

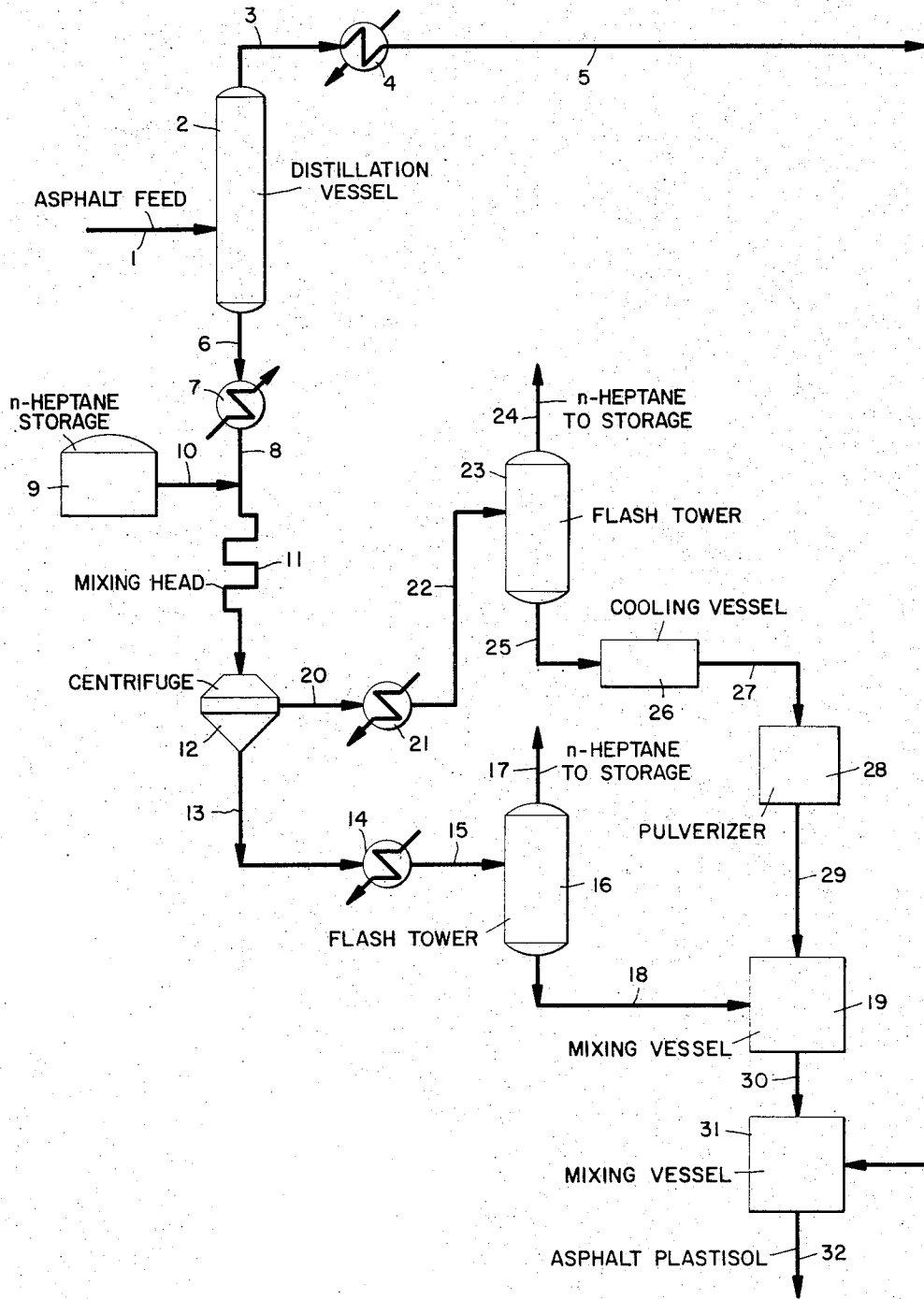
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ASPHALT PLASTISOLS

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3,360,455

ASPHALT PLASTISOLS

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ABSTRACT OF THE DISCLOSURE

Asphalts are fractionated into asphaltenes, high molecu-
lar weight petrolenes and low molecular weight petro-
lenes. The high molecular weight petrolene fraction is
coated with the asphaltene fraction and thereafter sus-
pended in the low molecular weight petrolene fraction to
form a stable asphalt plastisol.

