Y were mixed with asphalts A, B, D, and A, C, D respectively. This resulted in six different asphaltic concrete mixes. The mixer speed, mixing time, asphalt and aggregate temperatures were maintained constant during all mixing operations.

Heat Treatment of Asphalts

The four asphalts A, B, C, and D were subjected to heat treatment. Approximately 50 grams of each material was taken in 3 oz. glass bottles and heated in constant temperature ovens. The duration of heating was 2, 4, 8, 16, 24, and 72 hours in an oven set at 325°F and 4, 8, 16, 24, 72 and 120 hours in an oven set at 230°F. The glass bottles were open at top and stirred periodically with a glass stirrer. A loss on heat oven with a rotating shelf was used for the heat treatment.

Treatment of Mixes for Asphalt Recovery

Heat treatment

Representative samples of one thousand grams of the asphaltic concrete mixes were used for heat treatment. These

samples were subjected to heat in ovens set at 325, 230 and 140°F for a period of 2, 4, 8, 12 hours; 4, 8, 16, 72 hours; and 120, 240, 360 and 720 hours respectively. The asphaltic concrete mix was spread in enamel 16" x 11" x 2.5" pans to give a minimum sample thickness. Mixes were stirred periodically to insure uniform heating. After the specified time of treatment was over the samples were taken out of the ovens and allowed to cool to room temperature. The asphalt was recovered from these treated mixes as explained under "asphalt recovery". All the six types of asphaltic concrete mixes were similarly treated.

Ultra-violet radiation treatment

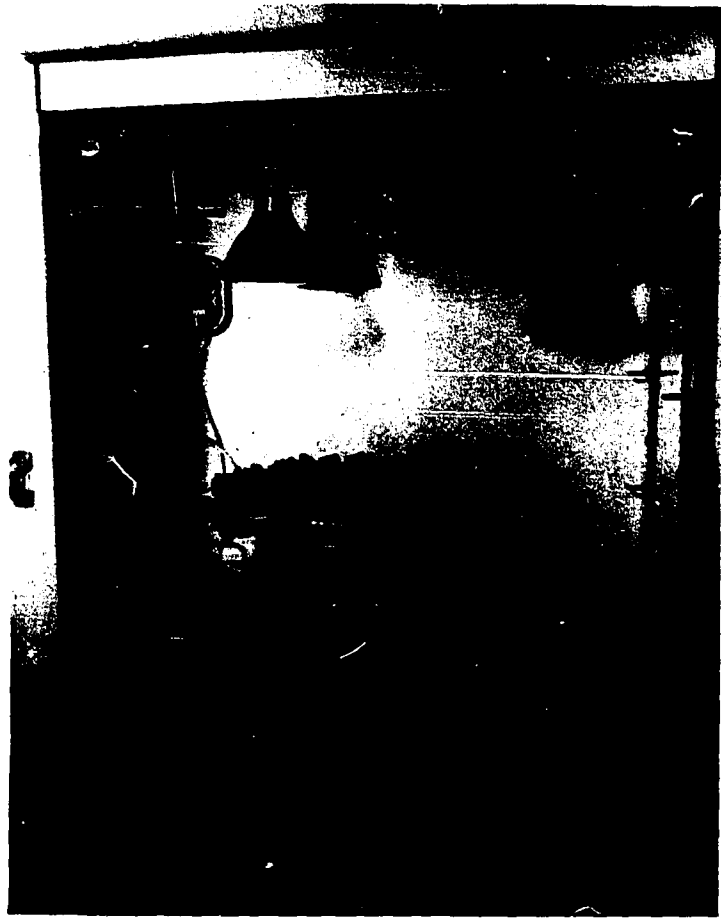
Asphaltic concrete mix samples similar to those used in heat treatment were subjected to ultra-violet radiation. Each sample was spread on a revolving shelf shown in Plate 3. The ultra-violet radiation source was so arranged as to have only half of the sample directly under the light at one time. The rotating speed of shelf was 1.5 revolution per minute. An ultra-violet light of 3700 Angstrom wave length was used. Temperature of mix due to ultra-violet radiation was maintained below 140°F. Each asphaltic concrete mix sample was subjected to such radiation for 4, 8, 16 and 24 hours.

Oxidation treatment

Similar samples of the mixes weighing 1000 grams, as

Plate 3. Treatment of loose asphaltic concrete mix by
ultra-violet radiation.

Plate 4. Pressure device used for oxidation treatment of
asphaltic concrete mixes.



used in heat treatment, were placed in pressure cooker as shown in plate 4. The pressure cooker was evacuated and then filled with oxygen up to a pressure of five pounds per square inch and then placed in preheated oven for a specified period. The duration of heat in an oven set at 325°F was 1, 2 and 4 hours and in an oven set at 230°F was 2, 4, and 8 hours. At the end of the specified time the cooker was removed from the oven, the oxygen released and the mixture allowed to cool to room temperature and stored until asphalt was recovered. Samples from all the six asphaltic concrete types were similarly treated.

Treatment of Mixes for Stability and Cohesion

Heat treatment

Approximately an eight pound sample of representative mix was placed in large size enamel pan and subjected to heat treatment. The temperatures and treatment time selected were the same as used for treatment of mixes for asphalt recovery. The mix was stirred periodically to insure uniform heating. After the specified time the mix was removed from the oven and three specimens 4 inches in diameter and 2.5 inches in height were compacted by static load, double plunger method. All of the six asphaltic concrete mixes were treated similarly.

Ultra-violet radiation treatment

Asphaltic concrete samples in the loose state weighing 1200 grams, were subjected to ultra-violet radiation as mentioned "under treatment of mixes for asphalt recovery", for a period of 4, 8, 16, and 24 hours. One 4 inches in diameter and 2.5 inches in height was compacted by static load double plunger method of each mix.

Oxidation treatment

Oxidation treatment of asphaltic concrete mixes for stability and cohesion was similar to the one as described "under treatment of mixes for asphalt recovery" except 1200 grams of material were used instead of 1000 grams. One specimen 4 inches in diameter and 2.5 inches in height was compacted by static load double plunger method from such oxidized material.

Compaction Procedure

Specimens, four inches in diameter and two and one half inches in height were compacted from treated and untreated asphaltic concrete mixes. A static load double plunger compaction procedure as used by the Iowa Highway Commission was followed. Compaction temperature of the mix was maintained at 250°F. Constant loading rate was used for all compaction work. Steel plates between the mold and the base plate were removed when the load reached 1000 lbs. (80 psi).

Further loading continued at the same rate until the load reached to 37,716 lbs. (3000 psi). This load was maintained for three minutes, and then released slowly and the sample extruded from the mold. All the samples were compacted under identical conditions. The tolerance in the height of samples was $\pm 1/16$ inches.

Extraction of Asphalt from Mixtures

Asphalt was extracted from asphaltic concrete mixes by standard A.A.S.H.O. rotarex centrifuge procedure, using benzene as a solvent. To accelerate centrifuging 1000 grams of the asphaltic concrete mixture was soaked in 250 ml. of benzene for one hour in one quart glass jars. The contents of the jars were stirred periodically by shaking the jar. The mixture (benzen + mix) having most of the asphalt dissolved was then centrifuged in the Rotarex centrifuge. Additional quantities of benzene were added so as to complete the extraction.

The asphalt solution in benzene, so obtained contained undesirable quantities of colloidal mineral matter. This was removed by further centrifuging the solution at 400 G for 30 minutes. Dust free asphalt-benzene solution was decanted from the centrifuge flasks, thus leaving mineral matter at the bottom of the flasks. Asphalt was recovered from this solution by modified Abson method.

Recovery of Asphalt

The Abson apparatus with some changes was used for recovering the asphalt. The distillation assembly is shown in Plate 5. One neck of the 500 ml. pyrex flask was fitted with a thermometer, the middle neck provided a connection between flask and condenser and the third neck was closed temporarily with a cork, to be replaced by a dispersion tube in the later stages of distillation. The flask was heated with an electric heating mantle. The mantle temperature was maintained at 350°F so as to keep the flask contents at 325 ± 10°F. Distillation was continued without gas until the rate of condensation of benzene decreased to 10 to 15 drops per minute. The cork in the third neck of the flask was then replaced by a dispersion tube for CO₂. Carbon dioxide was supplied at a slow rate at first and increased then to a maximum of 800-900 ml. per minute for 30 minutes. The flow of CO₂ gas was measured by a gas flow meter. At the end of thirty minutes the gas was discontinued and the asphalt poured into a container and stored for further studies.

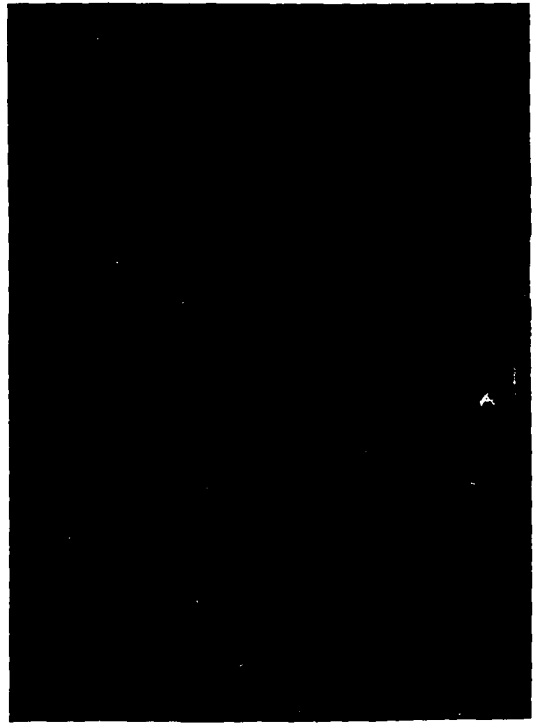
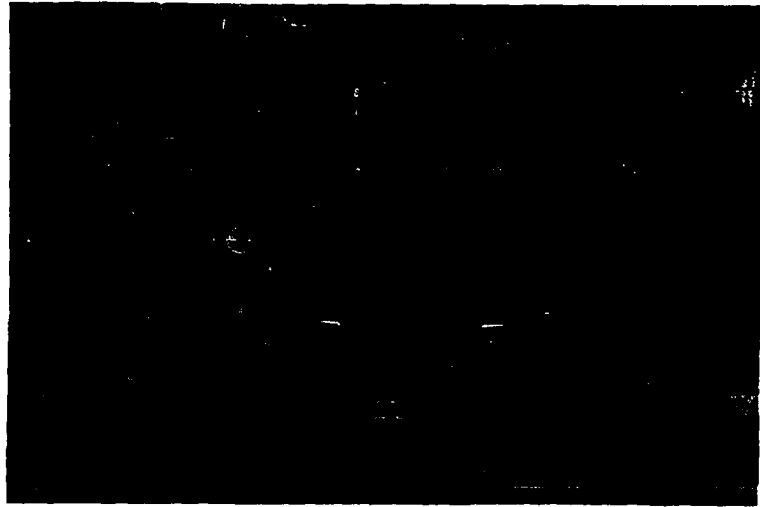
Testing procedure for asphalts

Penetration A.S.T.M. Standard Test D-5 procedure
"for penetration of bituminous materials" was followed to determine the penetration of original, treated and recovered

Plate 5. Distillation assembly for reclaiming asphalt.

Plate 6. A general view of the apparatus for determining the asphaltene content of asphalts.

Plate 7. Sliding plate microviscometer in operation.



asphalts (7). The load, time and temperature used for this test were 100 grams, 5 seconds and 77^oF (25^oC) respectively, and the units of penetration to indicate hundredths of a centimeter.

Softening point The A.S.T.M. Standard (D-36) ring and ball method was used to run the softening points of the asphalts (original, treated, recovered). The softening point results are expressed in degrees Fahrenheit.

Percent asphaltenes The asphaltene content for all the asphalts was obtained by the selective solvent method proposed by Csanyi and Fung (21). Skelly F was used as a solvent. Apparatus used for this determination is shown in Plate 6. The extraction time in the soxhlet extractors was maintained for 8 hours throughout this study. Asphaltene content is expressed as a percent of the total asphalt used for the test.

Viscosity The viscosity of all the asphalts was obtained by using a microviscometer. The details of procedure are given by inventors of this equipment (35), and also outlined by Griffin, Miles and Penther (28) and as supplied with the equipment. A general view of the equipment in operation is shown in Plate 7. The viscosity values so obtained are plotted on a log log paper and the viscosity value corresponding to shearing rate of $5 \times 10^{-2} \text{ sec}^{-1}$ is

obtained from the graph.

For reporting viscosities, $5 \times 10^{-2} \text{ sec}^{-1}$ shear rate was chosen by Griffin, Miles and Penther (28), because it is a convenient rate for thin film viscometer. Other shear rates for this viscometer between $5 \times 10^{-2} \text{ sec}^{-1}$ and $5 \times 10^{-1} \text{ sec}^{-1}$ can also be used as long as the same shear rate is used for all asphalts to be compared. The viscosity value is expressed in poises.

Testing procedure for compacted samples

Specific gravity and percent voids Bulk density and per cent voids in compacted samples were determined as follows:

$$\text{Bulk specific gravity} = \frac{\text{weight of sample in air, grams}}{\text{weight of sample in air, grams} - \text{weight of sample in water, grams}}$$

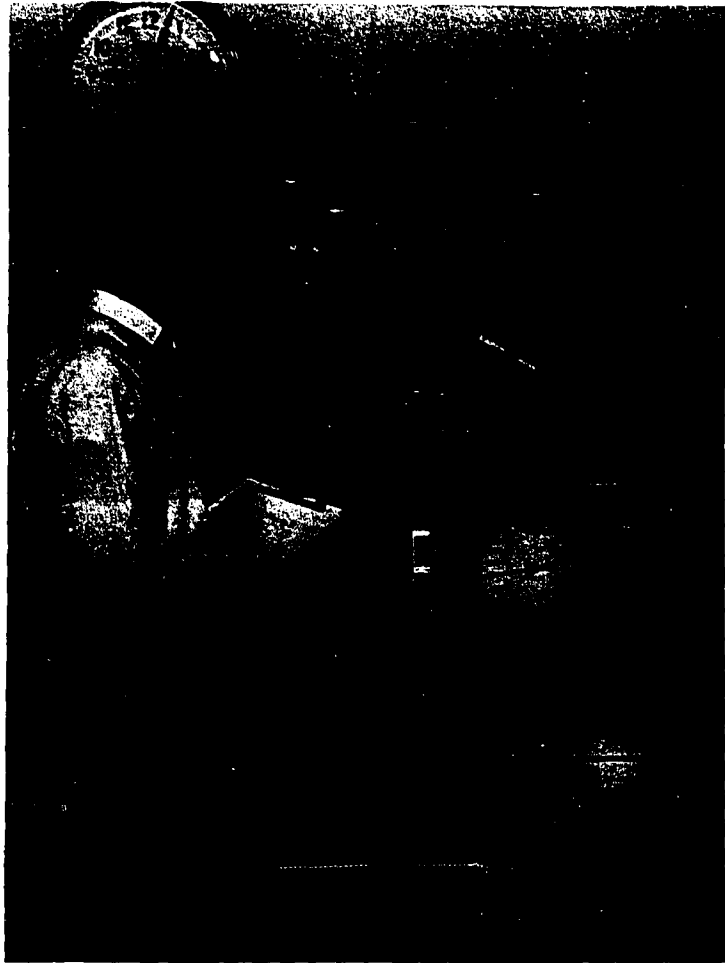
$$\text{Percent voids} = \frac{100 (\text{maximum theoretical specific gravity} - \text{bulk specific gravity})}{\text{maximum theoretical specific gravity}}$$

The maximum theoretical specific gravity was obtained by the method outlined by Serafin (60).

Stability Stability of the compacted specimens was obtained by Hveem stabilometer shown in Plate 8. The procedure outlined in the material and research department laboratory manual of California standard test procedures was followed (16). All samples were tested at 140°F. The calculated stability values (S) and the stability values used by

Plate 8. Hveem stabilometer in operation.

