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COAL TAR PITCH AND A METHOD FOR PREPARING THE SAME

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The present invention relates to improved coal tar pitches and a method for preparing the same.

U. S. Patent No. 2,224,685 of Dec. 10, 1940, relates to a process for obtaining difficultly volatile products from a bituminous substance containing such volatile products, comprising the following steps: said bituminous substance is preheated near to the working temperature to a temperature at which no decomposition occurs and at which said substance is liquid or liquefied; said bituminous preheated substance is then further heated to the working temperature for fractions of a second by mixing said preheated bituminous substance in a locally strictly limited zone with a flowing vaporous or gaseous substance preheated to a temperature above the working temperature; the temperature of the mixture obtained is immediately thereafter reduced, by introducing said mixture under reduced pressure into an unheated expansion chamber, to a temperature at which no decomposition can take place, and the volatilized fractions, which are substantially uncracked, are separated from the non-volatile residue which is substantially only slightly cracked by the process. According to another feature of this invention a process for obtaining difficultly volatile oils from a bituminous pitch selected from the group consisting of coal tar pitch, brown coal tar pitch and petroleum pitch, comprises the following steps: said bituminous pitch is preheated to a temperature of 370° to 390° C. and is mixed with flowing steam of about 600° C. for fractions of a second in a locally strictly limited zone; the temperature of the mixture obtained is immediately thereafter reduced to about 400° C., by introducing said mixture under reduced pressure into an unheated expansion chamber, and the volatilized vapors, which are substantially uncracked, are separated from the non-volatile residue which is substantially only slightly cracked by the process.

According to the process described in the said U. S. Patent No. 2,224,685 and all its claims it is possible to obtain from coal tar or coal tar pitches high-melting distillation residues which means hard pitches having a softening point of for instance 150–300° C. or more.

It has now been found that it is possible to obtain soft pitches of valuable properties from these high-melting coal tar hard pitches (distillation residues) by treating said hard pitches with coal tar oil, coal tar or mixtures of such substances in the heat until a homogeneous mixture is obtained.

Example 1

59 parts by weight of coal tar hard pitch (distillation residue) as prepared according to the

U. S. Patent No. 2,224,685, having a softening point of 280° C. are slowly heated with 41 parts by weight of anthracene oil to 290–300° C. in a vessel provided with an agitator, this temperature being maintained for three hours. The soft pitch thus obtained shows the following properties:

Softening point.....°C..	38
Breaking point.....°C..	—5
Temperature interval between softening point and breaking point.....°C..	43
Flow test (length of flow) 30 minutes at 55° C.....mm..	40
Height of fall at 0° C.....cm..	250

The "height of fall" can be determined by the method described in the periodical "Asphalt und Teer Strassenbautechnik," 1933, pp. 392–393. This method or ball test works as follows: The material to be tested is worked into little balls of 50 g. each. These balls are cooled to 0° C., and caused to fall from different heights upon an iron plate. In this way the height of fall is determined, which causes the test balls to burst or splinter.

Example 2

51 parts by weight of coal tar hard pitch (distillation residue) as prepared according to said U. S. Patent No. 2,224,685 having a softening point of 260° C. are heated for several hours with 49 parts by weight of heavy coal tar oil (impregnation oil) to 290° C. The light parts of the impregnation oil which have been distilled off during this operation are again added after cooling the mixture. The soft pitch obtained has the following properties:

Softening point.....°C..	32
Breaking point.....°C..	—8
Temperature interval between softening point and breaking point.....°C..	40
Flow test (length of flow).....mm..	60
Height of fall at 0° C.....cm..	250

Example 3

37 parts by weight of coal tar hard pitch (distillation residue) as prepared according to said U. S. Patent No. 2,224,685, having a softening point of 350° C. are heated for several hours with a mixture of 37 parts by weight of "prepared coal tar" and 26 parts by weight of benzene wash oil. The soft pitch thus obtained has the following properties:

Softening point.....°C..	37
Breaking point.....°C..	—10
Temperature interval between softening point and breaking point.....°C..	47
Flow test (length of flow).....mm..	28
Height of fall at 0° C.....cm..	225

