Patented June 6, 1944 2,350,649

UNITED STATES PATENT OFFICE

2,350,649

METHOD FOR ASPHALT SATURATION

Wallace E. Spelshouse, Jamaica Estates, and Arthur H. Boenau, Long Island City, N. Y., assignors to Socony-Vacuum Oil Company, Incorporated, New York, N. Y., a corporation of New York

No Drawing. Application July 2, 1941, Serial No. 400,814

5 Claims. (Cl. 117—168)

This invention relates to a non-foaming asphalt saturant and to the manufacture of that product.

In the manufacture of linoleum, waterproofing materials for the building trade, waterproofing packing materials, and similar fibrous materials, it has been customary to saturate burlap, and other fabrics, as well as paper, felt, and other fibrous materials, with melted bituminous saturants. In modern plants this process is accomplished by pulling a continuous sheet of fabric or paper through a vat containing the hot asphalt. There is always some moisture present in the fabric and sheet paper which suddenly evaporates when the fabric or sheet paper is immersed in the asphalt bath which is at temperatures above the boiling point of water. Usually the temperatures of such asphalt baths are between 250 and 450° F.

The foaming of the asphalt associated with the evaporation of water from the sheet is a highly undesirable feature both from the standpoint of efficiency of the plant and quality of the product.

It has been observed that asphalts derived from certain crudes produce a more tenacious foam while asphalts from selected crudes such as Mexican, produce a foam which subsides very quickly and does not cause difficulties in the process of saturation.

The method of control of foaming characteristics of saturant asphalts presently used in the art is wholly one of control by selecting the source of crude from which the asphalt is produced, no method as yet having been developed for producing non-foaming saturants from sources other than those wherein material of the grade desired is fortuitously endowed with the property.

This invention has for its object the provision of a non-foaming asphalt saturant and a method of producing that saturant, which is not restricted to the source materials formerly used. A principal object of this invention is the production of high quality non-foaming asphalt saturants from materials not heretofore believed to be capable of producing such saturants.

Conventional methods of preparing satisfactory asphalt saturants comprise first selecting a suitable crude oil, say Mexican asphaltic crude, and reducing it to the desired consistency by either conventional air blowing, or by steam and/or vacuum reduction, or by combination of the two methods.

We have found that by preparing an asphalt base from materials of a certain kind, and reducing that asphalt base to a controlled hardness greater than that specified for the asphalt saturant, and then cutting that base back to specification with a liquid or semi-liquid petroleum oil, which may be either a residual product or a distillate, it is possible to control the foaming characteristics of the asphalt saturant.

We can use as a raw material for the preparation of such a hard base any hydrocarbon residual product with a viscosity-gravity constant of .890 minimium. The conception of V. G. C. for expressing the naphthenicity, aromaticity or paraffinicity of the hydrocarbon fractions has been introduced by Hill & Coates in 1928 (see Industrial & Engineering Chemistry 1928, page 641). Solvent tars such as obtained from single and double solvent refining operations; pressure still tars (cracking still tars), low temperature carbonization tars and straight run petroleum residua from certain asphaltic crudes are representative of the above materials.

We may prepare the hard asphalt base either by blowing an asphaltic liquid or semi-liquid material with air or other oxidizing gases, or by reducing the asphaltic material to a certain consistency by means of a fire, steam or vacuum distillation.

The liquid or semi-liquid oil used in cutting back to the form the finished saturant may be any oil of the following characteristics:

Viscosity index, —50 minimum
Viscosity from 40'' Saybolt Universal @ 100° F. to 4000'' Saybolt Furol @ 210° F.

In making the asphaltic base for the saturant, the base should be reduced to a softening point at least five degrees Fahrenheit, as expressed by the Ring and Ball A. S. T. M. method D36–26 above the specification desired for the final saturant.

The amount of cutback oil to be used may, of course, vary, not only with the physical characteristics of the cut-back oil itself, but also with the excess hardness of the asphalt base. In general, however, the blending and related figures may be described as falling within the following limits.