This invention relates to asphalt compositions having
improved flow properties and more particularly to stable
asphalt plastisols and their preparation.

The general method used in preparing hot-mix pave-
ments is by preheating mineral aggregate to approxi-
mately 325° F. and coating this aggregate with asphalt
ranging in softening point from about 105° F. up to
about 140° F. This asphalt is also preheated to approxi-
mately 300° F. so that it will effectively spread over the
mineral particle surfaces. The practical difficulties en-
countered with this general method are the large amounts
of heat that must be employed in order to fluidize the
asphalt binder and the inherent problems encountered in
storing and transferring the asphalt in this fluid condi-
tion.

Thus, it is a primary object of this invention to pro-
vide useful asphalt compositions which are fluid and of
low viscosity at ordinary ambient temperatures and which
can be easily stored, transferred and mixed without the
application of large amounts of heat.

The present invention is predicated on the discovery
that asphalt can be separated into several fractions which
can subsequently be recombined in a particular manner
to form a stable asphalt plastisol having substantially im-
proved flow properties and which can be used in the same
application as the original asphalt composition. As is well
known in the art, asphalt can be regarded as comprising
two general components, i.e., petrolenes and asphaltenes.
The asphaltenes are distinguished from the petrolenes in
that asphaltenes have a much higher molecular weight,
a higher boiling point and are higher in aromaticity. Both
materials are themselves mixtures of hydrocarbons and
their heteroatom analogs.

In accordance with this invention, asphalt composi-
tions are rendered fluid by a process comprising the neces-
sary steps of: (1) admixing asphaltenes and nonvolatile
petrolene particles having an initial atmospheric boiling
point within the range between about 600° F. and 1400°
F. to form asphaltene-coated nonvolatile petrolene par-
ticles and (2) admixing the said coated particles with
liquid volatile petrolenes to produce an asphalt plastisol.

When starting with a typical asphalt feed, an embodi-
ment of this invention comprises (1) distilling the as-
phalt at elevated temperatures and reduced pressures to
separate the lower boiling fractions, i.e., the volatile
petrolenes, from the nonvolatile fraction comprised of
asphaltenes and nonvolatile petrolenes and having an ini-
tial boiling point within the range between 600° and
1400° F., (2) contacting the nonvolatile fraction with a
liquid paraffin to precipitate the asphaltenes and form an
asphaltene fraction and a nonvolatile petrolene fraction,
(3) separating said asphaltene fraction from said non-
volatile petrolene fraction, (4) removing said liquid

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paraffin from said asphaltene fraction and said nonvola-
tile petrolene fraction, (5) cooling the said nonvolatile
petrolene fraction, (6) pulverizing the said cooled non-
volatile petrolene fraction, (7) admixing the said as-
phaltenes and said pulverized nonvolatile petrolenes to
form asphaltene-coated nonvolatile petrolene particles
and (8) admixing the said coated particles with the said
volatile petrolenes to produce an asphalt plastisol.

The invention will be more fully understood by refer-
ring to FIGURE 1 which shows a flow diagram of one
embodiment of the same.

