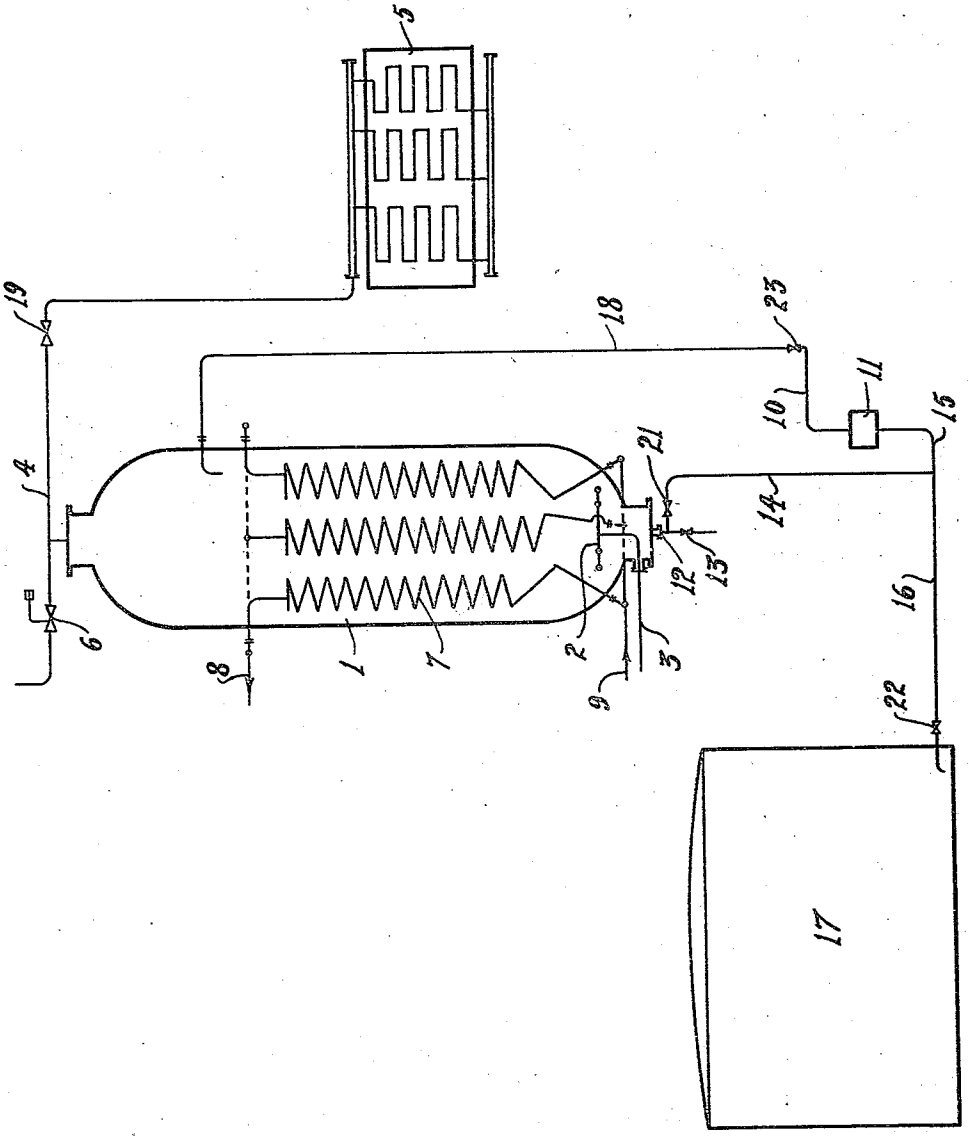
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PRODUCTION OF ASPHALTIC PRODUCTS FROM AROMATIC EXTRACTS OF MINERAL OILS

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PRODUCTION OF ASPHALTIC PRODUCTS FROM AROMATIC EXTRACTS OF MINERAL OILS

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This invention relates to the production of asphaltic products from aromatic extracts of mineral oils.

The manufacture of an asphaltic product (or straight asphalt) by the steam reduction or distillation of asphaltic petroleum residues containing paraffins and which are generally semi-solid at ordinary temperatures is well known, and it is also known to blow such products to obtain grades of harder consistency and correspondingly higher melting points. It is necessary however to effect the steam distillation prior to the blowing operation, in order to remove the bulk or major portion of the paraffins which would otherwise cause the blown product to form a sludge.

