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3,374,104

ASPHALT COMPOSITIONS AND PROCESS FOR PREPARING SAME

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This invention relates to heat-stable thermal asphalts and to a process for producing them.

In recent years the demand for thermal or cracked asphalts has significantly increased. These asphalt compositions which are widely used such as in the production of insulating building boards and sheathing, as saturants for fiber sewer pipe, as binders for coal briquets etc. are generally prepared by the thermal cracking of synthetic tower bottoms from catalytic cracking units such as Thermoform Catalytic Crackers (TCC); or aromatic gas oils.

The stocks normally used in preparing these thermal asphalts are relatively limited in quantity. Moreover, due to the increased usage of thermal asphalts there is a possibility that the demand for this type of stock may exceed the supply. It would therefore be advantageous to provide for the production of heat-stable thermal asphalts from stocks other than those presently used.

It has now been found that it is possible to produce a thermal asphalt of good heat stability using fractions which are normally by-products of other processes and are therefore relatively economical sources of supply.

It is an object of the present invention to provide a thermal asphalt of excellent heat stability using economical feed materials.

A further object is to provide a simple and effective process for producing these heat-stable thermal asphalts.

Other objects of the present invention and the advantages thereof will become apparent from the following description.

Broadly stated, the thermal asphalts of the present invention are prepared from resinous pitch materials using controlled distillation procedures.

These resinous pitch materials are highly aromatic pitches or tars. In general, they possess a low-boiling "solvent" fraction but they are deficient in the high boiling oils which act as plasticizers for the high molecular weight components thereof. More particularly, the resinous pitches used herein may be characterized as possessing the following properties:

Softening point, ring and ball (R&B) ° F.; from about 75 to about 140.

Specific gravity at 77° F.; from about 1.0 to about 1.30.

Conradson carbon, percent; from about 25 to 45.

A preferred pitch is one possessing softening points (R&B) from about 100 to about 130° F., specific gravities in the range from about 1.10 to about 1.20 and Conradson carbon values of from about 30 to about 40%.

Particularly desirable resinous pitches of the aforementioned type are those obtained as a by-product from the cracking of light crude and distillate in an ethylene unit to produce ethylene, propylene and heavier hydrocarbons. The pitch obtained from the cracking furnace of the ethylene unit is a solution of hard brittle-type resinous particles in a fairly low boiling solvent fraction. A typical analysis of two suitable resinous pitches is given hereinafter.

The thermal asphalts of the invention are produced by blending the resinous pitches with a selected material which functions to plasticize and upgrade the resulting

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asphalt product. These blending materials contain substantial amounts (at least about 20 wt. percent and up) of high-boiling aromatic oils. Suitable blending materials of this type are obtained from low softening point thermal asphalts, visbroken asphalts, water gas tars and coal tars and possess softening points (R&B) from about 80 to about 120° F., preferably from about 105 to about 115° F.; Specific Gravities at 77° F. of from about 1.0 to about 1.30, preferably from about 1.15 to about 1.20; and Conradson Carbon values of from about 20 to about 40%, preferably about 25 to 35%.

Resinous Pitch Analysis		(1)	(2)
15	Carbon, percent.....	93.5	
	Hydrogen, percent.....	5.9	
	Atomic C/H Ratio.....	1.32	1.38
	Sulfur, percent.....	.34	.24
	Barber Stain.....		
	Specific Gr., 77/77° F.....	1.178	1.190
	Softening Point R&B, ° F.....	122	139
10	Penetration 100 g./5 sec.: 77° F.....		
20	115° F.....	28	7
	Infrared Absorption Index.....	1.01	147
	Infrared Transmission Index.....	1.00	
	Conradson Carbon, Percent.....	31.6	36.0
	Flash COC, ° F.....	390	390
	Ash, Percent.....		.06
25	Viscosity at— 300° F. SFS/cs.....	20/42	193/411
	350° F. SFS/cs.....		37/79
	400° F. SFS/cs.....		12/26
	Solubility in CCl ₄ , Percent.....		94.97
	Solubility in CS ₂ , Percent.....		99.02
30	Benzene Insoluble, Percent.....	1.63	

A particularly preferred blending material is a thermal asphalt characterized by the following properties:

Softening point (R&B), about 112° F.,
Penetration (77° F./100 g./5 sec.), 69,
Viscosity at 250° F.=66 centistokes,
Specific gravity at 77° F.=1.165.

The resinous pitch materials may comprise, in general, from about 20 to about 80%, preferably about 40 to about 60% by weight of these blends.

