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⑤④ **Process for separation of solids from liquid hydrocarbons.**

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Description

This invention relates to the processing of liquid hydrocarbons and, more particularly, relates to the removal of insoluble material from liquid hydrocarbons.

Liquid hydrocarbons can include, for example, products derived from liquefaction of a mixture of coal derived liquids or non coal derived liquids plus coal, with or without a catalyst; or products derived from hydroprocessing of a mixture of coal or non coal derived liquids, with or without a catalyst; or combinations thereof.

Although the following description of the process of the invention will proceed with reference to the processing of products of liquefaction of carbonaceous material, it will be understood that this description is exemplary only of the process of the invention applied to the separation of solids from the above liquid hydrocarbons.

Liquid hydrocarbons can be classified into the basic components of oils, asphaltenes and pre-asphaltenes. Insoluble solids may comprise one or more of mineral matter, ash, spent catalyst and unreacted or undissolved carbonaceous residue. The oils are soluble in hexane, the asphaltenes are insoluble in hexane and soluble in toluene and the pre-asphaltenes are insoluble in toluene and soluble in tetrahydrofuran.

Reactor products from liquefaction of carbonaceous material, which is well known in the art for conversion of solid carbonaceous material such as anthracite, bituminous and sub-bituminous coal, lignite and peat, and other carbonaceous material to liquid products are usually in the form of a slurry which contains oils, asphaltene, pre-asphaltenes and insoluble solids.

Removal of insoluble solids from the products of coal liquefaction is desirable to permit optimum recovery and processing of liquid hydrocarbons. The presence of insoluble solids leads to difficulties in the subsequent downstream refining and upgrading of liquid hydrocarbons. Separation of insoluble solids from the coal extract liquids is difficult to effect due to the wide particle size range of the discrete insoluble solids, the relatively high viscosity of the liquid phase even at high temperatures, the small differences between the density of the liquid phase and the density of the solids, and the inherent characteristics of the constituents of the coal liquefaction products.

The separation of discrete mineral matter such as insoluble solids from the coal extract liquids remains a continuing problem. Filtration provides for a high liquid yield by means of washing with a light oil and subsequent recovery of the light oil by drying of the filter cake and separation from the filtrate. A dried filter cake contains typically by weight 5 to 10% liquid product. That is, the cake consists of 90 to 95% solids and 5 to 10% of the desired liquid product on a dried solids cake basis. Filtration, although it provides a good liquid yield, still has as drawbacks: slow filtration

rates, cost of pre-coat materials, and handling of the filter cake. Centrifuges do not achieve as sharp a separation of the solids as by filtration. Also, mechanical problems arise in the continuous removal of solids due to their abrasive and adhesive properties. A centrifuged 'solids cake' typically still contains 50 to 55% of liquid product on a total cake weight basis. Hydroclones achieve an even less sharp separation and are at best used for pre-thickening purposes in combination with other unit operations. Solvent extraction and leaching have been used for removal of only part of the solids. Coarser and heavier particles need to be removed by other means. Magnetic separation processes can only also remove part of the solids. The organic compounds of coal are diamagnetic while the ash, i.e. inorganic mineral matter compounds, are paramagnetic which makes it possible to separate these by magnetic means. However, the unconverted coal cannot be separated. Shou J. K. P. and Collins D. J. describe these problems in: "A Review of Solid-Liquid Separation Technology in Coal Liquefaction Processes", Proceedings of the 28th Can. Chem. Eng. Conf., Publ. by Can. Soc. for Chem. Eng., Ottawa, Canada, 1978.

Distillation or evaporation is a possible means of separation. Very sharp separation can be achieved but liquid carry-over must be minimized. The bottoms of such units typically comprise 55% liquid product and 45% solids, resulting in substantial liquid losses. Coking is another process which provides a sharp separation. However, a considerable amount of liquid product is lost due to gasification of the light oil fraction and due to coking of the heavier liquid hydrocarbon products.