While the process of the present invention may be
employed to fluidize asphalts, residua, heavy fuel oils and
the like, it has special application to asphalts. The present
invention contemplates the employment of any asphalt
composition comprising petrolenes and asphaltenes. The
asphalt composition is not critical because no modifica-
tion of the composition is produced by the practice of the
present invention but rather what is accomplished is the
physical modification of the asphalt in such a manner
that its flow properties are vastly improved. Illustrative of
this is the fact that a solid asphalt paving binder can be
processed according to the present invention to yield a
fluid, low viscosity asphalt paving binder which can sub-
sequently be added to the hot mineral aggregate to pro-
duce an asphalt pavement having the same properties as
would be obtained by the use of the hot mineral aggregate
with the normally solid asphalt paving binder. Broadly,
the asphalts which are operable in the concept
of this invention may contain from about 20 to about 60
wt. percent volatile petrolenes, from about 10 to about
50 wt. percent nonvolatile petrolenes (i.e., those petro-
lenes having an initial boiling point within the range be-
tween 600° and 1400° F.) and from about 5 to about 40
wt. percent asphaltenes. Nonlimiting examples of asphalts
which may be employed are straight reduced asphalts,
air blown asphalts and blended asphalts having the fore-
going compositions. The asphalts may be, for example,
derived from Lagunillas, Boscan, Aramco, Talco, Kuwait,
Oregon Basin, Lloydminster, Santa Maria, Kern River,
and Eucutta crudes and the like. In addition, the asphalt
plastisol may be formulated from the total amount of
materials fractionated from the asphalt or it may be
formulated from only a portion of the materials frac-
tionated provided, however, that the resultant product
composition is within the beforementioned operable
range.

The precipitant used to separate asphaltenes from the
nonvolatile petrolenes includes any liquid paraffin which,
when contacted with a petroleum fraction containing as-
phaltenes, causes said asphaltenes to precipitate as solids.
Such precipitants are well known in the art, e.g., U.S.
Patent 3,087,887, which is incorporated herein by refer-
ence. Such precipitants include the C₅ to C₁₂ paraffins,
preferably the saturated C₅ to C₉ normal paraffins. Non-
limiting examples of suitable precipitants are n-pentane,
n-hexane, n-octane, isooctane, dodecane and the like.

The asphalt is fed into a distillation zone where the
volatile petrolenes are separated in accordance with known
techniques from the nonvolatile fraction comprising non-
volatile petrolenes and asphaltenes. The temperature and
pressure employed will depend upon the particular asphalt
which is to be fractionated. In general, asphalts having
higher boiling points will require lower distillation pres-
sures and/or higher distillation temperatures. Broadly,
the conditions used include a temperature of from about 400°
to about 800° F. and a pressure less than atmospheric,
i.e., between about .1 and about 50 mm. of mercury
absolute. It is preferred, however, that the temperature
be less than 750° F. and the pressure be less than 2 mm.
Hg. Temperatures in excess of 800° F. should be avoided
in order to minimize any possible thermal degradation of

the asphalt. The temperature, pressure and time in the distillation zone should be adjusted so that the initial atmospheric boiling point of the nonvolatile fraction (i.e., the distillation bottoms) which is withdrawn will be within the range between about 600° and 1400° F. The boiling point range of the volatile petrolenes (i.e., the distillation overhead) will be, of course, within the range between the initial boiling point of the asphalt feed and the initial boiling point of the aforescribed nonvolatile fraction.

The nonvolatile fraction recovered from the distillation zone is contacted with the aforescribed liquid paraffin into which asphaltenes precipitate as solids. The temperatures and pressures used may vary widely. It has been found that this separation can be conveniently effected at a temperature of from about 50° to about 200° F. and at atmospheric pressure. Contact time is not critical since upon contact of the nonvolatile fraction with the liquid paraffin, precipitation of the asphaltenes as solids is very rapid. The precipitated asphaltene fraction is then subsequently removed from the petrole fraction by filtration or by centrifugation, preferably the latter. The separated asphaltene fraction and the nonvolatile petrole fraction are then separately treated in order to isolate the respective products. For example, the separated asphaltenes may be dried in air at ambient temperatures or at elevated temperatures (e.g., 150° F.) under inert atmosphere. The separated petrolenes may be conveniently recovered from solution by distilling off the liquid paraffin at a temperature of 100° to 175° F. and a pressure of 25 to 100 mm. Hg absolute.

The nonvolatile petrolenes recovered from the liquid paraffin solution are cooled to temperatures below about 60° F., preferably below about 40° F., and pulverized at this temperature to pass a number 8 U.S. Standard Sieve screen. Nonvolatile petrole particles of from about number 200 to about number 8 U.S. Standard Sieve size have been found to be particularly effective in the concept of this invention.

