United States Patent Office 3,457,093 Patented July 22, 1969

3,457,093

PRODUCTION OF HIGH SOFTENING POINT ASPHALT

Dean P. Montgomery, Bartlesville, Okla., assignor to Phillips Petroleum Company, a corporation of Delaware

No Drawing. Filed June 21, 1965, Ser. No. 465,766
Int. Cl. C08h *13/00*; C10c *3/02*
U.S. Cl. 106—273 8 Claims

ABSTRACT OF THE DISCLOSURE

An alkali metal, such as sodium, is mixed with an asphalt at an elevated temperature and in a non-oxidizing atmosphere to produce an asphalt having a high softening point and a relatively high penetration.

This invention relates to a process for the production of an improved asphaltic material. In another aspect, this invention relates to a process for the production of an asphalt having a high softening point and an improved penetration.

Resistance to changes in properties such as penetration, ductility and softening point with changes in temperature is a desirable characteristic of asphaltic materials which are employed as roofing materials, canal linings, pipeline coatings, electrical insulating material, etc. It is desirable that such asphaltic materials not become excessively hard, brittle or soft over moderate ranges of ambient temperatures. It is conventional to raise the softening point of an asphaltic material by air blowing the asphalt. Such an asphalt has a higher softening point for a given penetration than an asphalt produced from the same feedstock by vacuum reduction or solvent extraction. However, the process of air blowing the asphalt substantially reduces the penetration of the asphalt, producing a product asphalt with a high softening point and having an undesirably low penetration.

Accordingly, an object of my invention is to provide an improved process for the production of an asphalt having a high softening point.

Another object of my invention is to provide a process for the production of an asphalt having a high softening point and a relatively high penetration.

Other objects, advantages and features of my invention will be readily apparent to those skilled in the art from the following description and the appended claims.

Briefly, the invention comprises admixing an alkali metal with an asphalt at an elevated temperature and in a non-oxidizing atmosphere to produce an asphalt having a high softening point and a relatively high penetration.

Asphalt, as the term is herein employed, refers to a residual fraction obtained upon the distillation of petroleum crude oils. The invention is applicable to asphalts having a penetration in the range of about 25 to 300+ as determined by Test Method ASTM D5–61, and particularly for asphalts having a penetration in the range of about 150 to 250.

The asphalt is admixed at an elevated temperature in a non-oxidizing atmosphere with a molten alkali metal. Preferred alkali metals are lithium, sodium, potassium and cesium. The amount of alkali metal added to the asphaltic material is normally in the range from 0.1 to 10 weight percent of the asphaltic material, although it is within the scope of this invention to employ higher concentrations of the alkali metal.

Conventional mechanical means of agitating the mixture can be employed. Agitation can also be provided by a stream of non-oxidizing, non-reactive gas such as natural

gas, refinery residue gas, or the like. Furnace flue gas substantially free of oxidizing components or other non-reactive, non-oxidizing gases can also be employed.

The asphalt and molten alkali metal are mixed at an elevated temperature ranging from just above the melting point of the alkali metal employed to about 600° F. or higher. Preferably, the asphalt and alkali metal are mixed at an elevated temperature in the range from just above the melting point of the alkali metal to 300° F. The mixing or reaction time employed will range from one minute to 10 hours or more.

As previously noted, mixing of the alkali metal and asphalt at an elevated temperature is conducted in a non-oxidizing atmosphere. The mixing step can be conducted, for example, in a nitrogen atmosphere or, as previously noted, by employing a stream of non-oxidizing, non-reactive gas such as natural gas, refinery residue gas, or the like, as a means of agitating the mixture. By employing a stream of non-oxidizing, non-reactive gas as a means of agitating a mixture, a non-oxidizing atmosphere is produced.

The following examples are presented to illustrate the objects and advantages of my invention. It is not intended that the invention should be limited to the specific embodiments presented therein.