Plate 9. Hveem cohesiometer.



the Iowa Highway Commission are given under results and discussion. The Iowa Highway Commission used lateral pressure reading corresponding to a vertical pressure of 400 psi as their design criteria, instead of the calculated Hveem stability value "S".

Cohesion Cohesion values for the compacted specimens were obtained by using the Hveem cohesiometer shown in Plate 9. The California standard test procedure outlined in a Material and Research Department Laboratory Manual was followed (16). All samples were tested at 140°F and the cohesion value "C" calculated as outlined in that manual.

PRESENTATION AND DISCUSSION OF RESULTS

Four asphalts used in this study were subjected to heat treatments at 230 and 325^oF. Six asphaltic concrete mixes prepared with the four asphalts and two aggregates were subjected to heat, ultra-violet radiation and oxidation treatments in an uncompacted state. The asphalt from these treated asphaltic concrete mixes was recovered by the modified Abson method. Stability and cohesion of the compacted samples were determined by the Hveem stabilometer and cohesiometer. Test results are presented both in tabular as well as in graphical form to aid in the interpretation of the results.

Heat Treatment of Original Asphalts

The four asphalts A, B, C, and D used in this study were heat treated at 325 and 230^oF for various time intervals. The experimental results for these asphalts are as follows:

Experimental results

Penetration Consistency changes that take place during the heating of asphalts were measured by a standard penetration test. The penetration values so obtained are given in Table 5 and shown graphically in Figure 3. These four asphalts showed a loss in penetration of between 15 and 40 per cent at the end of 24 hours and between 20 and 55 per cent at the end of 72 hours of continuous heating at

Table 5. Penetration of asphalts before and after heat treatment

Treatment temperature OF	Treatment time hrs.	Penetration			
		A	B	C	D
—	0	34	76	86	140
325	2	31	74	80	130
325	4	29	67	77	128
325	8	28	62	74	120
325	16	28	56	67	115
325	24	28	47	62	104
325	72	26	35	40	69
230	4	32	74	83	134
230	8	32	73	82	128
230	16	32	70	78	126
230	24	31	70	76	126
230	72	31	62	69	117
230	120	28	55	60	112

325°F. The same asphalts when heated at 230°F showed a loss of 8 to 12 per cent after 24 hours and 8 to 20 per cent after 72 hours of continuous heating. In both of these cases asphalt B (85-100 penetration grade) showed the highest and asphalt A (30-40 penetration grade) showed the lowest per cent loss in penetration. Asphalt D (150-200 penetration grade) showed less per cent loss in penetration than the

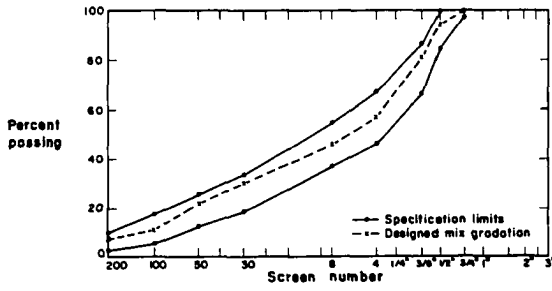


Figure 1. Gradation of combined aggregate X and Iowa Highway Commission specifications for type A asphaltic concrete mixes.

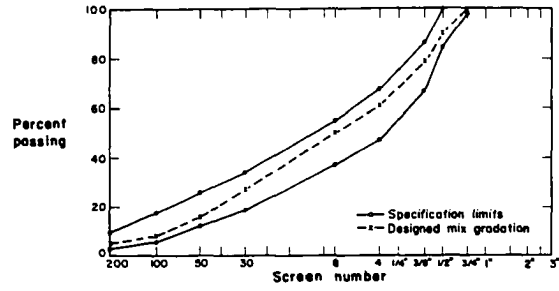


Figure 2. Gradation of combined aggregate Y and Iowa Highway Commission specifications for type A asphaltic concrete mixes.

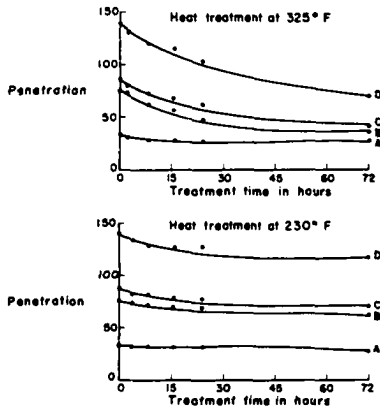


Figure 3. Penetration of asphalts after heat treatment at 230 and 325°F.

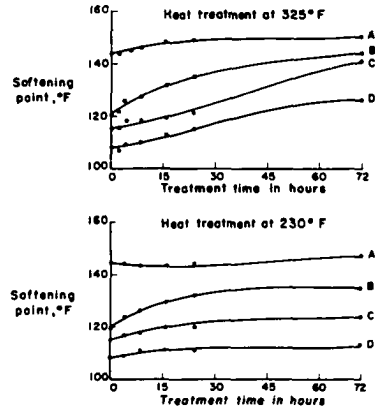


Figure 4. Softening point of asphalts after heat treatment at 230 and 325°F.

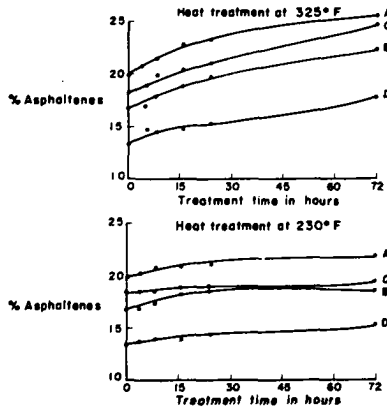


Figure 5. Percent asphaltene of asphalts after heat treatment at 230 and 325°F.

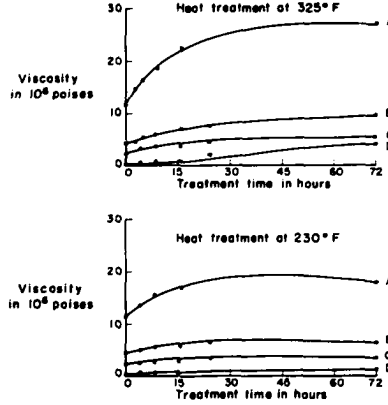


Figure 6. Viscosity of asphalts after heat treatment at 230 and 325°F.

85-100 penetration grade asphalts B and C. Per cent loss in penetration for these four asphalts when heated at 325 and 230°F for periods of 24 and 72 hours is shown in Figure 55. Volatilization of light molecular weight hydrocarbons or their changes to higher molecular weight hydrocarbons due to oxidation or dehydrogenation or polymerization reactions seems to bring these changes in penetration values.

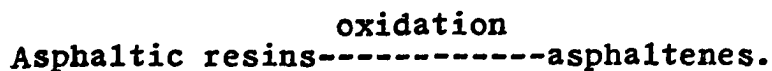
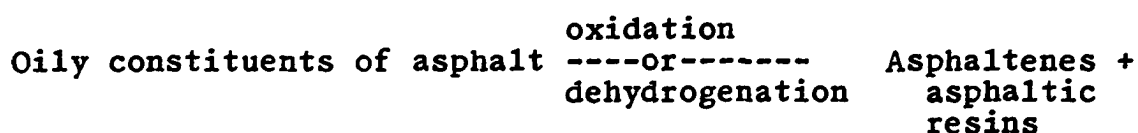
Softening point The effect of temperature on these asphalts was judged by the softening point test. The Ring and Ball method was used to determine the softening point. The results so obtained are given in Table 6 and shown graphically in Figure 4. The increase in softening point varied between 4 and 13 per cent after 24 hours and between 4 and 22 per cent after 72 hours of heating at 325°F. Per cent increase in softening point varied between 0 to 10 per cent and 0 to 13 per cent when the same asphalts were heated at 230°F for 24 and 72 hours respectively. Asphalt C (85-100 penetration grade) showed the highest and asphalt A (30-40 penetration grade) showed the lowest per cent increase in softening point. Asphalt D (150-200 penetration grade) showed comparatively lower per cent increase in softening point than the 85-100 penetration grade asphalts B and C. The increases in softening points indicate that heating of asphalts at higher temperatures make them less susceptible

Table 6. Softening point of asphalts before and after heat treatment

Treatment temperature of	Treatment time hrs.	Softening point of			
		A	B	C	D
—	0	144	120	115	108
325	2	145	122	115	109
325	4	145	126	118	109
325	8	146	127	118	110
325	16	148	131	119	115
325	24	149	135	121	118
325	72	150	144	141	126
230	4	144	124	117	109
230	8	143	126	118	111
230	16	143	129	120	111
230	24	144	132	120	111
230	72	144	135	124	113
230	120	147	136	131	115

to temperature but at the cost of other physical properties. Increase in softening point of asphalts A, B, C and D after continuous heating for 24 and 72 hours at 230 and 325°F is shown in Figure 55.

Per cent asphaltenes Asphaltenes, one of the three major constituents of asphalt, are dark brown or black in color consisting of high molecular weight hydrocarbons. The per cent asphaltenes was determined by the selective solvent method (21). The values so obtained are given in Table 7 and shown graphically in Figure 5. The per cent increase in asphaltenes is between 14 to 20 per cent after 24 hours and between 28 to 35 per cent at the end of 72 hours of heating at 325°F for all the four asphalts. The increase in asphaltene content is much less at lower temperatures; it is 3 to 10 per cent after 24 hours and up to 14 per cent after 72 hours of heating at 230°F. The increase in asphaltenes is considered due to the oxidation or dehydrogenation of oily constituents of asphalts. The oily constituents generally result in the formation of asphaltic resins, which in turn form asphaltenes by further oxidation as shown by the following equation:



Such a change in the constituents of asphalt to form more asphaltenes results in the hardening of asphalt.

Increases in the asphaltene content of asphalt A, B,

Table 7. Per cent asphaltenes of asphalts before and after heat treatment

Treatment temperature OF	Treatment time in hrs.	Per cent asphaltenes			
		A	B	C	D
—	0	19.94	16.60	18.20	13.40
325	2	20.06	16.65	18.45	14.29
325	4	20.68	16.80	18.80	14.50
325	8	21.49	17.82	19.90	14.68
325	16	22.89	18.60	20.40	14.80
325	24	23.10	19.90	21.00	15.26
325	72	25.54	22.30	24.60	17.86
230	4	20.02	16.75	18.40	13.60
230	8	20.62	17.20	18.45	13.90
230	16	20.73	18.28	18.60	13.82
230	24	21.00	18.35	18.84	14.38
230	72	21.66	18.50	19.40	15.33
230	120	23.08	18.60	19.85	17.87

C and D and due to 24 and 72 hours of heating at 230 and 325°F are shown in Figure 55.

Viscosity The viscosity values for all the asphalts in poises, were obtained by using a microviscometer. The viscosity values are given in Table 8 and shown graphically in Figure 6. Asphalts A, B and C increased in viscosity 2

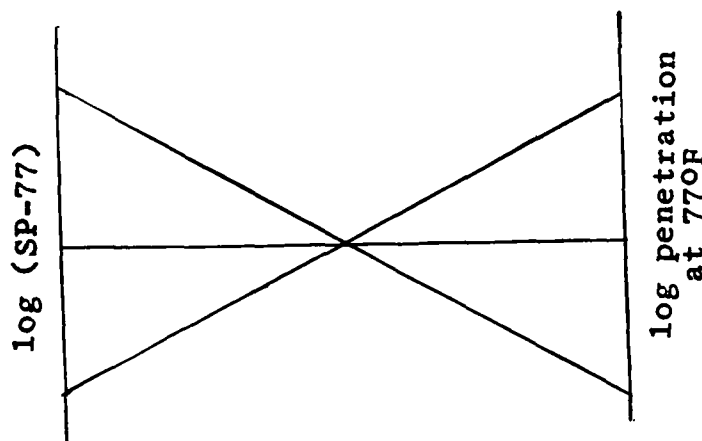
Table 8. Viscosity of asphalts before and after heat treatment

Treatment temperature OF	Treatment time in hrs.	Viscosity in 10 ⁶ poises			
		A	B	C	D
—	0	11.80	4.50	2.25	0.45
325	2	14.60	4.80	2.90	0.59
325	4	16.50	5.30	3.30	0.61
325	8	18.50	6.00	3.50	0.74
325	16	22.50	7.00	3.80	0.96
325	24	23.50	7.80	4.80	2.02
325	72	27.50	9.68	5.83	4.50
230	4	13.50	5.00	2.75	0.64
230	8	15.50	5.40	2.85	0.51
230	16	16.50	5.60	2.90	0.62
230	24	17.00	6.10	3.20	0.74
230	72	18.00	6.20	3.60	1.05
230	120	18.50	8.34	4.90	1.13

to 3 fold and asphalt D, 7 to 10 fold when heated at 325⁰F for 24 to 72 hours. At 230⁰F the viscosity changes were not too severe, however increase in viscosity up to 2 times the original value were obtained after 72 hours of heating. Increases in viscosity values due to 24 and 72 hours heating of all the four asphalts at 325 and 230⁰F are shown in Figure 55. The increase in viscosity of asphalt is indicative

of its hardening and is due to the changes in chemical composition brought by the heat treatment.

Characterizing factor According to Kinnaird (34) when the lines connecting the log (softening point -77) and log penetration at 77°F of all asphalts obtained from the same stock are drawn, they all pass through the same common point (see figure below). He developed what is known as



characterizing factor to establish a mathematical relationship between softening point and penetration of various asphalts.

$$P_{77} = F \left[\frac{SP-77}{100} \right]^{-4.25/3} \sqrt[3]{F}$$

P_{77} = penetration of asphalt at 77°F, 100 grams and 5 seconds,

SP = softening point in °F (ring and ball) method, and

F = characterizing factor.

The characterizing factor permits the classification of asphalts into groups that are similar in chemical composition. Asphalts having identical percentages of paraffinic constituents were found to have an identical characterizing factor, and this factor has the same mathematical value as the percent paraffinic constituent of asphalts. If changes take place in asphalt in such a way that the paraffinic constituent remains unchanged, the characterizing factor also remains unchanged, irrespective of the changes in penetration and softening point. On the other hand if the chemical composition of the asphalt is materially changed, there will also be a change in the characterizing factor. Changes in the characterizing factor then could be considered as a measure of the changes that take place when asphalts are subjected to various treatments.

The characterizing factor values for all asphalts were obtained from the nomograph (34). The values so obtained are given in Table 9. These values for asphalts subjected to heat treatment for various time intervals and different temperatures are essentially the same. The slight variations in these values might have been due to experimental error. No change in characterizing factor indicates that heat treatment has no effect on the paraffinic constituents of asphalts. However, other chemical constituents of asphalt are affected by heat treatment, which in turn bring some changes in such

Table 9. Characterizing factor for asphalts before and after heat treatment

Treatment temperature OF	Treatment time in hrs.	Characterizing factor			
		A	B	C	D
-	0	18	22	20	27
325	2	16	23	16	25
325	4	16	23	19	26
325	8	15	22	18	24
325	16	16	23	16	28
325	24	17	21	16	27
325	72	15	20	20	25
230	4	16	24	21	27
230	8	16	27	20	29
230	16	16	30	22	28
230	24	16	32	21	28
230	72	16	30	23	29
230	120	15	26	25	31

physical properties of the asphalt as penetration and softening point etc. The average characterizing factor values for asphalts A, B, C and D are 18, 22, 20 and 27 respectively. Kinnaird in his study used similar penetration grade asphalts as A, B, C and D and obtained characterizing factor values of 7, 16, 19 and 21 respectively. These asphalts were from different sources and had different penetration and softening points than the ones used in this study.