In accordance with this invention the cooled nonvolatile petrolenes are admixed after pulverizing with asphaltenes until the surface of the nonvolatile petrole particles are coated with the dust-like asphaltenes. The asphaltenes, being essentially infusible and dust-like, tend to coat or dust the surface of the ground petrolenes. This coating technique is desirable to prevent coalescing of the nonvolatile petrole particles. The foregoing pulverizing and mixing operations can be effected in any conventional equipment such as a pulverizing mill or a ball mill. The asphaltene-nonvolatile petrole blend is subsequently admixed at ambient temperatures (e.g., 60°-100° F.) with the volatile petrolenes which being liquid act as a plasticizer and produce an asphalt plastisol, i.e., a dispersion of a finely divided solid in a liquid. Broadly, these asphalt plastisols comprise from about 10 to about 50 wt. percent nonvolatile petrolenes, from about 5 to about 40 wt. percent asphaltenes and from about 20 to about 60 wt. percent volatile petrolenes. It is preferred, however, that they comprise from about 15 to about 30 wt. percent nonvolatile petrolenes, from about 20 to about 35 wt. percent asphaltenes and from about 35 to about 50 wt. percent volatile petrolenes.

Turning now to the figure, a straight reduced residuum from a Venezuelan crude having an ASTM softening point of 115° F., an ASTM penetration @ 77° F. of 90, and composed of 84 wt. percent petrolenes and 16 wt. percent asphaltenes is used as the feed in the specific embodiment to be described hereinafter. The feed is heated to 270° F. and pumped through line 1 into distillation vessel 2 which may be a vacuum pipestill. The temperature and pressure employed are approximately 700° F. and 5 mm. Hg absolute respectively. The low boiling material, i.e., the volatile petrolenes, are taken off overhead through line 3 and condensed in condenser 4 at about 180° F. and then pumped through line 5 into mixing vessel 31.

The bottoms from vessel 2, i.e., the nonvolatile fraction comprising asphaltenes and nonvolatile petrolenes and having an initial boiling point of about 1150° F., are withdrawn through line 6 as a liquid at about 500° F., cooled in heat exchanger 7 to about 300° F., and then passed through line 8. Liquid normal heptane from storage 9 in an amount sufficient to precipitate the asphaltenes is then pumped via line 10 into contact with the nonvolatile fraction in line 8. The mixture ratio is preferably 12/1 of normal heptane to nonvolatile fraction by volume, although this may vary from about 4/1 to 50/1. Other liquid paraffins as hereinbefore mentioned may be used in place of normal heptane. The mixture is then passed through a mixing head 11 or some other suitable mixing device and thence into a continuous type high speed centrifuge 12 wherein the precipitated asphaltenes are separated from the nonvolatile petrole-hexane solution. The asphaltene-hexane slurry rejected from the centrifuge is then continuously pumped through line 13 into heat exchanger 14 wherein it is heated to about 250° F. and thence to flash tower 16 by means of line 15 for stripping at atmospheric pressure. Normal heptane is taken overhead through line 17, subsequently condensed and sent to storage. The bottoms from flash tower 16, i.e. the asphaltenes, are withdrawn as solids via line 18 and sent to mixing vessel 19. The nonvolatile petrolenes from centrifuge 12 are withdrawn through line 20, heated in heat exchanger 21 to about 250° F. and sent to flash tower 23 via line 22 wherein normal heptane is taken overhead through line 24 for reuse and the nonvolatile petrole bottoms is withdrawn through line 25. The nonvolatile petrolenes are then cooled to about 20° F. in cooling vessel 26 and then conveyed as solids via line 27 to pulverizer 28 which may be a rod mill or ball mill. The ground nonvolatile petrolenes are then sent by means of line 29 into mixing vessel 19 for admixture with the asphaltenes at a temperature of about 20° F. The solid asphaltene-nonvolatile petrole blend is sent via line 30 into mixing vessel 31 wherein it is combined at 75° F. with the volatile petrolenes to yield an asphalt plastisol comprising 51 wt. percent nonvolatile petrolenes, 16 wt. percent asphaltenes and 33 wt. percent volatile petrolenes.

The invention can more fully be understood by reference to the following examples.