Anti-solvent deashing is a process whereby the solids are co-precipitated with some of the asphaltene and pre-asphaltene portion of the liquid liquefaction product due to the solution equilibrium imbalance brought about by the addition of an anti-solvent. The precipitated solids phase typically comprises 55 to 60% liquid product. Examples of such processes are described in U.S. Patents Nos. 3,790,467; 3,852,182; 3,856,675 and 4,180,456. U.S. Patent 3,790,467 is typical in disclosing the use of an anti-solvent to precipitate from solution "Quasi-solid" materials to cause an increase in size of smaller solids for enhanced separation using size as a separation parameter. Valuable liquefaction product thus is lost or tied up with the solids fraction.

Critical solvent processes affect separation by the greatly enhanced dissolving power of the solvent in the range of pressure and temperature near the critical values for the solvent. Two processes that apply this property are described in U.S. Pat. Nos. 3,607,716 and 3,607,717. By proper choice of solvent, pressure and temperature, such a process can effectively produce separate process streams enriched in solids, asphaltenes, pre-asphaltenes, and oils. After recovery of the critical solvent by evaporation, the

solids phase typically still comprise 35 to 40% of the liquid product.

The asphaltenes and pre-asphaltenes are considered to be non-distillable in that they "crack" into gaseous and liquid hydrocarbons and coke upon heating, with a poor liquid recovery. If the asphaltenes and pre-asphaltenes are separated with the insoluble solids from the oil by distillation, anti-solvent deashing or critical solvent deashing, subsequent recovery of the asphaltenes and pre-asphaltenes as liquid product becomes as best marginal. For low rank coals, these processes provide a low liquid yield.

According to the process of the present invention, mixtures of liquid hydrocarbons and insoluble solids are contacted with a volatile solvent compatible with the oils, asphaltenes and pre-asphaltenes for solubilizing said oils, asphaltenes and pre-asphaltenes as opposed to the above prior art processes in which the solvent functions as anti-solvent or a critical solvent. The said liquid hydrocarbons and solids are contacted with the volatile solvent in stages to form a carrier solution. The carrier solution is displaced by the volatile solvent, preferably by a countercurrent or crosscurrent contacting mode, to produce a slurry of insoluble solids with volatile solvent substantially free of the said liquid hydrocarbons to permit a separation and removal of said insoluble solids by gravity settling, preferably under centrifugal forces, such that a minimum of interstitial liquid containing a minor amount of the liquid hydrocarbons is discharged with the insoluble solids. The interstitial liquid, composed largely of the volatile solvent, is substantially recovered from the solids by evaporation.

The liquid hydrocarbons, including substantially all the asphaltenes and pre-asphaltenes, are thus effectively separated from the solids and can be in turn separated from the volatile solvent for conventional processing. High losses of the asphaltenes and pre-asphaltenes inherent in known processes, particularly for low rank coals such as lignite coals, are avoided.

In its broad aspect, the process of the present invention for separating insoluble solids from liquid hydrocarbons containing oils, asphaltenes and pre-asphaltenes comprises the steps of: contacting the liquid hydrocarbons with a volatile solvent compatible with the oils, asphaltenes and pre-asphaltenes to solubilize the said oils, asphaltenes and pre-asphaltenes to form a carrier solution; separating insoluble solids from the carrier solution by gravity separation and displacing said carrier solution from the solids by volatile solvent whereby said insoluble solids are discharged with interstitial volatile solvent; recovering said volatile solvent from the residual

solids; and recovering oils, asphaltenes and pre-asphaltenes substantially free of insoluble solids.

Gravity separation is applied, preferably by centrifugal forces which accelerate the rate of separation, utilizing the density differences between the insoluble solids and liquid phase. The volatile solvent is contacted with the liquid hydrocarbons in an amount in the range of about 10 to about 250% by weight of the liquid hydrocarbons preferably in countercurrent or cross-current stages applying centrifugal forces to each stage whereby the final insoluble solids residue is compacted with a minimum of interstitial liquid, said final interstitial liquid comprised largely of the volatile solvent for ease of recovery.

Coal liquifaction products are particularly suited for the application of the process of the invention with the use of a coal extract volatile solvent, said volatile solvent normally being recovered for recycle. The coal liquefaction products are processed for the asphaltene, pre-asphaltene and oils recovery and recycle of the volatile solvent.

The accompanying drawing is a simplified schematic flow diagram of the process of the invention applied to the processing of coal liquefaction products, it being understood that the scope of the invention is not to be limited thereby.