Penetration index Penetration index was introduced by Pfeiffer (50, 51) while studying the penetration-temperature susceptibility of asphalts. The derivation of the penetration index is based on the assumption that all asphalts have penetration equal to 800 at their softening point.

$$PI = \frac{30}{1 + 90 PTS} - 10$$

$$PTS = \frac{\text{Log } 800 - \text{Log } P}{t - 77}$$

PI = penetration index

PTS = penetration-temperature susceptibility

p = penetration at 77°F, 100 grams and 5 seconds

t = softening point °F (ring and ball method).

Based on the penetration index asphaltic bitumens were grouped into three classes merging into one another, the boundaries of which have been chosen more or less arbitrarily (52):

1. The class with index -1 to +1, the "N type". This class comprises a great many steam-refined bitumens, generally used for road construction. These are therefore sometimes called "normal bitumens".

2. The class with an index below -1, the "Z type". This class is also sometimes called the "coal-tar pitch type". Like the coal tar pitches, the bitumens of this type are characterized by their great susceptibility and in gener-

al also by their greater tendency to show brittleness.

3. The class with an index greater than 1, the "R type". In addition to a low temperature susceptibility, these bitumens are generally characterized by slight brittleness. As most of the blown bitumens have these characteristics, this type of bitumen is also sometimes indicated as the blown type.

It should be observed that not all straight run bitumens belong to the normal type, nor all blown bitumens to the R type. There are blown bitumens of the Z type and the normal type, and both the Z type and the R type are found among the straight-run bitumens.

However, knowing the original penetration index of a certain asphalt, changes that take place when subjected to various treatment can be investigated. The calculated values of penetration index for the heat treated asphalts are shown in Table 10. The penetration index values vary between -1 and +1. All asphalt with penetration index values between -1 and +1 belong to the "normal asphalt" category of Pfeiffer's classification and are essentially steam refined asphalts. Asphalts therefore when heated in large quantities, at temperatures up to 325°F even for longer periods of time do not seem to bring basic chemical changes which will change their general classification based on penetration index values.

Table 10. Penetration index for asphalts before and after heat treatment

Treatment temperature OF	Treatment time in hrs.	Penetration index			
		A	B	C	D
—	0	+0.544	-0.425	-0.895	-0.614
325	2	+0.445	-0.229	-1.090	-0.895
325	4	+0.413	+0.067	-0.640	-0.744
325	8	+0.253	+0.007	-0.666	-0.769
325	16	+0.544	+0.252	-1.090	+0.007
325	24	+0.749	+0.316	-0.845	+0.186
325	72	+0.478	+0.612	-0.612	+0.159
230	4	+0.316	+0.067	-0.507	-0.560
230	8	+0.221	+0.316	-0.692	-0.341
230	16	+0.714	+0.611	-0.221	-0.397
230	24	+0.348	+0.997	-0.452	-0.397
230	72	+0.348	+0.997	-0.692	-0.285
230	120	+0.446	+0.819	-0.446	-0.083

Overall results indicate that heat treatment of these asphalts brought considerable changes in their physical and chemical properties. Changes in penetration were comparatively high. A loss up to 50% was observed in 85-100 and 150-200 penetration asphalts after 72 hours of heat treatment at 325^oF. Asphalt A (30-40 penetration grade) showed much less changes. Softening point, per cent asphaltenes,

and viscosity values for these treated asphalts at 325^oF showed considerable changes. However changes in the properties of asphalt heat treated at lower temperatures such as 230^oF showed comparatively less changes in properties. Volatilization of the light molecular weight hydrocarbons or their oxidation or dehydrogenation and polymerization to higher molecular weight hydrocarbons constituents are considered to be the cause of changes in properties of these heat treated asphalts. Higher changes at 325^oF than at 230^oF heat treatment indicate that all these reactions which bring changes in the physical and chemical properties of asphalts are accelerated at higher temperatures. Changes at lower temperature are comparatively small but longer heating periods seem to bring changes similar to those of higher temperatures at short heating periods. The characterizing factor remains unaffected by heat treatment, both at 230 and 325^oF, indicating that the paraffinic constituents of asphalts are not materially affected by heat treatments. Penetration index values of the heat treated asphalts vary between -1 and +1 but show an increasing trend toward positive values with increasing heat treatment time and temperature. Heat treatment of asphalts for longer periods of time at 325^oF or higher will affect the properties of the asphalt materially. However shorter periods of heating which are necessary do not seem to bring on material change. Heating of asphalts during actual construction of

highways therefore should be kept to a minimum necessary temperature to avoid changes in their physical and chemical properties.

Recovered Asphalt from Treated Asphaltic Concrete Mixes

Asphalts from uncompacted mixes, subjected to heat, ultra-violet radiation and oxidation treatments were recovered by the modified Abson method. The penetration, softening point, per cent asphaltenes, viscosity of the recovered asphalts and other related data are presented below.

Heat treatment

Penetration Penetration values obtained for all the recovered asphalts are given in Table 11 and shown graphically in Figure 7. All the asphalts recovered from mixes, treated at 325°F for 8 hours showed a loss of 75 to 87 per cent of the original penetration. Asphalts recovered from heat treated mixes at 230°F for 24 to 72 hours and at 140°F for 720 hours showed a loss of 50 to 70 per cent and 45 to 65 per cent respectively. Figures 8, 9 and 10 indicate that the loss in penetration of almost all the asphalts, recovered from heat treated asphaltic concrete mixes with aggregate Y is comparatively lower than the asphalts recovered from asphaltic concrete mixes with Aggregate X. The effects of aggregate on

Table 11. Penetration of asphalts recovered from heat treated loose asphaltic concrete mixes

Treat- ment temp. OF	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0 ^a	34	34	76	86	140	140
--	0 ^b	28	28	53	59	98	103
325	2	12	19	37	47	52	63
325	4	8	12	19	34	40	51
325	8	8	8	10	21	24	33
230	4	23	21	47	50	71	92
230	8	21	19	40	48	61	81
230	16	19	17	34	44	55	71
230	24	17	15	27	40	49	63
230	72	12	13	23	34	45	52
140	120	22	25	43	56	73	81
140	240	17	20	29	44	69	79
140	360	14	19	28	38	53	76
140	720	12	16	27	32	50	75

^aPenetration of asphalts before mixing with aggregate.

^bPenetration of asphalts recovered from mixes just after mixing.

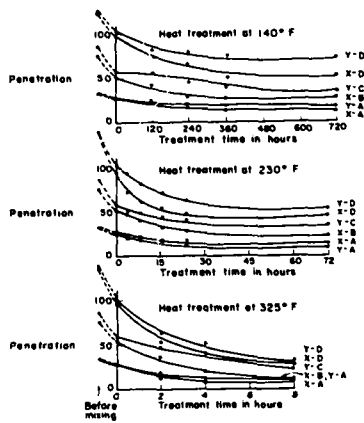


Figure 7. Penetration of asphalts recovered from heat treated asphaltic concrete mixes at 140, 230 and 325°F, showing penetration before mixing, after mixing and after heat treatments.

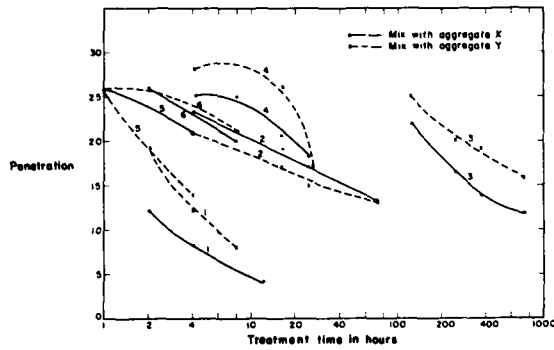


Figure 8. Penetration of asphalt A recovered from asphaltic concrete mixes that have been subjected to various forms of treatments.

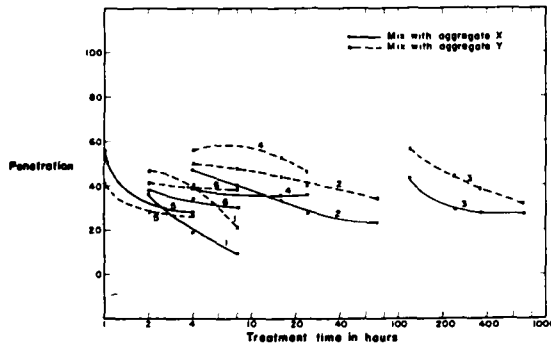


Figure 9. Penetration of asphalts B and C recovered from asphaltic concrete mixes that have been subjected to various forms of treatments.

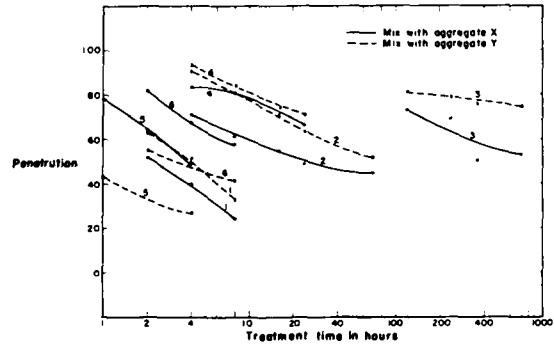


Figure 10. Penetration of asphalt D recovered from asphaltic concrete mixes that have been subjected to various forms of treatments.

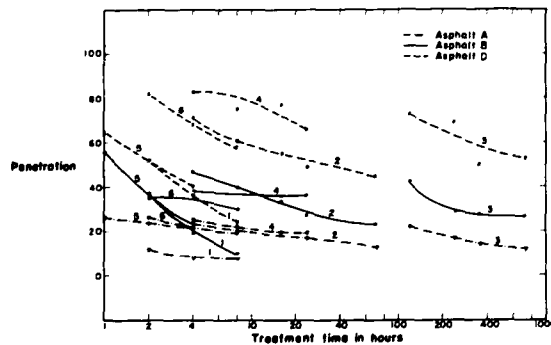


Figure 11. Penetration of asphalts A, B, and D recovered from asphaltic concrete mixes with aggregate X that have been subjected to various forms of treatments.

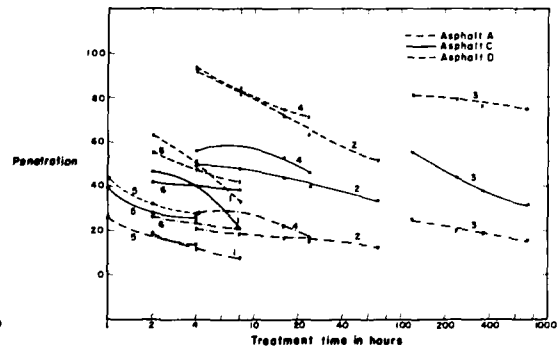


Figure 12. Penetration of asphalts A, C, and D recovered from asphaltic concrete mixes with aggregate Y that have been subjected to various forms of treatments.

the physical properties of recovered asphalts are discussed later.

Most of the highway departments require that the penetration of the recovered asphalt from asphaltic concrete mix samples taken during the construction period should have penetration not less than 50 per cent of the original value. Data show that, in order to have a loss in penetration of about 50 per cent, due to heat treatment of mixes in un-compacted state, it will take approximately 700 hours at 140°F, 16 to 24 hours at 230°F and 2 to 4 hours at 325°F. Much shorter periods can therefore be expected at higher temperatures for similar loss in penetration. These results indicate that the asphalt in pavement will therefore be hardened considerably if the asphaltic concrete mixes are subjected to higher temperatures even for very short periods, (such as mixing and handling time at higher temperatures). Lower temperatures for longer periods of time coupled with other field conditions also contribute to the hardening of asphalt in pavements. Figures 11 and 12 show the effect of different grades of asphalt (30-40, 85-100, 150-200) and two types of aggregate on the penetration of recovered asphalts from asphaltic concrete mixes subjected to various forms of treatments.

Softening point The softening points of recovered asphalts were obtained by the ring and ball method. The test

results so obtained are given in Table 12, and shown graphically in Figure 13. The per cent increase in softening point is 40 to 60 per cent for asphalts recovered from mixes treated for 8 hours at 325°F. Lower temperatures, however, show much less changes in softening point. There is 13 to 25 per cent increase after 24 hours and 18 to 30 per cent after 72 hours of heat treatment at 230°F. Asphalts recovered from mixes treated at 140°F for 72 hours show 9 to 33 per cent increase in softening point. Most of the asphalts recovered from asphaltic concrete mixes in which aggregate Y was used, show less increase in softening point than asphalts recovered from asphaltic concrete mixes with aggregate X. Comparative values of softening point for asphalts recovered from asphaltic concrete mixes "with X and Y aggregates but with same or similar asphalts", and "with same aggregate but with 30-40, 85-100, 150-200 penetration grade asphalts," subjected to various treatments are shown in Figures 14, 15, 16, 17 and 18 respectively.

Per cent asphaltenes The asphaltene content for all the asphalts was obtained by selective solvent method (21) and the results obtained are given in Table 13 and shown graphically in Figure 19. Asphalts recovered from heat treated asphaltic concrete mixes at 325°F for 8 hours showed 60 to 120 per cent increase in asphaltenes. Asphalts recovered from X-D and Y-D asphaltic concrete mixes showed the highest per cent

Table 12. Softening point of asphalts recovered from heat treated loose asphaltic concrete mixes

Treat- ment temp. OF	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X asphalt D	Aggre- gate Y, asphalt D
--	0 ^a	144	144	120	115	108	108
--	0 ^b	151	151	136	126	118	114
325	2	181	178	147	140	140	130
325	4	199	190	170	157	154	147
325	8	212	207	183	162	160	152
230	4	153	160	136	126	122	114
230	8	156	162	140	130	122	118
230	16	160	165	147	131	127	122
230	24	169	167	149	133	129	124
230	72	185	176	156	136	133	131
140	120	154	162	140	127	122	116
140	240	160	165	149	129	124	116
140	360	162	165	152	128	127	118
140	720	163	167	159	133	127	120

^aSoftening point of asphalts before mixing with aggregate.

^bSoftening point of asphalts recovered from mixes just after mixing.

Table 13. Per cent asphaltenes of asphalts recovered from heat treated loose asphaltic concrete mixes

Treat- ment temp. OF	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0 ^a	19.94	19.94	16.60	18.20	13.40	13.40
--	0 ^b	23.20	24.20	22.80	20.70	18.20	17.10
325	2	32.18	28.30	25.41	25.70	25.20	21.37
325	4	32.85	29.70	27.99	25.80	25.66	24.85
325	8	34.25	33.35	30.80	29.02	29.68	28.80
230	4	23.36	26.20	22.70	22.96	19.51	17.90
230	8	24.40	27.33	22.80	23.50	19.71	18.60
230	16	26.90	27.80	23.28	24.10	20.00	20.21
230	24	29.80	29.76	24.20	25.00	20.58	20.72
230	72	32.37	32.70	26.08	26.10	21.65	22.10
140	120	26.36	25.90	22.78	22.40	19.14	18.48
140	240	27.74	26.20	23.20	22.43	20.20	18.98
140	360	28.08	26.30	23.63	22.50	20.65	19.50
140	720	28.80	27.88	24.25	24.30	21.00	19.50

^aPer cent asphaltenes of asphalts before mixing with aggregate.

^bPer cent asphaltenes of asphalts recovered from mixes just after mixing.

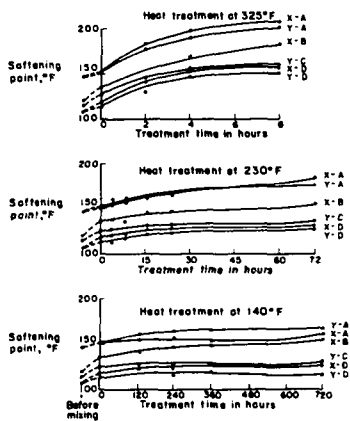
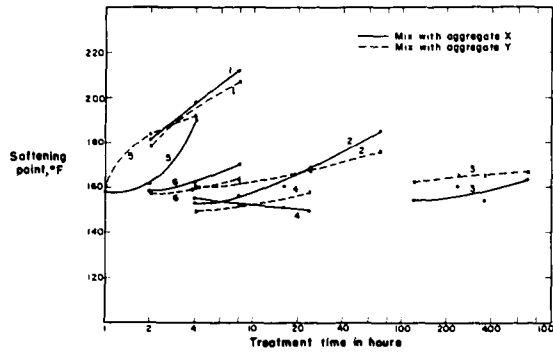


Figure 13. Softening point of asphalt A recovered from heat treated asphaltic concrete mixes at 140, 230 and 324°F. showing softening point before mixing, after mixing and after heat treatments.