Example 4

38 parts by weight of coal tar hard pitch (distillation residue) as prepared according to the U. S. Patent No. 2,224,685, having a softening point of 350° C. are heated for several hours with a mixture of 38 parts by weight of "prepared coal tar" and 24 parts by weight of anthracene oil. The soft pitch thus obtained has the following properties:

Softening point.....°C.	38
Breaking point.....°C.	-6
Temperature interval between softening point and breaking point.....°C.	44
Length of flow.....mm.	15
Height of fall at 0° C.....cm.	175

Example 5

36 parts by weight of coal tar hard pitch (distillation residue) as prepared according to said U. S. Patent No. 2,224,685, having a softening point of 280° C. are mixed in the heat with 41 parts by weight of distilled coal tar. Further 23 parts by weight of heavy coal tar oil are added to this mixture. The soft pitch thus obtained has the following properties:

Softening point.....°C.	32
Breaking point.....°C.	-5
Temperature interval between softening point and breaking point.....°C.	37
Length of flow.....mm.	39
Height of fall at 0° C.....cm.	145

Example 6

37 parts by weight of coal tar hard pitch (distillation residue) as prepared according to said U. S. Patent No. 2,224,685, having a softening point of 280° C. are treated for 4 hours at 300° C. with 54 parts by weight of "prepared coal tar" in a vessel provided with an agitator. After cooling the product obtained to about 150° C. the oil which was distilled off during the heating, is again added and the desired softening point is arrived at by adding further 9 parts of "prepared coal tar." The soft pitch thus obtained has the following properties:

Softening point.....°C.	33
Breaking point.....°C.	-5
Temperature interval between softening point and breaking point.....°C.	38
Length of flow.....mm.	27
Height of fall at 0° C.....cm.	160

The following table illustrates the progress obtained in the art by the present process, the soft pitches prepared according to the preceding examples being compared with a normal coal tar soft pitch and Mexico asphalt.

Table

Material	Softening point	Breaking point	Interval between softening point and breaking point	Length of flow at 65° C.	Height of fall at 0° C.
	° C.	° C.	° C.	Mm.	Cm.
Normal coal tar soft pitch.	40.5	+8.5	32	More than 140	20
Soft pitch—					
Example 1.....	38	-5	43	40	250
2.....	32	-8	40	60	250
3.....	37	-10	47	28	225
4.....	38	-6	44	15	175
5.....	32	-5	37	39	145
6.....	33	-5	38	27	160
Mexico asphalt.....	39.5	-12	51.5	48	120

The foregoing data show that the properties of the soft pitches prepared according to the present invention resemble more a typical petroleum asphalt than a typical coal tar soft pitch. Especially the soft pitches produced according to the present invention do not show the great sensitiveness to temperature commonly found with normal coal tar soft pitches which renders these normal pitches to be brittle and sensitive to shock and impact when exposed to low temperatures and to soften at even moderately increased temperatures.

Besides it has been found that further viewpoints regarding the addition of the diluent, namely of coal tar oil or coal tar must be taken into consideration, as they are of importance to the properties of the soft pitch obtained. In the first place it is advisable to use as diluent a product which is free of or poor in crystals, the presence of crystallizable ingredients being able to impair the properties of the soft pitch obtained. Furthermore the proportion between hard pitch and diluent in the mixture should be adjusted in such a way as to prevent surface skin formation on solidification of the mixture, if the mixed product is to be of good adhesivity. By adding further quantities of the diluting agent it is easily possible to remove any skin that may have been formed without essentially changing the softening point. Formation of skin otherwise adversely affects the adhesive power of the mixed product although the latter is equal to a high grade petroleum asphalt as regards its other technical properties.

In manufacturing the soft pitches according to the present invention attention must be given to the fact that the treatment of the hard pitches (distillation residues) prepared according to said U. S. Patent No. 2,224,685 with tar or tar oil in the heat requires a certain time in order to obtain a really homogeneous mixed product. In this case the hard pitch probably will swell in the diluting agent employed, that is to say a colloidal system is formed the homogeneity of which requires a certain time of treatment (see the examples). If the time of treatment is not sufficient then the originally obtained physical data of the mixed product are subject to changes owing to subsequent swelling of the hard pitch portion in the mixture.