The present invention consists in treating such distillates of petroleum crude oil as intermediate distillate or the oil obtained by pressing wax distillate or lubricating oil base by extraction in known manner with sulphur dioxide (the Edeleanu process) aniline, phenol or other selective solvents of the aromatic and unsaturated hydrocarbons and the petroleum resins, and converting the aromatic extracts so obtained and which are in general liquid at ordinary temperatures into bitumen or asphalt by blowing air through the extract at an elevated temperature and preferably under pressure in order to increase the creation of asphaltenes and reduce volatilization of the volatile constituents.

The products of this invention exhibit higher degrees of ductility for given melting points than are obtainable from straight asphalts and the aromatic extract obtained in the above manner yields, on blowing, asphaltic products with complete absence of sludge formation, owing to the removal of the bulk or major portion of the paraffins by the extraction process.

The present invention also yields products which are quite different in character from straight asphalts and further enables the production of asphalts of any desired consistency and melting point, and it is possible in particular to produce grades combining a relatively high melting point with a normal or medium consistency or penetration value and high ductility, particularly at low temperatures, whereas straight asphalts of high ductility, particularly at low temperatures, are only obtainable at relatively lower melting points and with correspondingly softer consistencies or higher penetration values. Straight asphalts combining a relatively high melting point with a normal or medium penetration value and high ductilities, particularly at low temperatures, have hitherto not been obtainable.

Any extract obtained by the solvents described from the previously mentioned petroleum crude oil distillates, whether the petroleum crude oil be asphaltic, non-asphaltic, or of mixed character may be employed, but it is preferred to use an extract obtained in the above manner from a distillate of low volatility (i. e., containing no constituents boiling below 250° C.). Such an extract is composed chiefly of high boiling aromatic and naphthenic hydrocarbons together with some unsaturated hydrocarbons, petroleum resins and possibly a trace of paraffins and having a nil content of asphaltenes and being entirely free from oxygenated derivatives such as are contained in crude tar oils from coal and similar minerals. Such an extract is of a viscous gummy consistency in contradistinction to those aromatic extracts obtained by the use of the above-mentioned solvents from such petroleum crude oil distillates as kerosene, which are quite fluid and which, when treated according to the process described herein yield a non-homogeneous mixture of oil, resins and coke-like pitch, which mixture has none of the properties of a blown asphalt.

The conversion to bitumen may be accomplished by blowing hot air through the extract in an autoclave, the extract being heated in the autoclave. The air and extract are preferably maintained at a temperature of about 200° C. and a pressure of about 50 pounds per square inch is maintained in the autoclave. The pressure may be increased to reduce volatilization of the charge which should preferably be reduced to below 5% of the charge for economic reasons, or the pressure may be increased to accelerate the asphalt production.

Up to approximately 100 per cent of the extract can be converted by this method into bitumen of any desired penetration value. When blowing has proceeded up to the point at which a bitumen of say 200 units of penetration at 77° F. (as measured by a standard needle type penetrometer) is obtained, lower penetration grades can be secured very rapidly and generally within one hour's further treatment.

The percentage of asphaltenes in the blown product is generally greater than that of steam reduced asphalts of equal consistency or penetration value.

The ductilities of the blown product of this invention are greater, particularly at lower tem-

peratures of say 10°–20° C. than those of steam reduced asphalts.

For example bitumens have been produced by the present invention from an aromatic extract having a setting point (temperature at which solidification occurs) below 15° F., and the air blown product has been found to contain 21.8% asphaltenes for a melting point of 37.5° C. and with the following penetration values and ductilities:—

At 10° C. penetration value 21, ductility over 120 cms.
At 15° C. penetration value 43, ductility over 120 cms.
At 20° C. penetration value 68, ductility over 120 cms.
At 25° C. penetration value 139, ductility over 120 cms.
At 30° C. penetration value 226, ductility over 120 cms.

(120 cms. being the maximum limit of the testing apparatus for the ductility).

Whereas a typical bitumen at present on the market for spray application to road surfaces which has a melting point of 46.5° C. contains 18% asphaltenes and exhibits the following characteristics:—

At 10° C. penetration value 12, ductility 4 cms.
At 15° C. penetration value 23, ductility 22 cms.
At 20° C. penetration value 33, ductility 60 cms.
At 25° C. penetration value 85, ductility over 120 cms.
At 30° C. penetration value 136, ductility over 120 cms.
At 35° C. penetration value 191, ductility over 55 cms.