Degree of overblowing of asphaltic base 5 to 100 degrees F. (R. & B.)
Physical characteristics of cutback oil
Viscosity 40'' S. U. V. @ 100° to 40,000'' S. U. V @ 210° F.
V. I. —50 minimum.
Percentage of cutback oil added, 5% to 75%

In order to give a clear idea of an acceptable asphalt saturant, the following is a typical specification as used in many industries.

Example

Soft. point (R. & B.)	150–160° F.
Penetration:	
@ 77/100/5	15–25
@ 32/200/60	5 min.
@ 115/50/5	100 max.
Foam break, seconds	420 max. (7 min.)

The roofing, linoleum and similar industries where asphalt saturants are used, have adopted a practical laboratory test for the evaluation of asphalts from the standpoint of foaming characteristics. This test, while not endorsed by the American Society of Testing Materials, or any similar recognized organization, has found wide application in industrial laboratories. This test can be described as follows:

250 grams of asphalt are weighed into a 1000 cc. high type glass beaker and heated to 400° F. on an oil bath. The level of the oil bath should be ¼" below the level of the asphalt and care should be taken that the oil level never rises above the level of the asphalt. An oil bath temperature of about 435° F. should maintain the proper asphalt temperature but this will have to be determined for the particular apparatus as no thermometer should be in the asphalt during the test.

Using a 3" x 12" piece of 40-point felt place 0.25 cc. of water on each side not more than one inch from one end. Allow the water to soak in one minute, then submerge the wet end of the felt in the asphalt and hold 15 sec. Remove the felt and note the time for the first break of bubbles to give a clear surface. As the foam breaks there will finally be a single layer of bubbles on the surface, then suddenly this layer will break revealing the surface of the asphalt. This is the end point.

Inasmuch as the results of the foaming test are expressed in terms of time necessary for the foaming to subside, a 7 minute asphalt would indicate a saturant with considerably poorer foaming characteristics than a 3 minute asphalt. A 3 minute asphalt may be considered as an example of an excellent non-foaming saturant, and 4–5 minute as good; and above 9 unsatisfactory.

In order to show results of compounding asphalt saturants as taught herein in comparison with other methods, the following data is given, in which a solvent tar derived from a "Duo-Sol" or counter-current propane-cresylic acid solvent treatment of a long residuum from mixed base crude was used, together with a straight run Coastal residuum. These starting materials had the following properties:

Table I

Tests	Duo-Sol tar	Straight run coastal residuum
Gravity A. P. I.°		14.0
Spec. Grav. @ 60/60° F	1.017	0.972
Flash, C. O. C. °F	545	Over 540
S. U. V. @ 210° F		2,000"–2,500"
Viscosity—Furol @ 210° F	241"	

The following table includes the physical characteristic of four asphalt saturants.

Column one represents a conventional asphalt saturant prepared from Mexican crude and meeting the above specifications previously given.

Column 2 represents an asphalt saturant prepared by blowing a 50/50 blend of Duo-Sol tar and a straight run residuum from a Coastal crude. This material is unsatisfactory with respect to foaming characteristics.

Column 3 represents a material produced by blending 74% of Duo-Sol tar blown to 180 softening point and cut back with 26% of the same straight run residuum from a Coastal crude as in column 2. This material is equivalent to that represented by column 1.

Column 4 represents an asphalt saturant produced by steam refining (vacuum distilling) Duo-Sol extract to 182 softening point and cutting back this hard asphalt base with the same straight run residum from Coastal crude as in columns 2 and 3. The ratio of the components is 70 S. R. Duo-Sol tar to 30 Coastal crude residue. This material is also satisfactory from the standpoint of foaming characteristics.

Table II

	1	2	3	4
	Mexican S. R. and Bl.	D. S. tar/coastal residuum blown after mix	Bl. D. S. tar +coastal residuum	St. Ref. D. S. tar +coastal residuum
MP (R. and B.) °F	150	153	152	150.5
Penetration:				
77/100/5	17	20	17	15
32/200/60	7	9	8	6
115/50/5	74	63	67	75
Foam	3' 27"	20' 48"	4' 40"	6' 25"

It will be noted that by first preparing an asphalt base considerably harder than required by the specifications and then by cutting it back to the proper consistency, satisfactory products are obtained whereas the oxidation of the premixed components gives a product with unsatisfactory foaming characteristics.