- 1 - Heat treatment at 325°F.
- 2 - Heat treatment at 230°F.
- 3 - Heat treatment at 140°F.
- 4 - Ultra violet radiation treatment.
- 5 - Oxidation at 325°F.
- 6 - Oxidation at 230°F.

Figure 14. Softening point of asphalt A recovered from asphaltic concrete mixes that have been subjected to various forms of treatments.

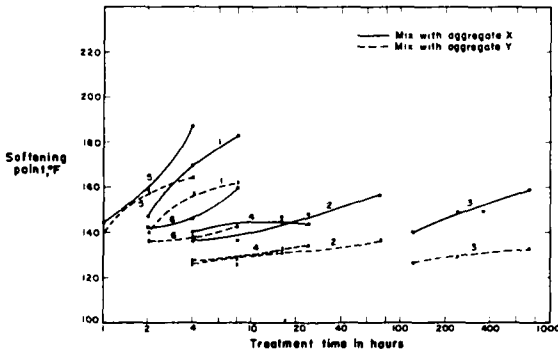


Figure 15. Softening point of asphalt B and C recovered from asphaltic concrete mixes that have been subjected to various forms of treatments.

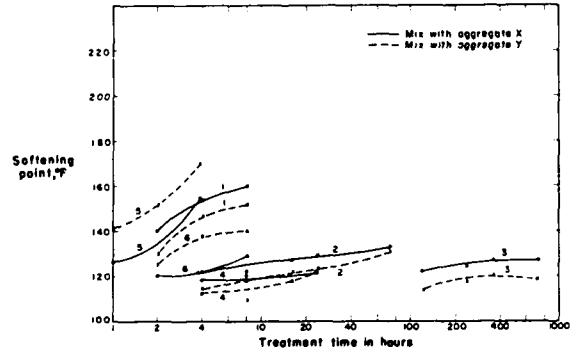


Figure 16. Softening point of asphalt D recovered from asphaltic concrete mixes that have been subjected to various forms of treatments.

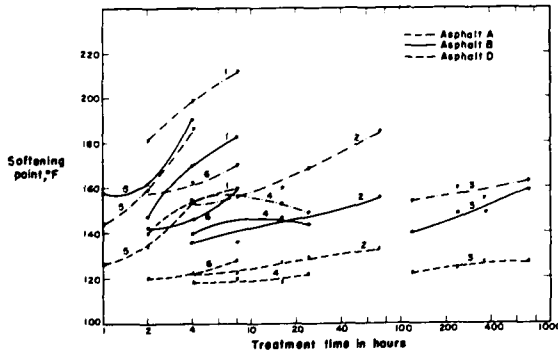


Figure 17. Softening point of asphalt A, B, and D recovered from asphaltic concrete mixes with aggregate X that have been subjected to various forms of treatments.

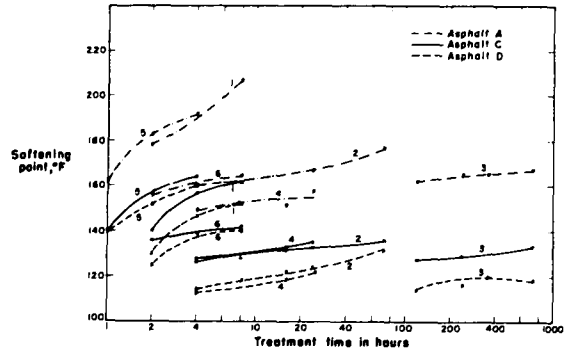


Figure 18. Softening point of asphalt A, C, and D recovered from asphaltic concrete mixes with aggregate Y that have been subjected to various forms of treatments.

increase in asphaltenes. This may be due to the fact that asphalt D, a 150-200 penetration grade has higher percentages of asphaltic resins and oily constituents which change to asphaltenes during oxidation or dehydrogenation reactions at higher temperatures. Asphalts recovered from mixes treated at 230^oF for 24 and 72 hours showed 37 to 55 per cent and 43 to 65 per cent increases respectively. Similar heat treatment of asphaltic concrete mixes at 140^oF showed 35 to 57 per cent increase after 720 hours. The asphalts recovered from asphaltic concrete mixes with aggregate X showed higher per cent increases in asphaltene content than asphalts recovered from asphaltic concrete mixes with aggregate Y. Lower absorption characteristics of aggregate X are considered responsible for higher asphaltene content. Details of the effect of aggregate characteristics on asphaltene content of recovered asphalts are discussed later. Comparative values of asphaltene content for recovered asphalts from asphaltic concrete mixes with "different aggregates and same or similar asphalts" and "same aggregate but with 30-40, 85-100, 150-200 penetration grade asphalts" subjected to various treatments are shown in Figures 20, 21, 22, 23 and 24 respectively.

Viscosity Viscosity values in poises for all the recovered asphalts were obtained by microviscometer and are presented in Table 14 and shown graphically in Figure 25.

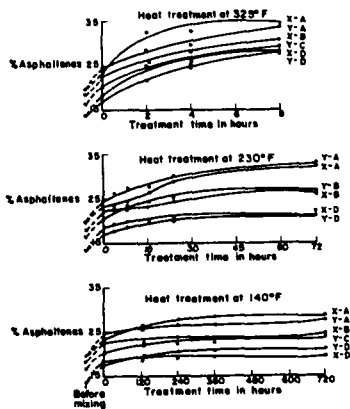
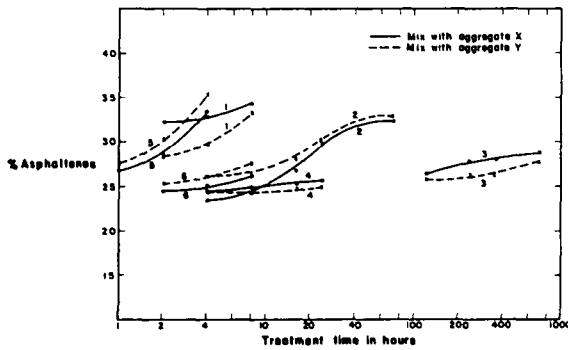


Figure 19. Percent asphaltenes of asphalts recovered from heat treated asphaltic concrete mixes at 140, 230 and 325°F. showing percent asphaltenes before mixing, after mixing, and after heat treatments.



- 1 - Heat treatment at 325°F.
- 2 - Heat treatment at 230°F.
- 3 - Heat treatment at 140°F.
- 4 - Ultra violet radiation treatment.
- 5 - Oxidation at 325°F.
- 6 - Oxidation at 230°F.

Figure 20. Percent asphaltenes of asphalt A recovered from asphaltic concrete mixes that have been subjected to various forms of treatments.

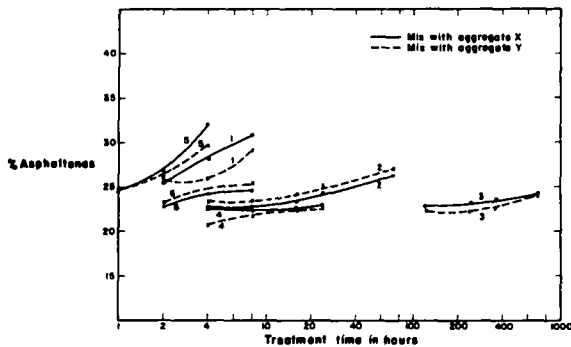


Figure 21. Percent asphaltenes of asphalts B and C recovered from asphaltic concrete mixes that have been subjected to various forms of treatments.

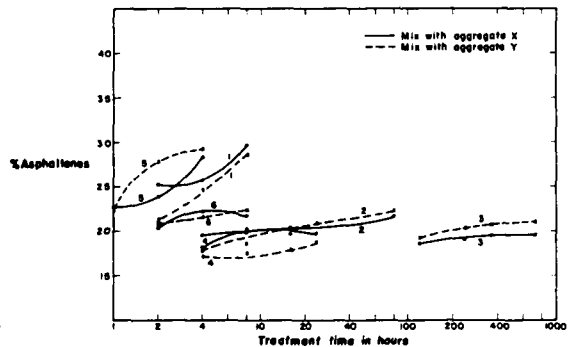


Figure 22. Percent asphaltenes of asphalt D recovered from asphaltic concrete mixes that have been subjected to various forms of treatments.

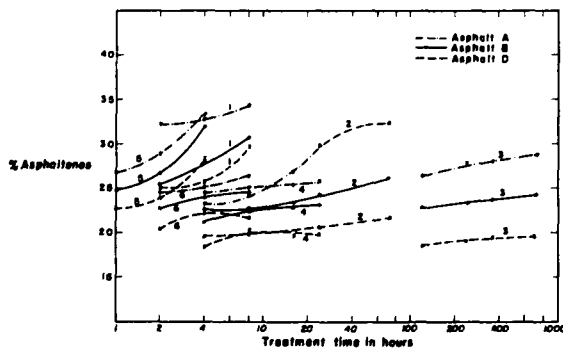


Figure 23. Percent asphaltenes of asphalts A, B, and D recovered from asphaltic concrete mixes with aggregate X that have been subjected to various forms of treatments.

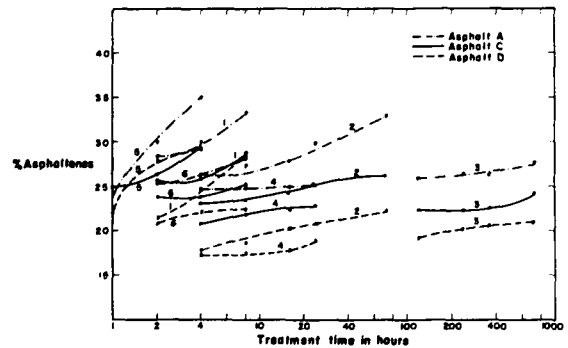


Figure 24. Percent asphaltenes of asphalts A, C, and D recovered from asphaltic concrete mixes with aggregate Y that have been subjected to various forms of treatments.

Table 14. Viscosity in 10^6 poises of asphalts recovered from heat treated loose asphaltic concrete mixes

Treat- ment temp. °F.	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0 ^a	11.8	11.8	4.5	2.25	0.45	0.45
--	0 ^b	20.0	20.5	10.2	5.80	3.8	4.4
325	2	--	--	14.5	14.0	8.8	9.9
325	4	--	--	20.0	20.5	18.5	14.3
325	8	--	--	32.0	30.0	23.5	22.5
230	4	32.0	20.8	11.0	6.6	3.8	4.6
230	8	--	22.0	11.6	6.8	4.2	4.6
230	16	--	24.0	13.5	6.9	4.7	4.8
230	24	--	--	15.0	7.4	7.2	4.9
230	72	--	--	17.0	9.2	8.6	7.8
140	120	21.5	22.2	14.5	6.9	3.4	5.1
140	240	24.0	23.5	17.0	7.4	4.1	5.8
140	360	27.0	25.3	17.5	9.4	5.2	6.2
140	720	29.0	27.4	20.0	11.0	5.5	6.4

^aViscosity of asphalts before mixing with aggregate.

^bViscosity of asphalts recovered from mixes just after mixing.

Recovered asphalts B, C, and D from heat treated asphaltic concrete mixes at 325°F for 8 hours showed viscosity increases about 8, 15 and 50 times the original viscosity. Determination of the viscosity of the asphalts recovered from asphaltic concrete mixes containing asphalt A, (30-40 penetration grade) and treated at 230 and 325°F, was very difficult. At lower temperatures, i.e. 230 and 140°F, the viscosity increases were comparatively small. Increases up to 5 fold, however were observed for asphalts recovered from the asphaltic concrete mixes made with B, and C asphalts and up to 15 times in asphalts recovered from asphaltic concrete mixes with asphalt D, after heat treatment at 230°F for 72 hours and at 140°F for 720 hours. Comparative values of viscosity of recovered asphalts from mixes "with different aggregates and same asphalt" and from mixes "with same aggregate but with 30-40, 85-100, 150-200 penetration grade asphalts" subjected to various treatments are shown in Figures 26, 27, 28, 29 and 30 respectively.

Characterizing factor Kinnaird's characterizing factor should remain unchanged for asphalts from the same stock and for the same asphalt after treatments which bring no change in the paraffinic constituents. The original asphalt when subjected to heat treatment showed that this factor remains constant even after heat treatment at 230 and 325°F for

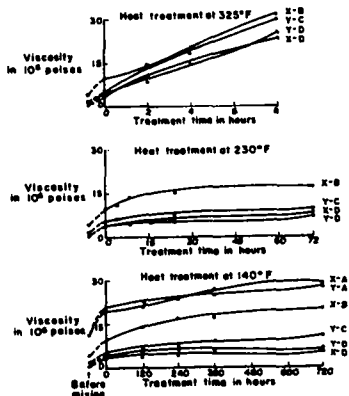
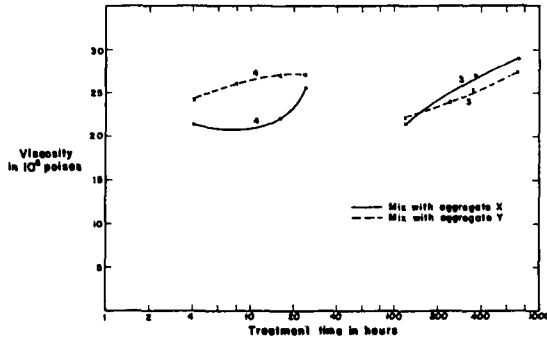


Figure 25. Viscosity of asphalts recovered from heat treated asphaltic concrete mixes at 140, 230 and 325°F. showing viscosity before mixing, after mixing and after heat treatments.



- 1 - Heat treatment at 325°F.
- 2 - Heat treatment at 230°F.
- 3 - Heat treatment at 140°F.
- 4 - Ultra violet radiation treatment.
- 5 - Oxidation at 325°F.
- 6 - Oxidation at 230°F.

Figure 26. Viscosity of asphalt A recovered from asphaltic concrete mixes that have been subjected to various forms of treatments.

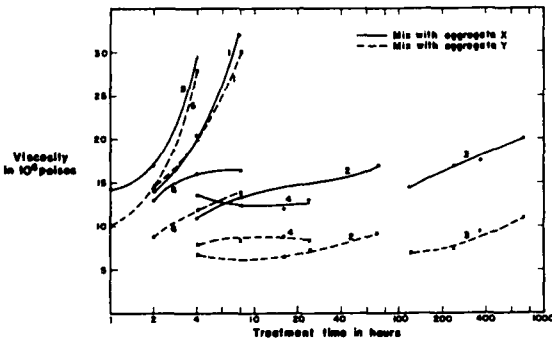


Figure 27. Viscosity of asphalts B and C recovered from asphaltic concrete mixes that have been subjected to various forms of treatments.

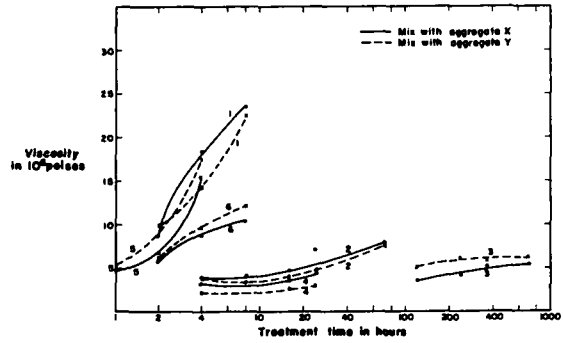


Figure 28. Viscosity of asphalt D recovered from asphaltic concrete mixes that have been subjected to various forms of treatments.