The bitumen produced by the present invention has greater non-skid properties than steam reduced asphalt, by reason of its high ductility and asphaltene content, the latter rendering the product also less liable to "sweat" in hot weather.

The operation may be performed in any suitable pressure vessel and, if desired, a reflux condenser of known type can be employed to further reduce evaporation. The portion volatilized can be returned to further charges or it may be incorporated for "cutting back" in known manner.

The accompanying drawing diagrammatically illustrates a form of apparatus for the production of asphalt in accordance with the present invention. The drawing shows a vessel 1 in which the conversion of the stock to asphalt is accomplished. In the lower part of the vessel is fitted a perforated blowing pipe 2 for the admission of air supplied by pipe 3. The top of the vessel 1 is connected to a vapor outlet pipe 4 which may lead to a condenser 5. The vapor outlet pipe 4 is fitted with a relief valve 6 which can be set to give any desired pressure within the vessel 1, which is fitted with cooling coils 7, the upper ends of which are connected to an outlet pipe 8. The lower ends of the pipe coils 7 are connected to an inlet pipe 9. The vessel 1 is connected at the bottom to a drain pipe 12 fitted with a drain cock 13. Branching off from drain pipe 12 is a draw-off pipe 14 connected by pipe 15 to the suction side of a pump 11. A pipe 16 also connects pipe 14 and the suction side of the pump 11 to a charging tank 17. Connected by pipe 10 to the delivery side of the pump 11 is a feed pipe 18 which opens into the upper part of vessel 1. A valve 19 controls connection of vapor outlet pipe 4 to condenser 5. A valve 21 controls pipe 14 and a valve 22 controls pipe 16. A valve 23 controls pipe 18.

The mode of operation is as follows:—The vessel 1 is first charged with asphalt-producing stock introduced at a temperature of say 180° to 200° C. To charge the vessel, the charging tank 17 is connected through valve 22 and pipe 16 to pipe 15 and pump 11 which pumps the stock from the tank 17 into vessel 1 through pipes 10 and 18, valve 23 being open. Hot air is then blown into the vessel 1 through pipes 2 and 3. The reaction causes the temperature of the charge to rise and necessitates cooling of the charge, which is effected by circulating a cooling medium through the pipe coils 7, the cooling medium entering through pipe 9 and leaving by pipe 8. Pressure is maintained in the vessel 1 by adjusting the relief valve 6 in the vapor outlet pipe 4 to the desired pressure. Valve 21 is now opened and the charge is drawn off from vessel 1 through pipes 14 and 15 by pump 11 and pumped through pipes 10 and 18 to the upper part of the vessel 1. By this means the temperature of the charge in vessel 1 is controlled and the charge circulated until it is blown to the required grade. When this has been accomplished, the blown charge is allowed to pass off through drain pipe 12 after opening drain cock 13. Preferably about 5 per cent of the blown charge is left in the vessel to mix with the next charge introduced into the vessel in the manner already described, the blown charge left in the vessel promoting more rapid reaction on the blowing of the fresh charge.

I claim:

1. The process of producing asphaltic products from an aromatic extract of a mineral oil obtained by refining the crude oil with a selective solvent chosen from the group consisting of liquid SO_2, aniline and phenol of the aromatic and unsaturated hydrocarbons, which consists in oxidizing the extract unmixed with other hydrocarbons after recovery of the solvent under conditions of oxidation inhibiting distillation.

2. The process of producing asphaltic products from the aromatic extract of a mineral oil obtained by refining the crude oil with a selective solvent chosen from the group consisting of liquid SO_2, aniline and phenol of the aromatic and unsaturated hydrocarbons, which consists in blowing air at an elevated temperature through the extract unmixed with other hydrocarbons, after recovery of the solvent, while maintaining the extract under pressure.

3. The process of producing asphaltic products from mineral oils which consists in eliminating the bulk of the paraffins from the mineral oil by extraction with a selective solvent chosen from the group consisting of liquid SO_2, aniline and phenol of the aromatic and unsaturated hydrocarbons, and thereafter blowing air under conditions inhibiting distillation through the extract unmixed with other hydrocarbons at an elevated temperature after removal of the solvent.