Referring now to the drawing, reactor products from coal liquefaction are mixed with a compatible volatile coal extract solvent introduced by line 52 either in pre-mixer 10 or directly in separator 14. Contact of the reactor products with the solvent is accomplished, preferably in a series of multiple-stage countercurrent or crosscurrent mixers with the application of gravity separation such as by the use of centrifuges at each stage, such that the solids residue in the final mixing and separating stage is contacted with fresh volatile solvent for discharge of compacted solids residue therefrom containing interstitially volatile solvent essentially free of coal liquefaction products. The use of pre-mixer 10 assists in the solubilizing of the asphaltenes and pre-asphaltenes by the volatile solvent. The multiple-stage contacting can be effected in a single device having multiple internal stages.

The volatile coal extract solvent is recovered from subsequent processing to be described and is compatible with the oils, asphaltenes and preasphaltenes. The volatile solvent is prepared from a coal derived oil fraction having at least 80% by volume distillation temperature between about 205° and 535°C for compatibility with the coal liquefaction products. A typical volatile solvent, shown in Table 1, comprises by volume about 98.3% distillation temperature between about 205° to 515°C.

60

65

3

TABLE 1
Coal extract solvent

Density (g/cm³) 0.98 at 217°C
Viscosity (cp) 23.50 at 20°C

Elemental analysis	Wt. (%)
C	89.0
H	9.5
N	1.0
S	0.0
O	0.5
Atomic H/C Ratio	1.28
Distillation characteristics	
Temp. Range (°C)	Vol. (%)
—205	1.2
205—275	21.3
275—350	37.5
350—450	31.5
450—515	8.0
515+ (Residue)	0.5

The volatile coal extract solvent is contacted and mixed with the reactor products in an amount by weight in the range of about 10 to about 250%, preferably about 20 to about 100%, of the coal liquefaction slurry product. The quantity employed will vary according to the particular volatile solvent used and the characteristics of the reactor products which are determined by the coal starting material and the manner of liquefaction. Separator 14 is maintained at a temperature in the range of about 50° to 350°C under a pressure within the range of sub-atmospheric pressure to about 3.5 MPa.

Separation 14 is effected by gravity separation, in a conventional gravity settling vessel or in a centrifuge with the application of multiplied settling forces, for separation primarily according to differences in densities between the homogeneous carrier solution comprised of solvent and liquefaction products and the insoluble solids. The carrier solution is recovered as an overflow substantially free of solids and the solids recovered as an underflow, the amount of underflow preferably being kept to a minimum such as by the use of centrifugal forces to compact the solids and to minimize the volume of interstitial carrier solution at each stage and to minimize the amount of volatile solvent escaping with the solids at the last stage.

The underflow containing solids with interstitial carrier solution, mainly volatile solvent, is withdrawn through line 16 and fed to recovery unit 18, which may constitute part of separator 14 or consist of a separate vessel in which the volatile solvent is evaporated at a temperature within the solvent boiling range. The evaporated solvent and any contained liquefaction product are fed by lines 22, 25 to series condensers 24, 26 with condensed product recycled to separator 40, to be described, by lines 28, 30, or discharged by line 32 as product.

The solids, substantially free of solvent, are withdrawn from unit 18 as dried, friable, non-sticking solids which may be crushed and conveyed by line 20 to a gasifier or burner. Separation of oil, asphaltenes and preasphaltenes in separator 14 from the solids is substantially complete due to the effective separation of the liquefaction products solubilized in the carrier solution and displaced by the volatile solvent, substantially eliminating loss of coal liquefaction product with solids in line 20.

The overflow of carrier solution from separator 14 is fed through line 36 to separator 40 and mixed with a coal derived light oil which is incompatible with the asphaltene and pre-asphaltene materials. The carrier solution and said light oil, such as light naphtha, are processed in separator 40 at a temperature in the range of about 50° to about 150°C at a pressure of from atmospheric pressure to about 3.5 MPa, the light oil being introduced in an amount by weight in the range of about 30% to about 100% of the carrier solution. The addition of the incompatible light oil precipitates a substantial part of the asphaltenes and pre-asphaltenes in the form of an immiscible liquid and/or solid phase having a greater density than the density of the carrier solution from which they are precipitated.