Example 1

An asphalt, i.e., a straight reduced Venezuelan crude oil residuum, having an ASTM softening point of 85° F. and a viscosity at 77° F. of 6.5×10^4 poises, was processed according to the process illustrated in the figure. The yield of the fractions separated from the asphalt were based on the total weight of the asphalt fractionated, 44 wt. percent volatile petrolenes, 13 wt. percent asphaltenes and 43 wt. percent nonvolatile petrolenes. These materials were combined in accordance with this invention to form an asphalt plastisol having an ASTM softening point of 50° F. This asphalt plastisol was then combined with a mineral aggregate commonly used in paving, i.e., an Asphalt Institute type VIa graded aggregate. This was done by the conventional hot-mix method except that the aggregate was heated to 400° F. and the binder was added cold as a plastisol rather than as a hot asphalt liquid. After mixing for 5 minutes, Marshall briquets were prepared and tested by the Marshall test method. The original solid asphalt was also combined with the aforescribed mineral aggregate by the conventional hot-mix process, i.e., both the nonplastisol asphalt and the mineral aggregate were preheated prior to admixture. This admixture was also tested by the Marshall test method. The comparative test results for test specimens containing 6.5 wt. percent of the plastisol or nonplastisol and 93.5 wt. percent of the aforescribed aggregate are shown in Table I. It is seen from the foregoing and Table I that the practice of the present invention results in a substantial improvement in the asphalt fluidity without any

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detrimental change in the performance properties of the original nonplastisol asphalt.

The foregoing admixture is repeated but the components are not combined according to the present invention, i.e., the volatile petroleues are admixed with the pulverized nonvolatile petroleues prior to admixture with the asphaltenes. This admixture results in an asphalt having essentially the same softening point and viscosity as the original asphalt and clearly demonstrates the criticality of the admixing sequence.

TABLE I.—MARSHALL TEST¹

	Binder Used	
	Nonplastisol Asphalt	Asphalt Plastisol
Voids, percent.....	6.0	6.0
Marshall Stability 140° F., lbs.....	760	750
Marshall Flow 140° F., .01".....	10	10

¹ ASTM D 1559-62T=Test for Resistance to Plastic Flow of Bituminous Mixtures Using Marshall Apparatus.

Example 2

As hereinbefore mentioned, asphalt plastisols may be formulated from the total amount of materials fractionated from the asphalt, as in Example 1, or they may be formulated from only a portion of the fractions separated. The latter procedure has the advantage that the performance properties can be improved by the proper selection of the amounts of materials to be combined to yield the plastisol. Portions of the separated volatile petroleues, asphaltenes and nonvolatile petroleues from Example 1 were combined to formulate a plastisol consisting of 47½ wt. percent volatile petroleues, 38 wt. percent asphaltenes and 14½ wt. percent nonvolatile petroleues. The resultant plastisol had an ASTM softening point of 60° F. and a viscosity at 77° F. of 7.8×10^6 poise. A portion of this plastisol was then fluxed at 400° F. to break down the plastisol and to yield a nonplastisol asphalt having the same composition of the plastisol. This nonplastisol asphalt had a softening point of 108° F. and a viscosity at 77° F. of 1.4×10^6 poise. As in Example 1, Marshall briquets were prepared from both the asphalt plastisol and the solid nonplastisol asphalt. The comparative test results for test specimens containing 6.5 wt. percent of the plastisol or nonplastisol and 93.5 wt. percent of an Asphalt Institute type VI aggregate are shown in Table II. It is again seen that the fluidity of the asphalt has been substantially improved without any detrimental effect on the asphalt performance properties.

TABLE II.—MARSHALL TEST¹

	Binder Used	
	Nonplastisol Asphalt	Asphalt Plastisol
Voids, percent.....	3.5	3.2
Marshall Stability 140° F., lbs.....	2,200	2,460
Marshall Flow 140° F., .01".....	10	9

¹ ASTM D 1559-62T.