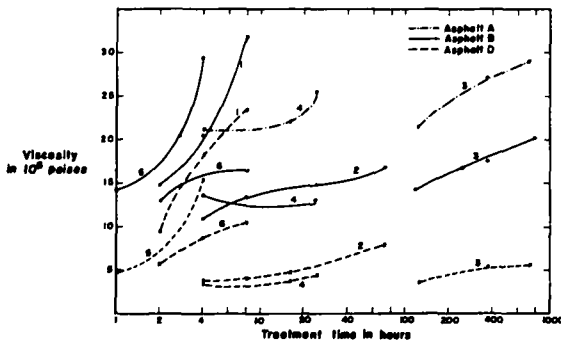


Figure 29. Viscosity of asphalts A, B, and D recovered from asphaltic concrete mixes with aggregate X that have been subjected to various forms of treatments.

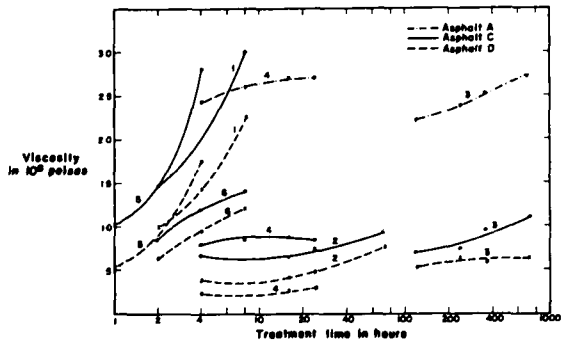


Figure 30. Viscosity of asphalts A, C, and D recovered from asphaltic concrete mixes with aggregate Y that have been subjected to various forms of treatments.

longer periods of time, within experimental accuracy. The values of the characterizing factor obtained from the nomograph (34) for recovered asphalts are given in Table 15. These values are not constant and do not show any definite trend. The asphalts recovered from asphaltic concrete mixes prepared with asphalt A, B, C and D and treated at 325°F up to 8 hours, 230°F up to 72 hours and 140°F up to 720 hours, have the characterizing factor values ranging between 12-20, 11-23, 11-25, 17-33 and 12-18, 15-25, 15-18, 17-30 and 9-18, 15-23, 15-22, 16-40 respectively. The characterizing factors for original asphalts A, B, C, and D are 18, 22, 20, and 27 respectively. Such an inconsistency in values indicate that the asphalts after recovery do not necessarily remain in the same original physical and chemical state. Aggregate absorption, treatment conditions and recovery procedure affect the quality of the recovered asphalts. Dust content in recovered asphalts although negligible percentwise could contribute a lot in bringing changes in their physical characteristics.

Penetration index The calculated values of the penetration index are given in Table 16. Asphalts recovered from asphaltic concrete mixes prepared with asphalt A, after heat treatment for 4 hours or more at 325°F results in the penetration index values greater than 1, thereby classifying these asphalts in the category referred to as "R type" by

Table 15. Characterizing factor for asphalts recovered from heat treated loose asphaltic concrete mixes

Treat- ment temp. OF	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0 ^a	18	18	22	20	27	27
--	0 ^b	17	17	25	20	30	25
325	2	12	20	23	25	27	26
325	4	11	15	17	25	29	33
325	8	14	13	11	16	18	23
230	4	15	15	22	15	22	20
230	8	14	14	20	17	16	21
230	16	14	13	20	16	20	22
230	24	15	12	16	15	17	20
230	72	14	15	15	13	18	22
140	140	14	20	22	19	22	18
140	140	12	16	17	15	23	16
140	140	10	15	18	11	18	18
140	140	9	13	20	11	16	21

^aCharacterizing factor for asphalts before mixing with aggregate.

^bCharacterizing factor for asphalts recovered from mixes just after mixing.

Table 16. Penetration index for asphalts recovered from heat treated loose asphaltic concrete mixes

Treat- ment temp. OF	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0 ^a	+0.544	+0.544	-0.425	-0.895	-0.614	-0.614
--	0 ^b	+0.818	+0.818	+0.714	-0.757	+0.006	-0.534
325	2	+1.650	+2.249	+1.033	+0.889	+1.144	+0.445
325	4	+2.116	+2.249	+1.650	+1.815	+1.900	+1.815
325	8	+2.864	+2.573	+1.489	+1.219	+1.334	+1.357
230	4	+0.612	+1.033	+0.446	-0.666	-0.342	-0.871
230	8	+0.714	+1.033	+0.478	-0.257	-0.718	-0.587
230	16	+0.853	+1.070	+0.854	-0.314	-0.314	-0.285
230	24	+1.411	+0.997	+0.570	-0.285	-0.314	-0.370
230	72	+1.900	+1.411	+0.889	-0.285	-0.023	+0.067
140	120	+0.612	+1.569	+0.067	-0.257	-0.260	-0.920
140	240	+0.645	+1.372	+0.714	-0.561	-0.120	-0.969
140	360	+0.478	+1.253	+0.924	-0.994	-0.397	-0.795
140	720	+0.316	+1.106	1.489	-0.769	-0.507	-0.257

^aPenetration index for asphalts before mixing with aggregate.

^bPenetration index for asphalts recovered from mixes just after mixing.

Pfeiffer, which are characterized by slight brittleness. All the other asphalts recovered from mixes treated at 230 and 140°F with a few exceptions have the penetration index value between -1 and 1 thereby classifying them in the category of normal asphalts, same as the original asphalts. The over-all penetration index data indicate that maintaining asphaltic concrete mixes at temperature over 300°F even for short periods of time, materially affect the physical and chemical properties of the asphalt. However treatment at lower temperatures even for longer periods of time do not seem to change the asphalt classification based on the penetration index values.

Asphalts recovered from asphaltic concrete mixes subjected to heat, ultraviolet radiation and oxidation treatments showed considerable changes in their physical and chemical properties. The per cent losses in penetration were very high. Asphalts recovered from heat treated mixes at 325°F for 8 hours brought the penetration of A, B, C and D asphalts down to 8, 10, 21 and 24-33 as compared to the original penetration of 34, 76, 86, and 140 respectively. Other properties such as softening point, per cent asphaltenes and viscosity of these recovered asphalts were considerably changed. Per cent asphaltenes showed between 60 and 120 per cent increase and viscosity 8 to 40 times the original values in

most of the asphalts recovered from the six types of asphaltic concrete mixes used in this investigation. Per cent change in the properties of recovered asphalts from mixes heat treated at 230 and 325^oF for 4 and 8 hours are shown in Figures 56 and 57 respectively. Mixes with softer asphalts showed comparatively higher per cent changes in recovered asphalts than the ones recovered from mixes with lower penetration asphalts. All these changes might be attributed to the following causes:

1. Changes that might have occurred if the asphalts were heated alone in thin films for the same time as the mixes.
2. Changes if any, which could have taken place during the recovery of the asphalt from asphaltic concrete mixes.
3. Changes occurring during the mixing of aggregate and asphalts at mixing temperatures.
4. Changes which are totally due to the treatment.

Examination of the data shows that major parts of these changes in the properties of the recovered asphalts are due primarily to the treatment especially at high temperatures. When the asphaltic concrete mixes are subjected to different treatments, some constituents of asphalts are absorbed in the pores of the aggregate and the rest of the asphalt surrounding the mineral matter still remains open to the reactions started or accelerated by the treatment conditions. Volatilization of the light molecular weight hydrocarbons or their

conversion to high molecular weight hydrocarbons by chemical reactions such as oxidation or dehydrogenation or polymerization seems to be the possible causes which bring changes in the properties changes noted in the recovered asphalts. High temperature alone or with oxygen bring the highest possible changes in the recovered asphalts even when these mixes are treated for short periods of time. Treatment of these mixes at lower temperatures such as 230 and 140^oF for short periods of time do not seem to bring appreciable changes in the properties of the recovered asphalts. However treatment at the same temperatures but for a long period of time seems to bring changes similar to the ones brought by high temperature treatment for shorter periods.

Ultra-violet radiation treatment

Exposure of the asphaltic concrete mixes to ultra-violet radiation affected the properties of recovered asphalts. The penetration, softening point, per cent asphaltenes and viscosity, of these recovered asphalts are given in Tables 17, 18, 19, and 20, and shown graphically in Figures 31, 32, 33 and 34 respectively. Penetration of all the recovered asphalts showed up to 50 per cent loss during 24 hours of radiation treatment time. Softening point is not much affected percentwise by the ultra-violet radiation treatment, however softening point increased with decrease in penetration

Table 17. Penetration of asphalts recovered from ultra-violet radiation treated loose asphaltic concrete mixes

Type of treatment	Treatment time (hrs.)	Aggregate X, asphalt A	Aggregate Y, asphalt A	Aggregate X, asphalt B	Aggregate Y, asphalt C	Aggregate X, asphalt D	Aggregate Y, asphalt D
--	0 ^a	34	34	76	86	140	140
--	0 ^b	28	28	53	59	98	103
UVR ^c	4	25	28	39	56	83	93
UVR	8	25	30	32	56	75	84
UVR	16	22	26	36	53	77	74
UVR	24	18	17	36	46	66	71

^aPenetration of asphalts before mixing with aggregate.

^bPenetration of asphalts recovered from mixes just after mixing.

^cUltra-violet radiation.

Table 18. Softening point of asphalts recovered from ultra-violet radiation treated loose asphaltic concrete mixes

Type of treat-ment	Treat-ment time (hrs.)	Aggre-gate X, Asphalt A	Aggre-gate Y, asphalt A	Aggre-gate X, asphalt B	Aggre-gate Y, asphalt C	Aggre-gate X, asphalt D	Aggre-gate Y, asphalt D
--	0 ^a	144	144	120	115	108	108
--	0 ^b	151	151	136	126	118	114
UVR ^c	4	153	149	140	126	118	113
UVR	8	153	151	138	128	120	116
UVR	16	154	152	145	133	120	118
UVR	24	159	158	144	133	122	122

^aSoftening point of asphalts before mixing with aggregate.

^bSoftening point of asphalts recovered from mixes just after mixing.

^cUltra-violet radiation.

Table 19. Per cent asphaltenes of asphalts recovered from ultra-violet radiation treated loose asphaltic concrete mixes

Type of treatment	Treat- time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0 ^a	19.94	19.94	16.60	18.20	13.40	13.40
--	0 ^b	23.20	24.20	22.80	20.70	18.20	17.10
UVR ^c	4	24.50	24.51	22.76	21.20	18.65	17.50
UVR	8	25.00	24.79	22.40	21.70	19.55	17.79
UVR	16	25.13	24.83	22.88	22.39	19.82	18.76
UVR	24	25.62	25.00	23.00	22.80	20.18	19.28

^aPer cent asphaltenes of asphalts before mixing with aggregate.

^bPer cent asphaltenes of asphalts recovered from mixes just after mixing.

^cUltra-violet radiation.

Table 20. Viscosity in 10^6 poises of asphalts recovered from ultra-violet radiation treated loose asphaltic concrete mixes

Type of treatment	Treatment time hrs.	Aggregate X, asphalt A	Aggregate Y, asphalt A	Aggregate X, asphalt B	Aggregate Y, asphalt C	Aggregate X, asphalt D	Aggregate Y, asphalt D
--	0 ^a	11.8	11.8	4.5	2.25	0.45	0.45
--	0 ^b	20.0	20.5	10.2	5.8	3.8	4.4
UVR ^c	4	21.5	22.3	12.0	6.4	3.8	2.15
UVR	8	22.0	26.0	12.5	8.3	3.6	2.24
UVR	16	22.0	27.0	13.2	8.3	3.9	2.54
UVR	24	25.5	27.0	13.5	8.8	4.3	3.0

^aViscosity of asphalts before mixing with aggregate.

^bViscosity of asphalts recovered from mixes just after mixing.

^cUltra-violet radiation.

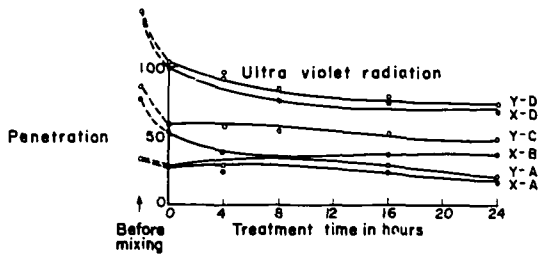


Figure 31. Penetration of asphalts recovered from asphaltic concrete mixes that have been subjected to ultra violet radiation treatment.

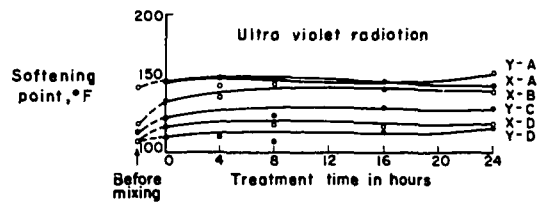


Figure 32. Softening point of asphalts recovered from asphaltic concrete mixes that have been subjected to ultra violet radiation treatment.

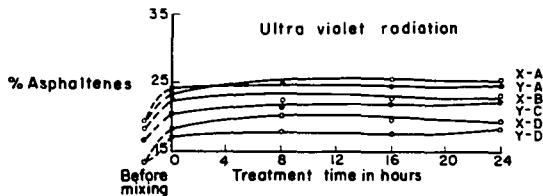


Figure 33. Percent asphaltenes of asphalts recovered from asphaltic concrete mixes that have been subjected to ultra violet radiation treatment.

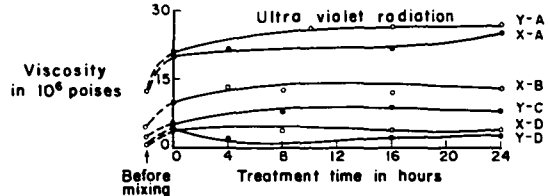


Figure 34. Viscosity of asphalts recovered from asphaltic concrete mixes that have been subjected to ultra violet radiation treatment.

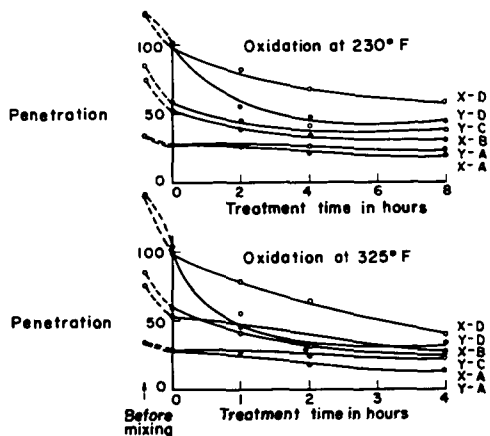


Figure 35. Penetration of asphalts recovered from asphaltic concrete mixes that have been subjected to oxidation treatment at 230 and 325 °F.

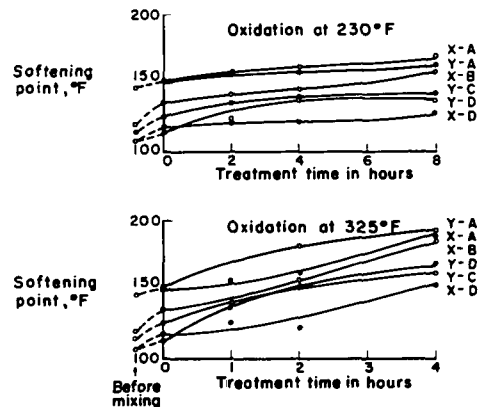


Figure 36. Softening point of asphalts recovered from asphaltic concrete mixes that have been subjected to oxidation treatment at 230 and 325 °F.

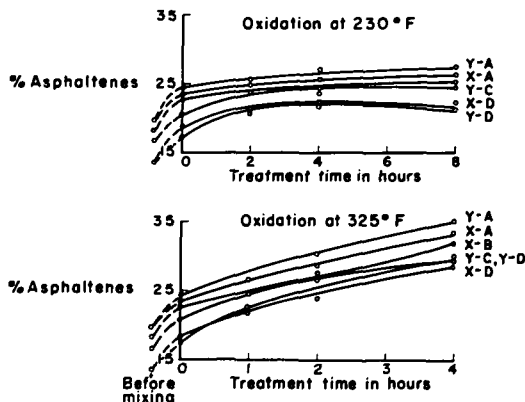


Figure 37. Percent asphaltenes of asphalts recovered from asphaltic concrete mixes that have been subjected to oxidation treatment at 230 and 325 °F.