4. The process of producing asphaltic products from mineral oils which consists in eliminating the bulk of the paraffins from the mineral oil by extraction with a selective solvent chosen from the group consisting of liquid SO_2, aniline and phenol of the aromatic and unsaturated hydrocarbons, and thereafter blowing air through the extract unmixed with other hydrocarbons at an elevated temperature while maintaining the extract under pressure.

5. The method of producing asphaltic products from a crude mineral oil which consists in first removing the more volatile constituents boiling

up to about 250° C., thereafter eliminating the bulk of the paraffins from the mineral oil by the selective solvent action of a non-reactive solvent of the aromatic and unsaturated hydrocarbons, separating the solvent therefrom, and finally blowing air through the remaining extract unmixed with other hydrocarbons at an elevated temperature under conditions inhibiting distillation.

6. The process of producing asphaltic products from an aromatic extract of a mineral oil obtained by refining the crude oil with a non-reactive selective solvent of the aromatic and unsaturated hydrocarbons, which consists in blowing air after recovery of the solvent through the remaining extract unmixed with other hydrocarbons at an elevated temperature, while maintaining the air and extract at a temperature of about 200° C. and the extract at a pressure of about 50 pounds per square inch.

7. The process of producing asphaltic products from an aromatic extract of a mineral oil obtained by refining the crude oil with a selective solvent chosen from the group consisting of liquid SO_2, aniline and phenol of the aromatic and unsaturated hydrocarbons, and blowing air after recovery of the solvent through the remaining extract unmixed with other hydrocarbons at an elevated temperature, while reducing volatilization to below 5% of the charge.

8. An asphaltic product obtained by air blowing under pressure and unmixed with other hydrocarbons a mineral oil from which the bulk of the paraffins has been eliminated by an extraction process according to claim 4 and characterized by combining a relatively high melting point with medium consistency and high ductility as exhibited by its higher degree of ductility at lower temperatures of say 10° to 20° C. than hitherto known steam reduced asphalts of similar consistency and melting point.

9. The method of producing asphaltic products, comprising treating a mineral oil with a non-reactive reagent capable of dissolving out aromatic and unsaturated hydrocarbons, recovering the reagent from the resulting aromatic extract, and subjecting the remainder of the aromatic extract unmixed with other hydrocarbons to a current of oxidizing gas while maintaining an elevated temperature and inhibiting distillation.

10. The method of producing asphaltic products which comprises treating a mineral oil with liquid sulphur dioxide, recovering the sulphur dioxide from the resulting extract, and subjecting the remainder of the extract unmixed with other hydrocarbons to a current of oxidizing gas while maintaining an elevated temperature and inhibiting distillation.

11. The method of producing asphaltic products, comprising treating a mineral oil with a non-reactive reagent capable of dissolving out aromatic and unsaturated hydrocarbons, recovering the reagent from the resulting aromatic extract, and subjecting the remainder of the aromatic extract unmixed with other hydrocarbons to a current of oxidizing gas while maintaining a temperature and pressure above atmospheric.

12. The method of producing asphaltic products from aromatic extracts of mineral oils which comprises treating a mineral oil with liquid sulphur dioxide, recovering the sulphur dioxide from the resulting aromatic extract, and subjecting the remainder of the aromatic extract unmixed with other hydrocarbons to a current of oxidizing gas while maintaining a temperature and pressure above atmospheric.

13. The production of asphaltic products which consists in treating a distillate of petroleum crude oil by extraction with selective solvents chosen from the group consisting of liquid sulphur dioxide, aniline and phenol of the aromatic and unsaturated hydrocarbons, and converting such aromatic extracts into asphalt by blowing air through the extract, unmixed with other hydrocarbons at an elevated temperature and under conditions inhibiting distillation after recovery of the solvents.

14. The method claimed in claim 13, characterized in that the extract is obtained from a distillate of low volatility i. e. containing no constituents boiling below 250° C. composed chiefly of high boiling aromatic and unsaturated hydrocarbons, but having no asphaltenes, the extract so obtained being characterized by its consistency.

15. A method of producing asphaltic masses, comprising treating mineral oils with a selective solvent chosen from the group consisting of liquid sulphur dioxide, aniline and phenol, to produce an extract substantially free of asphalt, and then air-blowing this extract unmixed with other hydrocarbons at an elevated temperature and under conditions inhibiting distillation.

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