The immiscible phases can be separated from each other by gravity settling, preferably under centrifugal forces, to produce a non-viscous liquid overflow and a sticky semi-solid underflow comprised mainly of asphaltenes and pre-asphaltenes. The underflow is withdrawn by line 42 and is: returned to the liquefaction reactor, not shown, for further conversion into lighter oils; discharged for use as a solids product with a low ash content; or upgraded such as by hydrocracking into distillable oils.

The liquid overflow from separator 40 is fed by line 44 to recovery unit 46 for stripping and recovery of the light oil fraction by flash evaporation and fractionation, or by distillation, for recycle by line 54 to separator 40. The bottoms are withdrawn by line 48 and discharged as product through line 50 or recycled by line 52 to separator 14 or pre-mix vessel 10. The bottoms of vessel 46 may be passed through a hydroprocessor 49 to convert remaining asphaltene and pre-asphaltene fractions to distilled and to increase the hydrogen concentration, i.e. to regenerate the volatile solvent. Replacement of light oil taken from the system by removal of the

two product streams 42, 50 can be made up by coal extract oil from coal liquefaction through line 38.

The overflow of carrier solution from separator 14 may be directly fed to alternative processing unit 58 instead of to separator 40. The unit depicted by numeral 58 may be a hydrocracker from which the liquids are subsequently separated in a distillation column into products, recycle oil for the slurring of coal, and recycle volatile coal extract solvent compatible with the asphaltenes and pre-asphaltenes; a distillation column for separation of overflow by boiling range; or a solvent deasphalting process such as a propane deasphalting process or Duosol process in which the asphaltenes and pre-asphaltenes are separated from the solvent.

The process of the invention was carried out for the processing of reactor product resulting from the direct liquefaction of lignite in separator 14

and recovery unit 18. Separator 14 was a batch centrifuge operated at 1500 G's at atmospheric pressure with carrier solution maintained at 150°C. The reactor liquid product consisted of 86.49 mass units of liquid hydrocarbons and 13.51 mass units of unreacted coal and ash. Contacting was carried out in a three-stage crosscurrent mode using a total of 205.34 mass units of volatile coal extract solvent. The last underflow was fed to a vacuum flask for evaporation of the volatile solvent from the residual solids. Table 2 indicates the distribution of components in the feed to the separator, combined separator overflow and final underflow and recovery unit overflow and bottoms. For a feed to the separator of 86.49 mass units of reactor liquid product, 1.84 units of reactor liquid product were lost with the insoluble solids in the recovery unit bottoms, resulting in a recovery of 97.9% of the reactor liquid product.

TABLE 2

Constituent	Feed to separator	Separator overflow	Separator underflow (feed to recovery unit)	Recovery unit (overflow)	Recovery unit bottoms
Oil	65.67	65.14	0.53	—	0.53
Asphaltenes	14.85	14.41	0.44	—	0.44
Pre-asphaltenes	5.97	5.10	0.87	—	0.87
	<u>86.49</u>	<u>84.65</u>			<u>1.84</u>
Insoluble solids	13.51	0.00	13.51	—	13.51
Volatile solvent	205.34	180.07	25.27	25.27	

Claims

1. A process for separating insoluble solids from liquid hydrocarbons containing oils, asphaltenes, pre-asphaltenes, comprising the steps of:

contacting the liquid hydrocarbons with a volatile solvent compatible with the liquid hydrocarbons to solubilize the liquid hydrocarbons and to form a carrier solution;

separating insoluble solids from the carrier solution by gravity separation and displacing said carrier solution from the solids by volatile solvent whereby said insoluble solids are discharged with interstitial volatile solvent;

recovering said volatile solvent from the insoluble solids; and

recovering said liquid hydrocarbons substantially free of insoluble solids.