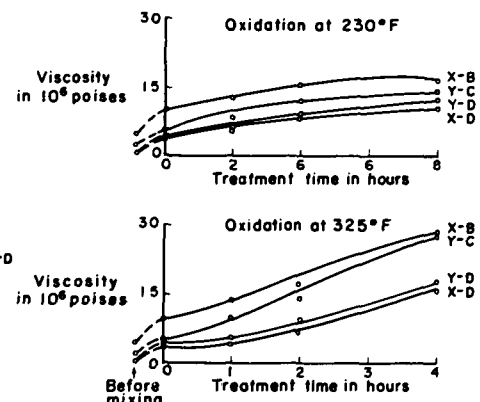


Figure 38. Viscosity of asphalts recovered from asphaltic concrete mixes that have been subjected to oxidation treatment at 230 and 325 °F.

value. The overall increase in softening point varied between 2 to 20 per cent. Asphalts recovered from asphaltic concrete mixes in which asphalt B and C of 85-100 penetration grade were used showed comparatively higher increase in the softening point than those in which asphalt A (30-40) and asphalt D (150-200) penetration grade were used. A maximum of 13 per cent increase in softening point was obtained in asphalts recovered from asphaltic concrete mixes in which asphalt D was used. The per cent increase in asphaltene content varied between 25 and 50 per cent after 24 hours of ultra-violet radiation exposure time. The asphalt D mixes showed the greatest increase in asphaltenes. Asphalts recovered from asphaltic concrete mixes in which aggregate Y was used showed lower per cent increases in asphaltenes, softening point and less loss in penetration, than the asphalts recovered from asphaltic concrete mixes with aggregate X. Viscosity values show about 2 to 5 fold increase in all the asphalts, with highest increase in the asphalts recovered from asphaltic concrete mixes with asphalt D and lowest in asphalts recovered from asphaltic concrete mixes with asphalt A. The changes brought about by the ultra-violet radiation are coupled with heating at 140°F, as the radiation developed that much heat to raise the temperature of the mixture to 140°F. The characterizing factor values for the recovered asphalts from mixes subjected to ultra-violet radiation treatment are given in

Table 21. These values are not the same as for the original asphalts before mixing with the aggregate. These values show no consistency as far as their increase and decrease is concerned. The penetration index values for all these asphalts are given in Table 22. These values vary between -1 and +1, thereby indicating that the Pfeiffer's classification of asphalts based on the penetration index remains unchanged even after 24 hours of ultra-violet radiation treatment.

The over all data for the recovered asphalts from mixes subjected to ultra-violet radiation treatment for short periods of time does indicate that such radiation does not seem to have much detrimental effect on the properties of the recovered asphalts. However, long periods of ultra-violet radiation treatment of mixes will materially affect the properties of the recovered asphalts.

Oxidation treatment

Asphalts recovered from oxidized asphaltic concrete mixes at 230 and 325°F showed great changes in their physical and chemical properties. The penetration, softening point, per cent asphaltenes and viscosity data is given in Tables 23, 24, 25, 26 and shown graphically in Figures 35, 36, 37 and 38 respectively. Asphalts recovered from mixes oxidized for four hours at 325°F showed 40 to 80 percent loss in penetration, 30 to 60 percent increase in softening point, 60 to

Table 21. Characterizing factor for asphalts recovered from ultra-violet radiation treated loose asphaltic concrete mixes

Type of treatment	Treatment time hrs.	Aggregate X, asphalt A	Aggregate Y, asphalt A	Aggregate X, asphalt B	Aggregate Y, asphalt C	Aggregate X, asphalt D	Aggregate Y, asphalt D
--	0 ^a	18	18	22	20	27	27
--	0 ^b	17	17	25	20	30	25
UVR ^c	4	16	16	19	18	22	18
UVR	8	16	19	13	19	20	19
UVR	16	14	16	20	23	21	17
UVR	24	12	11	20	18	19	22

^aCharacterizing factor for asphalts before mixing with aggregate.

^bCharacterizing factor for asphalts recovered from mixes just after mixing.

^cUltra-violet radiation.

Table 22. Penetration index for asphalts recovered from ultra-violet radiation treated loose asphaltic concrete mixes

Type of treatment	Treatment time hrs.	Aggregate X, asphalt A	Aggregate Y, asphalt A	Aggregate X, asphalt B	Aggregate Y, asphalt C	Aggregate X, asphalt D	Aggregate Y, asphalt D
--	0 ^a	+0.544	+0.544	-0.425	-0.895	-0.614	-0.614
--	0 ^b	+0.818	+0.818	+0.714	-0.257	+0.006	-0.533
UVR ^c	4	+0.784	+0.645	+0.445	-0.397	-0.506	-1.018
UVR	8	+0.784	+0.961	+0.348	-0.113	-0.479	-0.795
UVR	16	+0.612	+0.784	+0.784	+0.348	-0.397	-0.821
UVR	24	+0.680	+0.479	+0.679	+0.037	-0.534	-0.341

^aPenetration index for asphalts before mixing with aggregate.

^bPenetration index for asphalts recovered from mixes just after mixing.

^cUltra-violet radiation.

Table 23. Penetration of asphalts recovered from oxidized loose asphaltic concrete mixes

Treat- ment temp. °F	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0 ^a	34	34	76	86	140	140
--	0 ^b	28	28	53	59	98	103
325	1	26	24	56	41	79	43
325	2	24	18	38	28	64	32
325	4	21	14	28	27	39	27
230	2	26	28	38	42	82	65
230	4	22	24	35	40	68	48
230	8	20	21	30	39	58	42

^aPenetration of asphalts before mixing with aggregate.

^bPenetration of asphalts recovered from mixes just after mixing.

Table 24. Softening point of asphalts recovered from oxidized loose asphaltic concrete mixes

Treat- ment temp. °F	Treat- ment time hrs.	Aggre- gate X asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0 ^a	144	144	120	115	108	108
--	0 ^b	151	151	136	126	118	114
325	1	158	162	144	140	126	140
325	2	161	183	159	164	134	152
325	4	190	191	187	164	154	160
230	2	158	156	142	136	120	125
230	4	162	160	146	138	122	138
230	8	170	164	160	142	128	140

^aSoftening point of asphalts before mixing with aggregate.

^bSoftening point of asphalts recovered from mixes just after mixing.

Table 25. Per cent asphaltenes of asphalts recovered from oxidized loose asphaltic concrete mixes

Oxida- tion temp. °F	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0 ^a	19.94	19.94	16.60	18.20	13.40	13.40
--	0 ^b	23.20	24.20	22.80	20.70	18.20	17.10
325	1	26.61	27.20	24.66	24.82	22.73	22.23
325	2	28.69	30.38	26.59	26.19	23.89	27.86
325	4	33.44	35.06	31.98	29.56	28.24	29.40
230	2	24.43	25.20	23.71	23.93	20.50	20.74
230	4	25.16	26.07	24.02	23.74	21.73	21.98
230	8	26.29	27.53	24.47	25.31	22.16	22.43

^aPer cent asphaltenes of asphalt before mixing with aggregate.

^bPer cent asphaltenes of asphalts recovered from mixes just after mixing.

Table 26. Viscosity in 10^6 poises of asphalts recovered from oxidized loose asphaltic concrete mixes

Oxi- dation temp. °F	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0 ^a	11.80	11.80	4.50	2.25	0.45	0.45
--	0 ^b	20.00	20.50	10.20	5.80	3.80	4.40
325	1	--	--	14.30	10.00	4.80	5.10
325	2	--	--	17.00	14.20	6.90	9.40
325	4	--	--	29.50	28.00	15.50	17.30
230	2	--	--	13.00	8.70	5.90	6.20
230	4	--	--	16.00	12.00	8.62	9.60
230	8	--	--	16.50	14.00	10.50	12.30

^aViscosity of asphalts before mixing with aggregate.

^bViscosity of asphalts recovered from mixes just after mixing.

120 per cent increase in per cent asphaltenes and up to 40 fold increase in the viscosity values. Per cent changes in properties were comparatively much higher for asphalts recovered from asphaltic concrete mixes oxidized at 325°F, than for asphalts recovered from the heat treated mixes at the same temperature and for the same duration. Asphalts recovered from oxidized mixes at 230°F up to 8 hours were not too severe. Changes in physical and chemical properties of asphalts recovered from mixes oxidized at 230°F and heat treated at 230°F for same duration did not show much difference. Moreover these changes in properties were very low as compared to the changes in the properties of asphalts recovered from oxidized mixes at 325°F. Asphalts recovered from mixes with aggregate Y and oxidized at 325°F and mixes with aggregate Y and asphalt D oxidized at 230°F showed comparatively high changes in properties than the asphalts recovered from similarly treated mixes with aggregate X. Whereas asphalts recovered from mixes with aggregate X and asphalts A, and B oxidized at 230°F showed greater changes than the asphalts recovered from similarly treated mixes with aggregate Y.

Characterizing factor values for these asphalts are given in Table 27. These values are not the same as those of the original asphalts. No definite trend is shown by these values, indicating that the recovered asphalts are not the

Table 27. Characterizing factor for asphalts recovered from oxidized loose asphaltic concrete mixes

Oxidation temp. °F	Treatment time hrs.	Aggregate X, asphalt A	Aggregate Y, asphalt A	Aggregate X, asphalt B	Aggregate Y asphalt C	Aggregate X, asphalt D	Aggregate Y, asphalt D
--	0 ^a	18	18	22	20	27	27
--	0 ^b	17	17	25	20	30	25
325	1	19	19	34	20	31	22
325	2	18	20	30	23	31	22
325	4	25	17	32	22	27	20
230	2	19	20	20	18	24	22
230	4	16	18	21	18	20	23
230	8	18	16	23	21	21	22

^aCharacterizing factor for asphalts before mixing with aggregate.

^bCharacterizing factor for asphalts recovered from mixes just after mixing.

same chemically as the original asphalts. Penetration index values for these recovered asphalts are given in Table 28. Asphalts recovered from mixes oxidized at 325^oF showed penetration index values greater than 1 thereby classifying them in the category referred to as "R type" by Pfeiffer which are characterized by slight brittleness. All the asphalts recovered from mixes oxidized at 230^oF for 8 hours except the ones recovered from mixes with asphalt A, have penetration index values between -1 and +1 and thus belong to the "normal type" class, same as the original asphalts. Asphalts recovered from mixes with asphalt A and oxidized at 230^oF have penetration index values greater than 1 and thus belong to the "R type" class of Pfeiffer's classification.

Stability and Cohesion of the Treated Asphaltic Concrete Mixes

The essential properties required of bituminous paving mixtures comprise stability, durability, flexibility and workability during construction operation. A durable bituminous pavement must withstand detrimental effects of traffic, water, air and temperature. During this investigation various treatments of asphaltic concrete mixes in an uncompacted state showed tremendous changes in the physical and chemical properties of the recovered asphalts. Such changes in asphalt are detrimental to the durability of the pavements. The stability

and cohesion of these treated asphaltic concrete mixes were therefore studied.

Hveem stability

Heat treatment Loose asphaltic concrete mixes were subjected to heat treatment at 325, 230 and 140^oF, ultra-violet radiation and oxidation treatments as mentioned under "treatment of mixes for stability and cohesion" on page 35. These treated mixes were heated to compaction temperatures, i.e. 250^oF and specimens 4 inches in diameter and 2.5 inches in height were compacted by static load double plunger method. The Hveem stability values "S" and the lateral stress values corresponding to a vertical pressure of 400 PSI as used by the Iowa Highway Commission are given in Tables 29 and 30, respectively. The Hveem stability values "S" are also shown graphically in Figure 39. The specified stability "S" values for both medium and heavily traveled bituminous concrete pavements is 35 minimum (44). All the six asphaltic concrete mixes used in this study had stability values greater than 35. The Iowa Highway Commission requires that type A asphaltic concrete mixes should develop lateral pressure of less than 60 psi, when tested in the Hveem stabilometer at 140^oF. All the asphaltic concrete mixes used in this study satisfy such a requirement. A quick look at the stability data indicates a very high increase in stability due to various treatments.

Table 28. Penetration index for asphalts recovered from oxidized loose asphaltic concrete mixes

Oxida- tion temp. °F	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0 ^a	+0.544	+0.544	-0.425	-0.895	-0.614	-0.614
--	0 ^b	+0.818	+0.818	+0.714	-0.257	+0.006	-0.533
325	1	1.30	+1.489	+1.774	+0.540	+0.545	+0.650
325	2	1.41	+2.480	+2.250	+1.985	+2.111	+1.257
325	4	3.27	+2.570	+3.686	+1.900	+1.860	+1.570
230	2	+1.30	+1.295	+0.578	+0.159	-0.230	-0.141
230	4	+1.30	+1.33	+0.819	+0.284	-0.450	+0.710
230	8	+1.77	+1.37	+1.77	+0.650	-0.020	+0.610

^aPenetration index for asphalts before mixing with aggregate.

^bPenetration index for asphalts recovered from mixes just after mixing.

Table 29. Hveem stabilometer "S" values for heat treated loose asphaltic concrete mixes

Treat- ment temp. OF	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0	56	52	54	45	41	40
325	2	57	56	60	52	48	47
325	4	66	61	62	58	58	57
325	8	72	71	69	62	65	60
230	4	60	52	57	49	43	41
230	8	64	54	60	51	44	47
230	16	69	55	62	52	47	52
230	24	70	56	63	56	53	55
230	72	76	72	69	68	65	62
140	120	63	53	58	50	52	40
140	240	64	54	62	51	56	42
140	360	67	56	65	52	56	43
140	720	70	57	67	53	57	43

Table 30. Iowa Highway Commission stabilometer values for heat treated loose asphaltic concrete mixes

Treat- ment temp. °F	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0	24	26	22	31	40	35
325	2	22	20	16	24	30	24
325	4	15	16	18	19	21	20
325	8	9	10	12	17	14	15
230	4	20	28	28	25	38	39
230	8	15	17	17	26	34	31
230	16	14	22	19	28	30	24
230	24	13	19	17	23	27	20
230	72	10	10	13	13	15	26
140	120	14	22	20	27	25	30
140	240	14	19	16	27	21	32
140	360	15	20	17	34	20	34
140	720	12	22	14	25	22	23

Asphaltic concrete mixes with asphalt D after 8 hours of heat treatment at 325°F attain the same stability as asphaltic concrete mixes with asphalt A, B, and C after two to four hours of heating at 325°F. Heat treatment of all the asphaltic concrete mixes in this study at 230°F for 72 hours resulted in similar stability at 8 hours at 325°F. The effect of the heat treatment of asphaltic concrete mixes at 140°F was very small on mixes with asphalt D, whereas mixes with asphalt A, B and C showed considerable increase in stability after 360 and 720 hours. A part of this increase in stability of the mixes heat treated at 140°F may be due to the heating of the mixes to compaction temperatures of 250°F for preparing standard size specimens. Asphaltic concrete mixes with similar asphalts but with aggregate Y showed much lower stabilities than the mixes in which aggregate X was used. Comparative stability values for all the asphaltic concrete mixes "with different aggregates and same asphalt" and "same aggregate but with three penetration grades (30-40, 85-100, 150-200) of asphalts" subjected to various treatments are shown in Figures 40, 41, 42 and 43, 44 respectively. Increases in stability apparently result from the hardening of the asphalts in mixes.