The thermal asphalts of this invention are prepared using either of two methods. According to the first method, pitch is distilled, preferably under vacuum to drive off the volatile solvent fraction, and the remaining bottoms fraction admixed with the blending materials. In the second method the pitch is admixed with the blending material and the resulting composition then distilled to remove the low-boiling fraction of the resinous pitch and produce the desired thermal asphalt product.

The distillation conditions employed will vary depending on such factors as the particular nature of the materials used, the relative proportions of pitch and blending material, etc.

When employing the first method wherein the resinous pitch alone is distilled, the distillation step is carried out to produce a bottoms product possessing a softening point (R&B) in the range from about 280 to about 300° F. Depending on the characteristics of the pitch, the bottoms may comprise, for example, from about 20 to about 80% of the original feed. In the case where the admixture of resinous pitch and blending material is distilled, the distillation is conducted to the extent required to produce a thermal asphalt in the softening point (R&B) range from 150 to 270° F.

The distillation conditions suitable for producing resinous pitch bottoms and thermal asphalt products possessing the above softening points will be apparent to a worker skilled in the art. A preferred distillation is conducted under vacuum at temperatures (corrected to

760 mm. of Hg) from about 800 to about 900° F. The thermal asphalts produced according to the present invention are advantageous in that they are produced from an inexpensive pitch material of otherwise limited utility. In addition, the instant thermal asphalts possess superior heat stability. For example, upon being exposed to elevated temperatures these thermal asphalts exhibit a minimal percent weight loss and only a relatively small increase in their softening points.

The following specific embodiments are given to further illustrate the present invention.

EXAMPLE 1

The resinous pitch bottoms obtained from the cracking furnace of ethylene unit is vacuum (2 mm. Hg) distilled to 870° F. corrected vapor temperature and a bottoms fraction comprising about 60% by weight is recovered and evaluated. The characteristics of the bottoms fraction are reported in the following Table I.

TABLE I.—VACUUM REDUCED RESINOUS PITCH
(60% by wt. bottoms)

S.P. (R&B), ° F	286
Specific gravity 77/77° F.	1.219
Penetration:	
180/100/5	1/2
180/100-5	2

TABLE III.—VACUUM DISTILLATION OF BLENDS OF RESINOUS PITCH AND THERMAL ASPHALT

Product percent by Wt.:	75	50	25
Thermal Asphalt	25	50	75
Resinous Pitch			
Btms. percent by Wt.	79.5	78.5	69.0
S.P. (R&B), ° F	211	254	236
Sp. Gr., 77/77° F	1.219	1.203	1.209
Penetration at—			
130/100/5	1	5	0
140/100/5	5	12	0
150/100/5	12	26	1
160/100/5		60	3
170/100/5			217
180/100/5			
Viscosity at—			
300° F. SFS/cs.	691/1470	346/736	3137/6675
350° F.	85/181	565/1202	52/111
400° F.	22/47	86/183	15/32
Solubility:			
CCl ₄ , percent	99.02	95.60	95.18
CS ₂ , percent	99.35	96.39	99.37
Conradson Carbon, percent	41.0	45.9	33.4
Sulfur	0.62	0.61	0.43
Heat Hardening (485° F., 5 hr., 200 g.):			
Percent Loss	6.0	6.5	11.0
S.P. (R&B), ° F	233	284	242
S.P. Increase	22	30	46
Sp. Gr., 77/77° F	1.212	1.219	1.208
			1.212
			1.203
			1.207

Solubility:	
CCl ₄ , percent	90.11
CS ₂ , percent	98.20
Viscosity at:	
400° F. SFS/cs.	370/787
450° F.	78/195
Flash, COC, ° F.	550
Conradson carbon, percent	50.2
Heat hardening, (485° F., 5 hr., 200 g.):	
Percent Loss	3.5
S.P. (R&B), ° F.	296
S.P. increase	10
Sp. gr., 77/77° F.	1.219
Pen. 180/100/5	1/2

This bottoms fraction of Example 1 is then blended with various proportions of 112° F. Softening point thermal asphalt fraction as reported in Table II.

The characteristics of the resulting product are measured and, in addition, this thermal asphalt is subjected to a heat hardening test to determine its heat stability.

Heat hardening test

According to this test, 200 grams of a thermal asphalt product are placed in a seamless tin box and heated

to a temperature of 480° F. for a period of 5 hours. At the termination of the test, the sample is removed from the tin box and its weight, softening point and specific gravity determined. From this data the percent weight loss and the softening point increase for the sample are obtained.