2. A process for separating insoluble solids from liquid hydrocarbons containing oils, asphaltenes and pre-asphaltenes comprising the steps of:

contacting and mixing the liquid hydrocarbons and insoluble solids with a volatile solvent recoverable by evaporation which is soluble with the said oils, asphaltenes and pre-asphaltenes throughout all the process steps;

separating the insoluble solids from the solution by gravity separation and displacement of the asphaltenes and pre-asphaltenes from the solids by forming a compacted solids phase with minimal residual liquid hydrocarbons in the interstitial spaces;

recovering oils, asphaltenes and pre-asphaltenes substantially free of insoluble solids in a clarified liquid phase;

extracting residual asphaltenes and pre-asphaltenes in the compacted solids phase by contacting and mixing said compacted solids phase with a volatile solvent and separating the insoluble solids therefrom by gravity separation by forming a compacted solids phase with minimal residual liquid hydrocarbons in the interstitial spaces until said interstitial liquid hydrocarbon mixture in a final gravity separation is

substantially free of said asphaltenes and pre-asphaltenes;

and recovering said residual interstitial liquid hydrocarbon mixture from the insoluble solids from the final separation by evaporation.

3. A process according to Claim 2 in which said insoluble solids are discharged with interstitial solvent substantially free of said asphaltenes and pre-asphaltenes.

4. A process according to Claim 2 or 3 in which volatile solvent is recovered from said oils, asphaltenes and pre-asphaltenes.

5. A process according to Claim 1 or 4 in which said liquid hydrocarbons are products of coal liquefaction and said volatile solvent is a coal extract.

6. A process according to any preceding claim applying said gravity separation in a centrifuge whereby said insoluble solids are compacted to minimize the volume of interstitial carrier solution or volatile solvent.

7. A process according to Claim 4, 5 or 6 in which said volatile solvent is added in an amount by weight in the range of about 10 to 250% of the liquid hydrocarbons.

8. A process according to any of Claims 1 to 5 in which the liquid hydrocarbons are contacted with the volatile solvent in a pre-mixer prior to the gravity separation for a time sufficient to effect substantial solubilization of the liquid hydrocarbons.

9. A process according to any of Claims 1 to 5 in which the liquid hydrocarbons are contacted with the volatile solvent in a single stage or a multi-stage cross-current or countercurrent system.

10. A process according to any of Claims 1 to 5 in which the gravity separation is effected under centrifugal forces sequentially with addition and mixing of the volatile solvent with the liquid hydrocarbons, said volatile solvent being added in an amount by weight in the range of 10 to 250% by weight of the liquid hydrocarbons crosscurrent or countercurrent to the flow of liquid hydrocarbons whereby the insoluble solids in a final mixing and separating stage is contacted with fresh volatile solvent for discharge of compacted insoluble solids containing interstitially volatile solvent essentially free of said liquid hydrocarbon.

11. A process according to any of Claims 1 to 5 in which the gravity separation is conducted at a temperature in the range of about 50° to 350°C at a pressure in the range of sub-atmospheric pressure to about 3.5 MPa.

12. A process according to Claim 4, 5 or 6 in which at least 80% by volume of the volatile solvent has a distillation temperature in the range of 205° to 535° C.

13. A process according to any of Claims 1 to 12 in which the substantially solids-free carrier solution recovered from the gravity separation is fed to one of a hydrocracker from which the liquids are separated in a distillation column into products, recycle oil for slurring of coal and recycle solvent compatible with the asphaltenes

and pre-asphaltenes; a distillation column for separation of said overflow by boiling range; or a solvent deasphalting process in which the asphaltenes and pre-asphaltenes are separated from the solvent.

14. A process according to any of Claims 1 to 5 in which the substantially solids-free carrier solution recovered from the gravity separation is contacted with a light oil incompatible with the asphaltenes and pre-asphaltenes in an amount sufficient to precipitate a portion of said asphaltenes and pre-asphaltenes, separating said precipitated asphaltenes and pre-asphaltenes from the liquid phase, and recovering the light oil for recycle to the substantially solids-free carrier solution.

15. A process according to any of the preceding claims in which the recovered asphaltenes and pre-asphaltenes substantially free of insoluble solids are converted by hydrocracking to distillates.

16. A process according to any of the preceding claims in which the liquid hydrocarbons are at least one of:

products derived from liquefaction of a mixture of coal-derived liquids plus coal; products derived from the liquefaction of non coal-derived liquids plus coal; products derived from hydroprocessing of a mixture of coal-derived liquids; products derived from the hydroprocessing of non coal-derived liquids; said products derived with a catalyst; and combinations thereof.