Heat treatment of these mixes at 325°F even at short periods of time showed tremendous increases in stability. During heating at higher temperatures the increase in aggregate

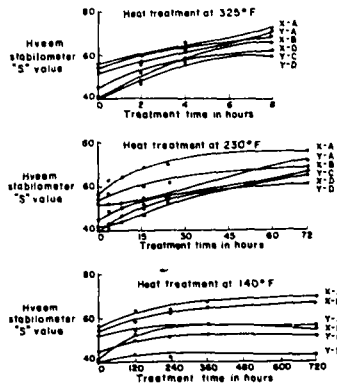


Figure 39. Hveem stability of asphaltic concrete mixes after heat treatment at 140, 230, and 325°F.

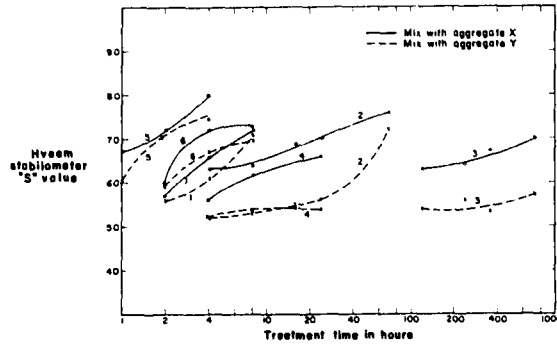


Figure 40. Hveem stability of asphaltic concrete mixes with asphalt A after various forms of treatments.

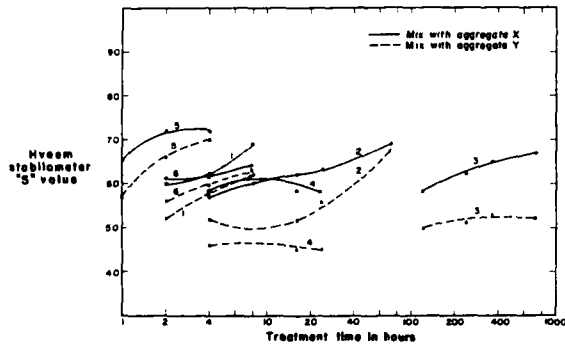


Figure 41. Hveem stability of asphaltic concrete mixes with asphalt B and C after various forms of treatments.

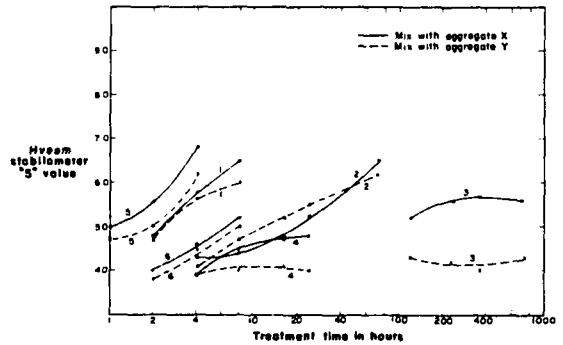


Figure 42. Hveem stability of asphaltic concrete mixes with asphalt D after various forms of treatments.

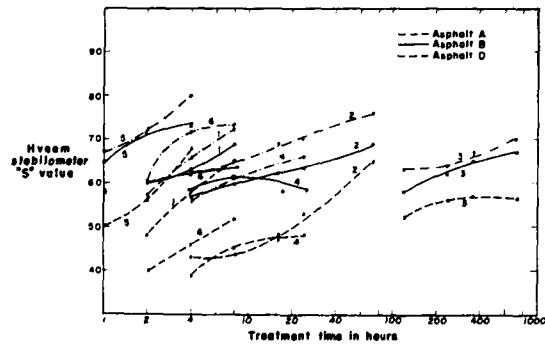


Figure 43. Hveem stability of asphaltic concrete mixes containing aggregate X after various forms of treatments.

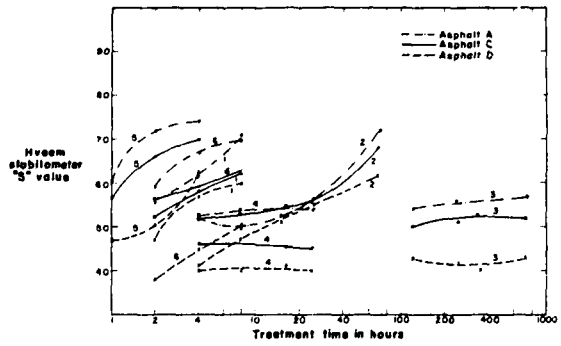


Figure 44. Hveem stability of asphaltic concrete mixes containing aggregate Y after various forms of treatments.

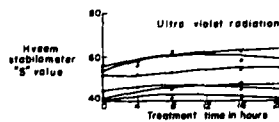


Figure 45. Hveem stability of asphaltic concrete mixes after ultra violet radiation treatment.

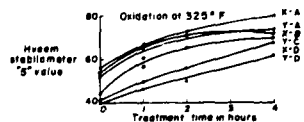
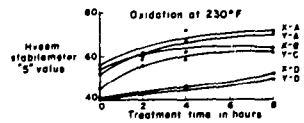


Figure 46. Hveem stability of asphaltic concrete mixes after oxidation treatment at 230 and 325°F.

selective absorption, volatilization of some asphaltic constituents and other possible chemical reactions resulted in the increase in viscosity, decrease in film thickness and better bond between aggregate particles. All these changes in asphaltic concrete mixes resulted in higher stability values. These values were very high for mixes treated at 325°F even for short periods of time. The above mentioned reactions or changes were not very severe at lower temperatures such as 230 and 140°F for short periods of time. However long periods of treatment seems to bring changes similar to higher temperature treatments for shorter time. Stability values for heat treated mixes at 230 and 325°F for 4 hours and at 230 and 325°F for 8 hours are shown in Figures 56 and 57 respectively.

Ultra-violet radiation treatment Loose asphaltic concrete mixes subjected to ultra-violet radiation up to 24 hours did not show much changes in stability. Hveem stability "S" values and the one used by Iowa Highway Commission are given in Tables 31 and 32 respectively. Hveem stability values "S" are shown graphically in Figure 45. Very slight increases in stability is indicated by mixtures in which aggregate X was used whereas mixes with aggregate Y showed slight decreases in stability values. The effect of the ultra-violet radiation on the ductility of asphalts was studied by Vallergera, Monismith and Granthem (68). Some asphalts showed tremendous increases while others considerable losses for similar treatment

Table 31. Hveem stabilometer "S" values for ultra-violet radiation treated loose asphaltic concrete mixes

Type treatment	Treatment time hrs.	Aggregate X, asphalt A	Aggregate Y, asphalt A	Aggregate X, asphalt B	Aggregate Y, asphalt C	Aggregate X, asphalt D	Aggregate Y, asphalt D
None	0	56	52	54	45	41	40
UVR ^a	4	56	52	58	46	39	39
UVR	8	62	53	62	46	45	40
UVR	16	62	54	58	45	48	41
UVR	24	66	54	58	45	48	40

^aUltra-violet radiation.

Table 32. Iowa Highway Commission stabilometer values for ultra-violet radiation treated loose asphaltic concrete mixes

Type treatment	Treatment time hrs.	Aggregate X, asphalt A	Aggregate Y, asphalt A	Aggregate X, asphalt B	Aggregate Y, asphalt C	Aggregate X, asphalt D	Aggregate Y, asphalt D
None	0	24	26	22	31	40	35
UVR ^a	4	21	23	21	31	36	36
UVR	8	17	24	18	40	25	36
UVR	16	14	23	20	32	27	35
UVR	24	28	26	17	29	23	40

^aUltra-violet radiation.

condition; no definite reason was forwarded to account for it. The ultra-violet radiation effect on the stability of the asphaltic concrete mixtures used in this study was very small, at the same time no serious detrimental effect was observed. Some abnormalities do exist in the data, which could be either due to the ultra-violet radiation or experimental error or heating of the treated mixes to compaction temperatures. It is concluded therefore that such radiation does not seem to play any major role in bringing changes in the stability values of the treated asphaltic concrete mixes.

Oxidation treatment Oxidation studies show a considerable increase in the stability values of the asphaltic concrete mixtures. Very short periods of oxidation resulted in very high stability values. The stability values "s" and the one used by Iowa Highway Commission for all mixes oxidized at 230 and 325°F are given in Tables 33 and 34 and the Hveem stability value "s" are shown graphically in Figure 46. The high increase in stability value does indicate that high temperatures accelerate the oxidation or de-hydrogenation reactions. Stability values for mixes oxidized at 230°F for 8 hours are the same as the one obtained for same mixes after 72 hours of heat treatment alone at the same temperature. Stability values of mixes after heat treatment at 325°F, for 8 hours are pretty much the same as after 2 to 4 hours of oxida-

Table 33. Hveem stabilometer "S" values for oxidized loose asphaltic concrete mixes

Oxida- tion temp. OF	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0	56	52	54	45	41	40
325	1	67	60	65	57	50	47
325	2	72	72	72	66	56	50
325	4	80	74	72	70	68	62
230	2	60	59	61	56	41	41
230	4	70	67	62	59	46	45
230	8	72	70	64	62	52	50

Table 34. Iowa Highway Commission stabilometer values for oxidized loose asphaltic concrete mixes

Oxida- tion temp. OF	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0	24	26	22	31	40	35
325	1	21	22	11	24	28	23
325	2	8	9	9	10	28	14
325	4	6	9	9	14	12	22
230	2	21	24	18	22	44	38
230	4	8	11	16	13	31	29
230	8	8	12	18	23	37	24

tion at 325°F. Higher increases in stability values of these oxidized asphaltic concrete mixes are due to oxidation of the absorbed oily constituents of asphalt in the pores of aggregate and also due to severe oxidation of the asphalt film surrounding the mineral matter, which in turn lowers the viscosity of asphalt and increases bonding between mineral matter. High temperature accelerates these oxidation reactions and therefore the stabilities of mixes oxidized at higher temperature will be greater than the mixes oxidized at low temperatures.

Hveem cohesion

Heat treatment Hveem cohesion values were obtained on the same specimens as used for the stability test. The results so obtained are given in Table 35 and shown graphically in Figure 47. The minimum cohesion values specified both for medium and heavily traveled bituminous concrete pavements is 50. All the six asphaltic concrete mixes used in this study had cohesion values greater than 50. The increase in cohesion values after heat treatment of the asphaltic concrete mixes at 325°F for 8 hours is 90 to 140 per cent in mixtures with aggregate Y and 40 to 50 per cent in mixes with aggregate X. The high cohesion values for heat treated mixes does not necessarily mean better durability. However, it does indicate the hardening of the asphalts in these mixes. As mentioned previously the asphalts recovered from mixes heat treated at

Table 35. Hveem cohesiometer "C" values for heat treated loose asphaltic concrete mixes

Treat- ment temp. °F	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0	354	261	319	230	257	166
325	2	460	330	393	340	382	343
325	4	503	433	440	380	434	352
325	8	540	516	450	440	384	402
325							
230	4	420	272	375	208	264	187
230	8	480	350	386	242	280	210
230	16	534	353	400	277	298	245
230	24	550	366	422	291	308	275
230	72	590	433	414	310	347	282
140	120	362	286	341	240	262	168
140	240	410	348	388	263	259	181
140	360	411	372	396	285	268	192
140	720	404	392	397	291	276	214

325°F were very hard and some even showed the sign of slight brittleness. However no sign of brittleness was observed during the testing of cohesion for the compacted specimens. Heat treatment of asphaltic concrete mixes at 230°F for 72 hours and at 140°F for 720 hours did show increases in cohesion values 30 to 55 per cent and 7 to 25 per cent for mixes with aggregate X and 35 to 70 per cent and 25 to 42 per cent for mixes with aggregate Y respectively. Cohesion values for mixes "with different aggregates and with the same asphalt" and "same aggregate but with 30 to 40, 85 to 100 and 150 to 200 penetration grade asphalt" subjected to various treatments are shown in Figures 48, 49, 50 and 51, 52 respectively. Increases in stability values are due to the hardening of asphalts and better bond between mineral matter as discussed under stability of heat treated mixes. Cohesion values for heat treated mixes at 230 and 325°F for 4 and 8 hours are shown in Figures 56 and 57 respectively.

Ultra-violet radiation treatment Cohesion data for asphaltic concrete mixes subjected to ultra-violet radiation treatment is given in Table 36 and shown graphically in Figure 53. Small increases in the cohesion values of asphaltic concrete mixes with asphalts A, B and C were observed after 8 to 16 hours of ultra-violet radiation treatment. However 24 hours of such treatment for the same mixes showed slight de-

Table 36. Hveem cohesiometer "C" values for ultra-violet radiation treated loose asphaltic concrete mixes

Type treat- ment	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
None	0	354	261	319	230	257	166
UVR ^a	4	381	312	414	231	234	150
UVR	8	394	343	361	213	178	162
UVR	16	340	278	369	231	182	180
UVR	24	337	317	344	219	163	183

^aUltra-violet radiation.

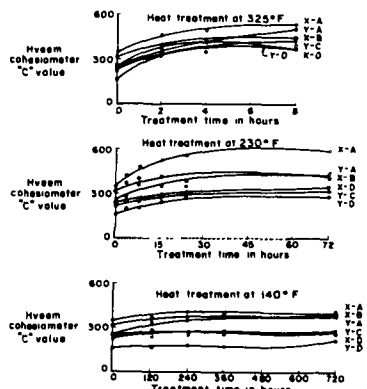


Figure 47. Hveem cohesion of asphaltic concrete mixes after heat treatment at 140, 230, and 325°F.

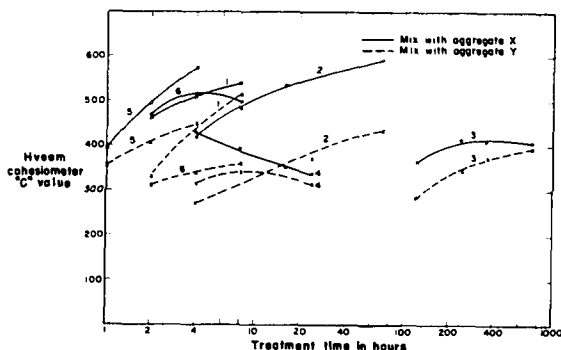


Figure 48. Hveem cohesion of asphaltic concrete mixes with asphalt A after various forms of treatments.

- 1 - Heat treatment at 325°F.
- 2 - Heat treatment at 230°F.
- 3 - Heat treatment at 140°F.
- 4 - Ultra violet radiation treatment.
- 5 - Oxidation at 325°F.
- 6 - Oxidation at 230°F.

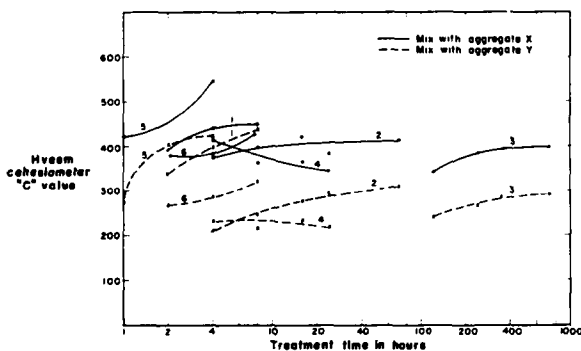


Figure 49. Hveem cohesion of asphaltic concrete mixes with asphalt A after various forms of treatments.

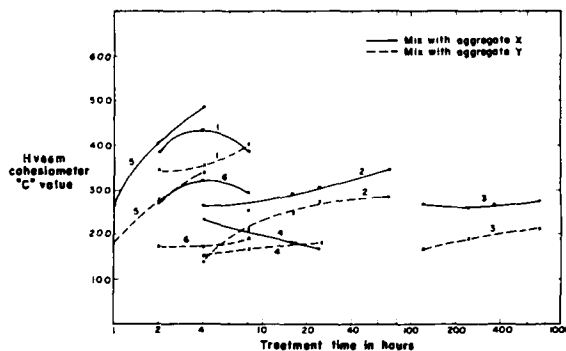


Figure 50. Hveem cohesion of asphaltic concrete mixes with asphalt D after various forms of treatments.