As examples of blends using extreme proportions of the ingredients, the following data is offered:

Table III

	Composition	Foam
A	91% 158.5° F. soft point blown Duosol tar 9% residual Coastal oil	6 min. 23 sec.
B	37% 196° F. soft point blown Duosol tar 63% 129° F. soft point steam refined asphalt from Talco crude.	7 min.

It will be noted that these represent saturants with foaming characteristics which are acceptable, though not highly satisfactory. In other words, they illustrate approaches to the previously mentioned limits of blending. In Example A, almost the minimum of cutback that can be used is illustrated and in B, the maximum is similarly illustrated. Obviously as more cutback is used, it must itself be more nearly of the desired saturant specification as to soft point, and if used to excess, its character predominates, and the mixture becomes merely the non-satisfactory foaming saturant produced from ill-chosen starting materials by the prior art.

We claim:

1. A method for avoiding excessive foaming in the saturation of moisture-containing fibrous material with asphalt at a temperature at which evaporation of moisture contained in said material normally tends to produce excessive foaming comprising submerging a moisture-contain-

ing fibrous material to be saturated in a heated bath of an asphaltic material produced by reducing a petroleum fraction having a viscosity-gravity-constant of at least .890 to an asphalt having a hardness of at least about 5° F. (R. & B. soft point) greater than that desired for the finished saturant, and cutting the asphalt back to desired saturant specifications with a less viscous oil having a viscosity index of at least —50.

2. A method for avoiding excessive foaming in the saturation of moisture-containing fibrous material with asphalt at a temperature at which evaporation of moisture contained in said material normally tends to produce excessive foaming comprising submerging a moisture-containing fibrous material to be saturated in a heated bath of an asphaltic material produced by reducing a petroleum fraction having a viscosity-gravity-constant of at least .890 to an asphalt having a hardness of at least about 5° F. (R. & B. soft point) greater than that desired for the finished saturant, and cutting the asphalt back to desired saturant specifications by blending from 95 to 25 parts of the asphalt with from 5 to 75 parts of a less viscous oil having a viscosity index of at least —50.

3. A method for avoiding excessive foaming in the saturation of moisture-containing fibrous material with asphalt at a temperature at which evaporation of moisture contained in said material normally tends to produce excessive foaming comprising submerging a moisture-containing fibrous material to be saturated in a heated bath of an asphaltic material produced by reducing solvent tar derived from petroleum, which solvent tar has a viscosity-gravity-constant of at least .890 to an asphalt having a hardness of at least about 5° F. (R. & B. soft point) greater than that desired for the finished saturant, and cutting the asphalt back to desired saturant specifications with a less viscous oil having a viscosity index of at least —50.

4. A method for avoiding excessive foaming in the saturation of moisture-containing fibrous material with asphalt at a temperature at which evaporation of moisture contained in said material normally tends to produce excessive foaming comprising submerging a moisture-containing fibrous material to be saturated in a heated bath of an asphaltic material produced by reducing a residual petroleum fraction having a viscosity-gravity-constant of at least .890 to an asphalt having a hardness of at least about 5° F. (R. & B. soft point) greater than that desired for the finished saturant, and cutting the asphalt back to desired saturant specifications with a less viscous oil having a viscosity index of at least —50.

5. A method for avoiding excessive foaming in the saturation of moisture-containing fibrous material with asphalt at a temperature at which evaporation of moisture contained in said material normally tends to produce excessive foaming comprising submerging a moisture-containing fibrous material to be saturated in a heated bath of an asphaltic material produced by blowing a "Duo-Sol" tar, having a viscosity-gravity-constant of at least .890 to an asphalt having a hardness of at least about 5° F. (R. & B. soft point) greater than that desired for the finished asphalt saturant, and cutting back the asphalt to desired saturant specifications by adding at least five percent of a less viscous oil thereto.

WALLACE E. SPELSHOUSE.
ARTHUR H. BOENAU.