17. A process according to any of Claims 1 to 5 in which part of the carrier solution from which the light oil and most of the asphaltenes and pre-asphaltenes have been separated is fed to a hydroprocessor to regenerate the volatile solvent.

Patentansprüche

1. Verfahren zur Abtrennung unlöslicher Feststoffe aus Öle, Asphaltene und Pre-Asphaltene enthaltenden flüssigen Kohlenwasserstoffen, umfassend die folgenden Schritte:

In-Berührung-Bringen der flüssigen Kohlenwasserstoffe mit einem flüchtigen, mit den flüssigen Kohlenwasserstoffen verträglichen Lösungsmittel, um die flüssigen Kohlenwasserstoffe löslich zu machen und eine Träger-Lösung zu bilden;

Abtrennen der unlöslichen Feststoffe von der Träger-Lösung durch Schwerentrennung und Verdrängen der Träger-Lösung von den Feststoffen durch flüchtiges Lösungsmittel, wodurch die unlöslichen Feststoffe mit eingelagertem flüchtigem Lösungsmittel ausgetragen werden;

Rückgewinnen des flüchtigen Lösungsmittels von den unlöslichen Feststoffen; und

Gewinnen der von unlöslichen Feststoffen im wesentlichen freien flüssigen Kohlenwasserstoffe.

2. Verfahren zur Abtrennung unlöslicher Feststoffe aus Öle, Asphaltene und Asphaltene-Vorstufen (Pre-Asphaltene) enthaltenden flüssigen

Kohlenwasserstoffen, umfassend die folgenden Schritte:

In-Berührung-Bringen und Vermischen der flüssigen Kohlenwasserstoffe und unlöslichen Feststoffe mit einem durch Abdampfen rückgewinnbaren flüchtigen Lösungsmittel. ssigen Kohlenwasserstoff-Gemisch, das mit den Ölen, Asphaltene und Pre-Asphaltene in sämtlichen Verfahrensschritten löslich ist;

Abtrennen der unlöslichen Feststoffe von der Lösung durch Schwereretrennung und Verdrängen der Asphaltene und Pre-Asphaltene von den Feststoffen durch Bildung einer verdichteten Feststoff-Phase, in der flüssige Kohlenwasserstoff-Reste in den Zwischenräumen nur in minimaler Menge zurückbleiben;

Gewinnen der von unlöslichen Feststoffen im wesentlichen freien Öle, Asphaltene und Pre-Asphaltene in einer geklärten flüssigen Phase;

Extrahieren der restlichen Asphaltene und Pre-Asphaltene in der verdichteten Feststoff-Phase durch In-Berührung-Bringen und Vermischen der verdichteten Feststoff-Phase mit einem flüchtigen Lösungsmittel und Abtrennen der unlöslichen Feststoffe daraus durch Schwereretrennung unter Bildung einer verdichteten Feststoff-Phase, in der flüssige Kohlenwasserstoff-Reste in den Zwischenräumen nur in minimaler Menge zurückbleiben, bis das flüssige Kohlenwasserstoff-Gemisch in den Zwischenräumen in einer letzten Schwereretrennung im wesentlichen frei von Asphaltene und Pre-Asphaltene ist; und Rückgewinnen des flüssigen Kohlenwasserstoff - Gemischs aus den Zwischenräumen der unlöslichen Feststoffe der letzten Trennung durch Abdampfen.

3. Verfahren nach Anspruch 2, dadurch gekennzeichnet, daß die unlöslichen Feststoffe mit in den Zwischenräumen befindlichem Lösungsmittel im wesentlichen frei von den Asphaltene und Pre-Asphaltene ausgetragen werden.

4. Verfahren nach Anspruch 2 oder 3, dadurch gekennzeichnet, daß das flüchtige Lösungsmittel von den Ölen, Asphaltene und Pre-Asphaltene zurückgewonnen wird.

5. Verfahren nach Anspruch 1 oder 4, dadurch gekennzeichnet, daß die flüssigen Kohlenwasserstoffe Produkte der Kohleverflüssigung sind und das flüchtige Lösungsmittel ein Kohle-Extrakt ist.