According to the present invention it is possible to obtain from coal tar a product which is equal to and even in some respects better than petroleum asphalt as regards its technical properties.

What we claim is:

1. A process for producing a coal tar pitch of low sensitiveness to changes of temperature, which comprises mixing a coal tar hard pitch which has been substantially freed from volatile constitu-

ents and is at most slightly cracked, with a material selected from the group consisting of coal tar and coal tar oils, and heating said mixture to about 290°-300° C. for several hours.

2. A process for producing a coal tar pitch of low sensitiveness to changes of temperature, which comprises mixing a coal tar hard pitch which has been substantially freed from volatile constituents and is at most slightly cracked, with a material which is poor in crystalline constituents and is selected from the group consisting of coal tar and coal tar oils, and heating said mixture to about 290°-300° C. for several hours.

3. A process for producing a coal tar pitch of low sensitiveness to changes of temperature, which comprises mixing a coal tar hard pitch which has been substantially freed from volatile constituents and is at most slightly cracked, with a material selected from the group consisting of coal tar from which the lower-boiling constituents had been expelled, and high-boiling coal tar oils, and heating said mixture to about 290°-300° C. for several hours.

4. A process for producing a coal tar pitch of low sensitiveness to changes of temperature, which comprises mixing a coal tar pitch with a softening point of 150° to 400° C. which has been substantially freed from volatile constituents and is at most slightly cracked, with a material selected from the group consisting of coal tar and coal tar oils, and heating said mixture to about 290°-300° C. for several hours.

5. A stable coal tar pitch of low sensitiveness to changes of temperature, which shows an interval of about 37° to 43° C. between its softening and breaking point, a length of flow of about 15 to 60 mm. at 65° C., and a height of fall of about 145 to 250 cm. at 0° C., said stable coal tar pitch consisting of a mixture of a coal tar hard pitch which has been substantially freed from volatile constituents, and is at most slightly cracked, with a material selected from the group consisting of coal tar and coal tar oils.

6. A stable coal tar pitch of low sensitiveness to changes of temperature, which shows an interval of about 37° to 43° C. between its softening point and breaking point, a length of flow of about 15 to 60 mm. at 65° C., and a height of fall of about 145 to 250 cm. at 0° C., said stable coal tar

pitch consisting of a mixture of a coal tar hard pitch which has been substantially freed from volatile constituents and is at most slightly cracked, with a material which is poor in crystalline constituents, and is selected from the group consisting of coal tar and coal tar oils.

7. A stable coal tar pitch of low sensitiveness to changes of temperature, which shows an interval of about 37° to 43° C. between its softening point and breaking point, a length of flow of about 15 to 60 mm. at 65° C., and a height of fall of about 145 to 250 cm. at 0° C., said stable coal tar pitch consisting of a mixture of a coal tar hard pitch which has been substantially freed from volatile constituents, and is at most slightly cracked, with a material selected from the group consisting of coal tar from which the lower-boiling constituents had been expelled and high-boiling coal tar oils.

8. A stable coal tar pitch of low sensitiveness to changes of temperature, which shows an interval of about 37° to 43° C. between its softening point and breaking point, a length of flow of about 15 to 60 mm. at 65° C., and a height of fall of about 145 to 250 cm. at 0° C., said stable coal tar pitch consisting of a mixture of coal tar hard pitch with a softening point of 150° C. to 400° C., which has been substantially freed from volatile constituents and is at most slightly cracked, with a material selected from the group consisting of coal tar and coal tar oils.

9. A stable coal tar pitch of low sensitiveness to changes of temperature, which shows an interval of about 37° to 43° C. between its softening point and breaking point, a length of flow of about 15-50 mm. at 65° C., and a height of fall of about 145-250 cm. at 0° C., said stable coal tar pitch consisting of a mixture of a coal tar hard pitch with a softening point of 150 to 400° C., which has been substantially freed from volatile constituents, and is at most slightly cracked, with a material selected from the group consisting of coal tar from which the lower-boiling constituents had been expelled, and high-boiling coal tar oils.

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