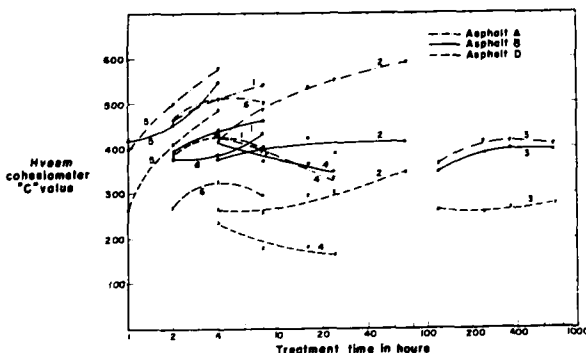


Figure 51. Hveem cohesion of asphaltic concrete mixes containing aggregate X after various forms of treatments.

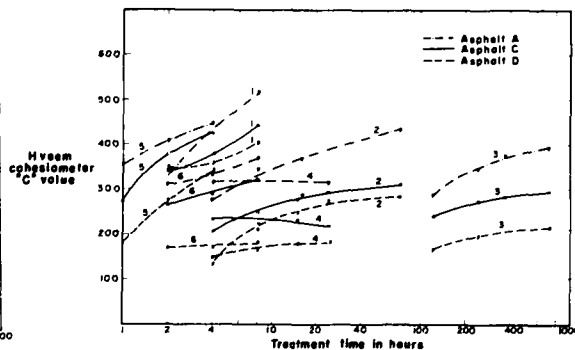


Figure 52. Hveem cohesion of asphaltic concrete mixes containing aggregate Y after various forms of treatments.

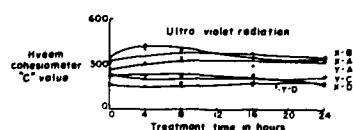


Figure 53. Hveem cohesion of asphaltic concrete mixes after ultra violet radiation treatment.

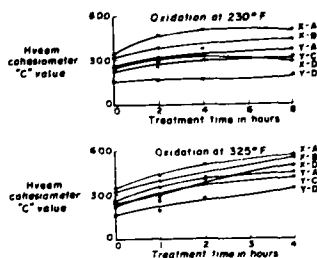


Figure 54. Hveem cohesion of asphaltic concrete mixes after oxidation treatment at 230 and 325°F.

creases in cohesion value. Asphaltic concrete mixes with asphalt D and aggregate X showed steady decrease in cohesion values but the reverse was true for mixes with aggregate Y and asphalt D. The reason of this slight increase and decrease of the cohesion values is not quite clear. The overall data show at least that such a treatment does not have any severe detrimental effect on cohesion values of the treated asphaltic concrete mixes.

Oxidation treatment Cohesion values for asphaltic concrete mixes subjected to oxidation treatment at 325 and 230°F are given in Table 37 and shown graphically in Figure 54. Asphaltic concrete mixes with asphalt D and aggregate Y showed the highest increase of 105 per cent in cohesion values after 4 hours of oxidation at 325°F. Figures 48, 49 and 50 show that all mixes with aggregate Y have higher percent increases in cohesion values than mixes with aggregate X, after similar oxidation treatments. Oxidation at 230°F does not show too severe effect on the cohesion values of the mixes. However 8 hours of oxidation of all mixes at 230°F did show 10 to 40 per cent increase in cohesion values. Cohesion values for all six asphaltic concrete mixes oxidized at 325 and 230°F were higher than the cohesion values for same mixes after heat treatment at the same temperatures and for the same time. Similar changes or reactions as discussed under the "Stability of

Table 37. Hveem cohesiometer "C" values for oxidized loose asphaltic concrete mixes

Oxida- tion temp. OF	Treat- ment time hrs.	Aggre- gate X, asphalt A	Aggre- gate Y, asphalt A	Aggre- gate X, asphalt B	Aggre- gate Y, asphalt C	Aggre- gate X, asphalt D	Aggre- gate Y, asphalt D
--	0	354	261	319	230	257	166
325	1	397	355	420	274	269	180
325	2	498	407	409	380	408	276
325	4	574	449	546	425	485	340
230	2	469	310	380	264	269	170
230	4	516	337	399	288	328	169
230	8	499	367	434	321	294	183

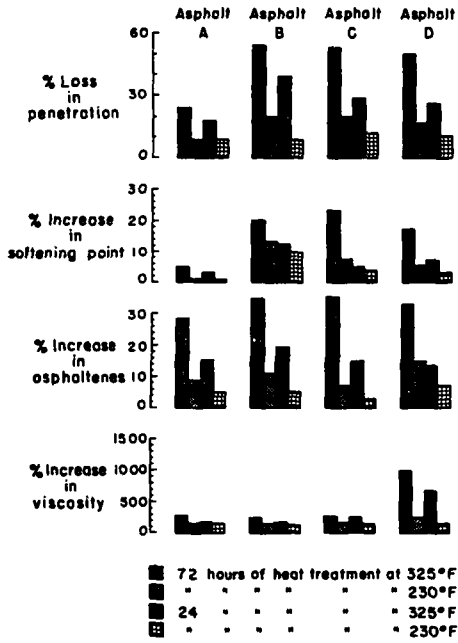


Figure 55. Percent change in the properties of recovered asphalts due to heat treatment.

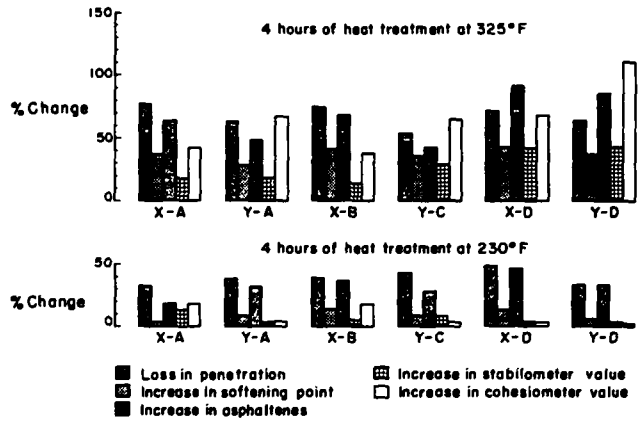


Figure 56. Percent change in the properties of recovered asphalts and asphaltic concrete mixes after heat treatment.

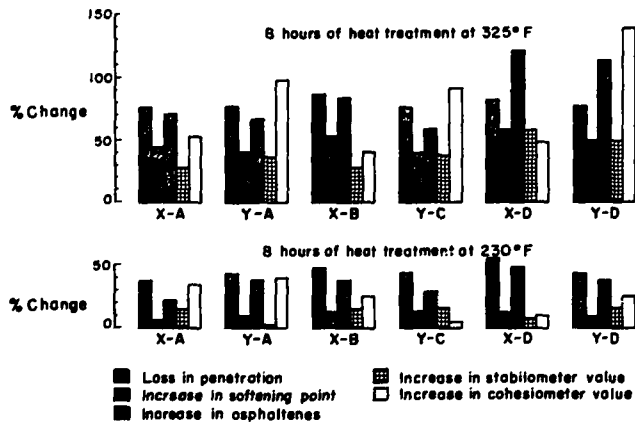


Figure 57. Percent change in the properties of the recovered asphalts and asphaltic concrete mixes after heat treatment.

heat treated and oxidized mixes" result in high cohesion values for oxidized mixes.

Effect of Aggregate Type on Physical Properties
of Recovered Asphalts and Asphaltic Concrete
Mixes

Two aggregates, namely X and Y, were used in this study. Aggregate Y is much more absorptive in character than aggregate X. Type of the aggregate has shown considerable effect on the physical properties of the asphaltic mixtures and the asphalts recovered from them. Aggregate Y is believed to absorb some of the oily constituents of asphalt from the films of binder surrounding the mineral matter. This type of absorption is referred as selective absorption. Such an absorption therefore changes the chemical composition of the overall asphalt film covering the aggregate. Absorption of the oily constituents of asphalt by the mineral matter therefore reduces the available reactants for oxidation or dehydrogenation reactions, in the films of asphalt surrounding the aggregate. Oily constituents are known to change to asphaltenes and asphaltic resins due to oxidation or dehydrogenation reactions and asphaltic resins further changes to asphaltenes by further action. During the recovery of the asphalt from these asphaltic concrete mixes these absorbed constituents of asphalt are desorbed by the solvent. It is therefore the absorptive characteristic of the aggregate Y which results in the comparatively

less loss in penetration and less increase in softening point, per cent asphaltenes and viscosity of the asphalts recovered from treated asphaltic concrete mixes with aggregate Y than the asphalts recovered from similarly treated asphaltic concrete mixes with aggregate X. Such comparisons are particularly more significant in the cases where the asphaltic concrete mixes are heat treated at temperatures such as 325°F or higher. High temperatures accelerate oxidation or dehydrogenation reactions and materially increase absorption of asphalt constituents by porous aggregates. However the asphalts recovered from asphaltic concrete mixes with aggregate Y oxidized at 325°F show higher percent losses in penetration, higher percent increases in the softening point, percent asphaltenes and viscosity than the asphalts recovered from the mixes with aggregate X, after similar treatments. Higher porosity of the aggregate Y results in severe oxidation of the absorbed oily constituents of asphalt during oxidation of the mixes at 325°F due to the presence of the oxygen in pores of the mineral matter; Thus resulting in higher percent changes in the physical and chemical properties of recovered asphalts.

Stability and cohesion values of treated asphaltic concrete mixes were similarly affected by the absorptive character of the aggregates. Absorption of the asphalt in the pores of the aggregates reduces the film thickness of the asphalt,

increases the viscosity and thereby increases the bonding between the aggregate particles. However such effects as reduction in the film thickness due to the absorption of oily constituents of asphalt from asphalt films results, in the severe oxidation or dehydrogenation of the remaining asphalt constituents, especially when asphaltic concrete mixes are subjected to heat and oxidation at higher temperatures. All this results in the hardening of the asphalt in the asphaltic concrete mixtures. It is due to this fact that percent increases in stability and cohesion values in most of the treated asphaltic concrete mixes (especially after treatment at high temperatures) with aggregate Y were comparatively higher than for the asphaltic concrete mixtures with aggregate X. Higher temperature treatments, heat or oxidation of asphaltic concrete mixes result in higher percent increase of stability and cohesion values.

SUMMARY AND CONCLUSIONS

In this investigation asphalts and loose asphaltic concrete mixes were subjected to heat, ultra-violet radiation and oxidation treatments for various temperatures and durations. The effect of heat on original asphalts and asphalts recovered from treated asphaltic concrete mixes by modified Abson method was studied. Physical and chemical changes due to treatments were judged on the basis of the penetration, softening point, per cent asphaltenes, viscosity, characterizing factor and the penetration index results. Stability and cohesion of the treated asphaltic concrete mixes were obtained by Hveem stabilometer and cohesiometer. Following is a summary of the important results obtained with limited testing, in this experimental study and the conclusions drawn therefrom:

1. Asphalts when heated do show considerable changes in their physical and chemical properties. These changes become much greater when heated at high temperatures and for long periods of time. Asphalts of 85-100 and 150-200 penetration grades showed similar changes in penetration, softening points and per cent asphaltenes. Asphalt of low penetration grade (30-40) showed much less changes in the properties than the high penetration asphalts. Viscosity increases were the highest in asphalts with high penetration and lowest in asphalt with low penetration.

2. Characterizing factor for all these heat treated asphalts did not show much deviation from the original values for the same asphalts before heat treatment, consequently it appears that no change occurs in the paraffinic constituents of asphalts by heating up to 325°F for periods as long as 72 hours.

3. Penetration index values for all these asphalts heat treated at 230 and 325°F for various time intervals vary between -1 and +1, thereby classifying these asphalts in the category of "normal asphalts" which is the same, as for the original asphalts prior to heat treatment.

4. Asphalts recovered from asphaltic concrete mixes subjected to heat treatment showed great changes in the physical and chemical properties. These changes become more severe in asphalts recovered from mixes treated at higher temperatures and for longer periods of time.

5. All the asphalts recovered from heat treated mixes at 325°F for 8 hours showed 75 to 85 per cent losses in penetration. Heat treatment at lower temperatures but for long periods of time seems to produce similar results.

6. Per cent changes in penetration, softening point, per cent asphaltenes and viscosity of the recovered asphalts from heat treated loose asphaltic concrete mixes increases with increase in treatment time and temperatures.

7. Heat treatment of asphaltic concrete mixes at 325°F

for 2 to 3 hours, at 230°F for 24 to 72 hours and at 140°F for about 700 hours seems to produce similar effect as far as the per cent loss in penetration of the recovered asphalts is concerned.

8. Asphalts recovered from asphaltic concrete mixes do not seem to represent the original asphalts according to Kinnaird's characterizing factor values. Type of aggregate and asphalt used in these mixes, treatment conditions, recovery method and small quantities of mineral matter left in recovered asphalts seem to affect the characterizing factor values.

9. Recovered asphalts from all the treated asphaltic concrete mixes except the ones subjected to heat treatment and oxidation at 325°F for 4 or more hours have penetration index values between -1 and +1, thereby classifying these asphalts in the category of the "normal asphalts" which is the same as for the original asphalts prior to mixing with the aggregate. Whereas the asphalts recovered from mixes heat treated or oxidized at 325°F for 4 or more hours have penetration index values greater than 1, which classify them in the "R type" class of Pfeiffer's classification, which are characterized by slight brittleness.

10. Ultra-violet radiation treatment does not show much effect on the physical and chemical properties of recovered asphalts from the asphaltic concrete mixes subjected to such

radiation for short periods of time. However long treatment periods seem to affect the properties of recovered asphalts considerably.

11. Oxidation of asphaltic concrete mixes at 325°F show much higher changes in physical and chemical properties of recovered asphalts than the asphalts recovered from heat treated mixes at 325°F for the same period of time. Although oxidation and heat treatment of asphaltic concrete mixes at 230°F showed similar changes in physical and chemical properties of the recovered asphalts A, B and C, but recovered asphalts D from oxidized mixes underwent much higher changes than the corresponding asphalts recovered from heat treated mixes.

12. The changes in properties of the recovered asphalts and asphaltic concrete mixes brought about by heating and oxidation treatments of these mixes are materially affected by the physical characteristics of the aggregate used in those mixes.

13. All the asphalts recovered from heat treated asphaltic concrete mixes with high absorption aggregate Y showed less loss in penetration and smaller increase in softening point, per cent asphaltenes and viscosity than the asphalts recovered from the asphaltic concrete mixes with aggregate X. Such differences are due to the higher absorption of the aggregate Y

than aggregate X. However the reverse was true for most of the asphalts recovered from oxidized asphaltic concrete mixes.

14. Asphaltic concrete mixes subjected to various treatments in loose state showed increases in stability and cohesion values. Such increases were very high in mixes treated at high temperatures and for long periods of time.

15. Asphaltic concrete mixes made with high penetration asphalts showed similar stability and cohesion values as the low penetration asphalt after 2 to 4 hours of heat and oxidation treatments at 325°F. However much longer periods of treatment at 230°F are needed to attain the same cohesion and stability values as for the asphaltic concrete mixes with low penetration asphalt.

16. Higher per cent increases were obtained in stability and cohesion values in almost all the asphaltic concrete mixes with aggregate Y than the asphaltic concrete mixes with aggregate X.

17. Oxidation of all the asphaltic concrete mixes at 230 and 325°F showed greater stability and cohesion values than the same mixes subjected to heat treatment alone at the same temperatures and for same periods of time.

18. Although the penetration values of some recovered asphalts were extremely low and the appearance of the corresponding mixes were very dull and dry no sign of brittleness was observed during cohesion testing of compacted specimens

from such mixes. These mixes on the other hand gave very high cohesion values.

19. Although treatment of asphaltic concrete mixes at temperatures such as 230 and 140^oF for shorter periods of time brought changes in the physical characteristics of the recovered asphalts, these do not appear to affect the physical characteristics of the mixes materially.

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