The characteristics of the thermal asphalt product and the heat hardening test results are given in the following Table II.

It will be noted from these test results that thermal asphalt product obtained from the blends of resinous pitch and thermal asphalt exhibited percent weight losses of only 10.2%, 8.4% and 5.6% respectively compared to percent weight loss of 22.2% incurred by the thermal asphalt containing no resinous pitch.

EXAMPLE 2

In this embodiment, the resinous pitch 60% bottoms fraction described in Table I is blended with varying amounts of the 112° F. softening point asphalt fraction and the resulting blend is then vacuum distilled under 2 mm. (Hg) pressure and at 870° F. corrected vapor temperature to a desired softening point grade.

The characteristics of the resulting thermal asphalt product and the heat hardening test results thereof are reported in Table III.

It will be seen from the foregoing results that the thermal asphalts produced according to this aspect of the invention possessed good thermal stability as evidenced by their low weight losses and the relatively small increases in their softening points.

It will be appreciated that many variations and modifications can be practiced without departing from the scope or spirit of the present invention.

Having thus described the invention, what we desire to secure and claim by Letters Patent is:

1. A process for preparing heat-stable thermal asphalts which comprises:

blending a resinous pitch material deficient in high-boiling aromatic oils and having a softening point (R&B) from about 75° F. to about 140° F., a specific gravity at 77° F. from about 1.0 to about 1.3 and a Conradson Carbon number from about 25 to 45%, with

a solid material containing at least about 20% by weight of high boiling aromatic oils and having a softening point (R&B) from about 80° F. to about 120° F.; a specific gravity at 77° F. from about 1.0 to about 1.3; and a Conradson Carbon number from about 20 to about 35%; and

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distilling the resulting blend to recover a heat-stable thermal asphalt having a softening point (R&B) from about 150° F. to about 270° F.

2. The process of claim 1, wherein said resinous pitch possesses a softening point (R&B) from about 100 to about 130° F. 5

3. The process of claim 1, wherein said resinous pitch is a bottoms fraction obtained from the cracking furnace of an ethylene unit.

4. The process of claim 1, wherein the resinous pitch is used in proportions ranging from about 20 to about 80% of said blend. 10

5. The heat-stable thermal asphalt produced by the process of claim 1.

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UNITED STATES PATENT OFFICE

Certificate of Correction

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It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Columns 3 and 4, Table III, first column, line 13 thereof, for "180/100/5" read —170/100/5—; column 4, after line 15 insert the following:

TABLE II.—PROPERTIES OF BLENDS OF RESINOUS PITCH BOTTOMS AND THERMAL ASPHALT FRACTION

Product Percent Wt.:				
Thermal Asphalt 112° F. S. P.-----	100	75	50	25
60% Vac. Red. Pitch Btms.-----	0	25	50	75
S.P. (R&B), ° F.-----	112	144	189	230
Sp. Gr., 77/77° F.-----	1. 165	1. 183	1. 200	1. 213
Penetration at—				
77/100/5-----	69	21½	-----	-----
115/100/5-----	-----	86	6	-----
130/100/5-----	-----	246	19	-----
140/100/5-----	-----	-----	35	1
150/100/5-----	-----	-----	74	3
160/100/5-----	-----	-----	-----	9
170/100/5-----	-----	-----	-----	18
Viscosity at—				
250° F. SFS/cs-----	31/66	206/438	-----	-----
300° F-----	9/19	35/74	204/434	234/5179
350° F-----	-----	11/23	40/85	228/485
400° F-----	-----	-----	13/28	48/102
450° F-----	-----	-----	-----	-----
Solubility:				
CCl ₄ , Percent-----	98. 60	97. 47	93. 86	94. 20
CS ₂ , Percent-----	99. 70	99. 05	99. 15	98. 88
Flash, COC, ° F-----	455	485	500	530
Conradson Carbon, Percent-----	26. 7	32. 7	38. 8	45. 0
Sulfur, Percent-----	0. 83	-----	-----	-----
Heat Hardening (480° F., 5 hr., 200 g.):				
Percent Loss-----	22. 2	10. 2	8. 4	5. 6
S.P. (R&B), ° F-----	184	189	224	253
S.P. Increase-----	72	45	35	23
Sp. Gr., 77/77° F-----	1. 195	1. 197	1. 209	1. 215
Penetration at—				
130/100/5-----	17	15	1	0
140/100/5-----	-----	33	2	0
150/100/5-----	-----	72	7	½

Signed and sealed this 22nd day of July 1969.

[SEAL]

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