Example 3

As in Example 2 only a portion of the separated fractions from Example 1 were combined according to the present invention. The composition of the resultant plastisol was 35 wt. percent volatile petroleues, 15 wt. percent asphaltenes and 50 wt. percent nonvolatile petroleues. This asphalt plastisol had an ASTM softening point of 77° F. In contrast, the nonplastisol asphalt having the same composition as the plastisol had an ASTM softening point of 109° F. and an ASTM penetration at 77° F. of 145. Marshall briquets were prepared as in Example

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1 from both the foregoing asphalt plastisol and the nonplastisol asphalt. The test results for test specimens containing 6.5 wt. percent of the plastisol or nonplastisol and 93.5 wt. percent of an Asphalt Institute type VI aggregate are given in Table III and again show that this invention affords a method for substantially improving the fluidity of asphalt without adversely affecting the performance characteristics of the asphalt.

TABLE III.—MARSHALL TEST¹

	Binder Used	
	Nonplastisol Asphalt	Asphalt Plastisol
Voids, percent.....	4.0	4.5
Marshall Stability 140° F., lbs.....	1,500	1,425
Marshall Flow 140° F., .01".....	11	12

¹ ASTM D 1559-62T.

It is not intended to restrict the present invention to the foregoing examples but rather it should be only limited by the appended claims.

25 What is claimed is:

1. An asphalt plastisol comprising nonvolatile petroleum particles, having an initial atmospheric boiling point within the range of 600° and 1400° F., passing a number 8 U.S. Standard Sieve screen, coated with asphaltenes and suspended in liquid volatile petroleues having a boiling point range below that of the said nonvolatile petroleues, said asphalt plastisol being comprised of from about 10 to about 50 wt. percent of said nonvolatile petroleues, from about 5 to about 40 wt. percent of said asphaltenes and from about 20 to about 60 wt. percent of said liquid volatile petroleues.

2. An asphalt plastisol according to claim 1 wherein the said asphalt plastisol comprises from about 15 to about 30 wt. percent of said nonvolatile petroleues, from about 20 to about 35 wt. percent of said asphaltenes and from about 35 to 50 wt. percent of said liquid volatile petroleues.

3. A process for the manufacture of asphalt plastisols which comprises admixing asphaltenes and nonvolatile petroleum particles having an initial atmospheric boiling point within the range between about 600° F. and 1400° F. to form asphaltene-coated nonvolatile petroleum particles, admixing the said coated particles with liquid volatile petroleues having a boiling point range below that of said nonvolatile petroleues to produce an asphalt plastisol comprising from about 10 to about 50 wt. percent nonvolatile petroleues, from about 5 to about 40 wt. percent asphaltenes and from about 20 to about 60 wt. percent volatile petroleues.

4. A process according to claim 3 wherein the said nonvolatile petroleum particles have been passed through a number 8 U.S. Standard Sieve screen.

5. A process for the manufacture of asphalt plastisols which comprises:

(1) functioning an asphalt comprised of asphaltenes and petroleues into (a) a volatile petroleum fraction having an atmospheric boiling point below about 600° F. and (b) a nonvolatile fraction comprised of asphaltenes and nonvolatile petroleues and having an initial atmospheric boiling point within the range between about 600° and 1400° F.;

(2) contacting the nonvolatile fraction with a C₅-C₉ normal paraffin to precipitate the asphaltenes and form an asphaltene fraction and a nonvolatile petroleum fraction;

(3) separating said asphaltene fraction from said nonvolatile petroleum fraction;

(4) removing the C₅-C₉ normal paraffin from the asphaltene fraction and the nonvolatile petroleum fraction;

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- (5) cooling the nonvolatile petrolene fraction to a temperature below about 60° F.;
- (6) pulverizing the cooled nonvolatile petrolene fraction;
- (7) admixing the asphaltenes and the pulverized nonvolatile petrolenes to form asphaltene-coated nonvolatile petrolene particles; and
- (8) admixing the coated particles with the volatile petrolenes produced in step (1) to form a stable asphalt plastisol consisting essentially of from about 10 to about 50 wt. percent of said nonvolatile petrolenes, from about 5 to about 40 wt. percent of said asphaltenes and from about 20 to about 60 wt. percent of said volatile petrolenes.

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