Example I

To illustrate a conventional process of producing a high softening point asphalt by air blowing an asphaltic material, a 231 penertation asphalt (as determined by ASTM D5–61 Test Method) having a ring and ball softening point (ASTM D36–26) of 103 was air blown at 490° F. using 200 s.c.f. air/ton/minute. Data obtained from the product air blown asphalt using the ring and ball softening point and penetration test methods previously noted were as follows:

Softening point, ° F.:	Penetration, 100 g./5 sec./77° F.
140	48
160	34
180	26
200	21
220	18

Example II

To illustrate the process of my invention, a sample of the 231 penetration asphalt of Example I was agitated with sodium using a Dispersator agitator and employing a nitrogen atmosphere. A sodium concentration of 2.0 weight percent of the asphalt was employed and mixing temperatures in the range of 230–400° F. were utilized. Times of mixing ranged from 15 minutes to 5 hours. Plots of the test results (employing the test methods of Example I) yielded smooth curves from which the following points were taken:

Softening point, ° F.:	Penetration, 100 g./5 sec./77° F.
140	59
160	48
180	41
200	36
220	33

A comparison of the results obtained in Examples I and II clearly demonstrate the effectiveness of the invention to produce a high softening point asphalt having a substantially improved penetration. Comparing, for example, the asphaltic materials having a softening point of 220° F., the asphalt produced by the invention has a penetration 83.3 percent higher than the penetration of the corresponding air blown asphalt.

Example III

In order to demonstrate the criticality of employing a non-oxidizing atmosphere in the process of my invention, the 231 penetration asphalt of Examples I and II was air blown at a temperature of 490° F., using an air flow rate of 200 s.c.f. air/ton/minute, in the presence of 2 weight percent sodium. Tests, employing the penetration and softening point test methods of Examples I and II, of the air blown product asphalt produced the following results:

Softening point, ° F.:	Penetration, 100 g./5 sec./77° F.
140	54
160	41
180	32
200	25
220	20

A comparison of the data obtained in Examples II and III demonstrate the criticality of conducting the sodium treatment of the asphaltic material in a non-oxidizing atmosphere. By operation in an oxidizing atmosphere to produce an asphalt having a ring and ball softening point of 220° F., the penetration was reduced by 39.5 percent.

As will be evident to those skilled in the art, various modifications of this invention can be made, or followed, in the light of the foregoing disclosure, without departing from the spirit or scope thereof.

I claim:

1. A process which comprises admixing an asphalt with an alkali metal at a temperature above the melting point of said alkali metal and below 600° F. in a non-oxidizing atmosphere, agitating said mixture for a time, within the range of one minute to ten hours while maintaining said non-oxidizing atmosphere and recovering said asphalt.

2. The process of claim 1 wherein said elevated temperature is below 300° F. and said alkali metal is sodium.

3. The process of claim 1 wherein the concentration of said alkali metal is in the range of 0.1 to 10 percent by weight of said asphalt and wherein said elevated temperature is below 400° F., said recovered asphalt having a high softening point and a relatively high penetration.

4. A process which comprises admixing an asphalt having a penetration in the range of 150–250 with 2 percent by weight of sodium at a temperature ranging from the melting point of said sodium to 400° F. in a nitrogen atmosphere.

5. A process which comprises contacting an asphalt with an alkali metal at a temperature above the melting point of said alkali metal and below 400° F., and passing a non-oxidizing gas through the formed mixture.

6. A composition produced by admixing an asphalt with an alkali metal at a temperature above the melting point of said alkali metal and below 400° F. in a non-oxidizing atmosphere.

7. The composition of claim 6 wherein the concentration of said alkali metal is in the range of 0.1 to 10 percent by weight of said asphalt.

8. The composition of claim 6 wherein said alkali metal is sodium.

References Cited

UNITED STATES PATENTS

1,865,235	6/1932	Cross	252—476 XR
2,175,189	10/1939	Gambrill	208—44

FOREIGN PATENTS

897,843	5/1962	Great Britain.

JULIUS FROME, Primary Examiner

J. B. EVANS, Assistant Examiner

U.S. Cl. X.R.

208—9, 10, 44; 260—695