6. Verfahren nach irgendeinem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß die Schwereretrennung in einer Zentrifuge durchgeführt wird, wodurch die unlöslichen Feststoffe verdichtet werden, um das Volumen der Träger-Lösung oder des flüchtigen Lösungsmittels in den Zwischenräumen auf ein Minimum zu verkleinern.

7. Verfahren nach Anspruch 4, 5 oder 6, dadurch gekennzeichnet, daß das flüchtige Lösungsmittel in einer Menge im Bereich von 10 bis 250 Gew.%, bezogen auf die flüssigen Kohlenwasserstoffe, zugesetzt wird.

8. Verfahren nach irgendeinem der Ansprüche 1 bis 5, dadurch gekennzeichnet, daß die flüssigen Kohlenwasserstoffe mit dem flüchtigen Lösungs-

mittel in einem Vormischer vor der Schwereretrennung eine Zeitspanne in Berührung gebracht werden, die ausreicht, um die flüssigen Kohlenwasserstoffe im wesentlichen löslich zu machen.

9. Verfahren nach irgendeinem der Ansprüche 1 bis 5, dadurch gekennzeichnet, daß die flüssigen Kohlenwasserstoffe mit dem flüchtigen Lösungsmittel in einem einstufigen oder mehrstufigen Kreuzstrom- oder Gegenstrom-System in Berührung gebracht werden.

10. Verfahren nach irgendeinem der Ansprüche 1 bis 5, dadurch gekennzeichnet, daß die Schwereretrennung unter Zentrifugalkräften sequentiell mit Zugabe und Vermischen des flüchtigen Lösungsmittels mit den flüssigen Kohlenwasserstoffen durchgeführt wird, wobei das flüchtige Lösungsmittel in einer Menge im Bereich von 10 bis 250 Gew.%, bezogen auf die flüssigen Kohlenwasserstoffe, im Kreuzstrom oder Gegenstrom zu dem Strom der flüssigen Kohlenwasserstoffe, zugesetzt wird, wodurch die unlöslichen Feststoffe in einer letzten Stufe des Mischens und Abtrennens mit frischem Lösungsmittel in Berührung gebracht werden, wodurch verdichtete unlösliche Feststoffe ausgetragen werden, die in den Zwischenräumen von flüssigen Kohlenwasserstoffen im wesentlichen freies flüchtiges Lösungsmittel enthalten.

11. Verfahren nach irgendeinem der Ansprüche 1 bis 5, dadurch gekennzeichnet, daß die Schwereretrennung bei einer Temperatur im Bereich von etwa 50°C bis 350°C unter einem Druck im Bereich von unterhalb des Atmosphärendrucks bis etwa 3,5 MPa durchgeführt wird.

12. Verfahren nach Anspruch 4, 5 oder 6, dadurch gekennzeichnet, daß wenigstens 80 Vol.-% des flüchtigen Lösungsmittels eine Destillationstemperatur im Bereich von 205°C bis 535°C aufweist.

13. Verfahren nach irgendeinem der Ansprüche 1 bis 12, dadurch gekennzeichnet, daß die bei der Schwereretrennung gewonnene, im wesentlichen feststofffreie Träger-Lösung eingeleitet wird in eine der folgenden Verfahrensstufen:

einen Hydrocracker, aus dem die Flüssigkeiten in einer Destillationskolonne in Produkte, Rücklauf-Öl zum Aufschlännen der Kohle und mit den Asphaltene und Pre-Asphaltene verträgliches Rücklauf-Lösungsmittel aufgetrennt werden;

eine Destillationskolonne zum Trennen des Überlaufs mittels des Siedebereichs; oder

ein Lösungsmittel-Entasphaltierungs-Verfahren, in dem die Asphaltene und Pre-Asphaltene von dem Lösungsmittel getrennt werden.

14. Verfahren nach irgendeinem der Ansprüche 1 bis 5, dadurch gekennzeichnet, daß die bei der Schwereretrennung gewonnene, im wesentlichen feststofffreie Träger-Lösung mit einem mit den Asphaltene und Pre-Asphaltene unverträglichen Leichtöl in solcher Menge in Berührung gebracht wird, die zur Asfällung eines Teils der Asphaltene und Pre-Asphaltene ausreicht, wobei die abgeschiedenen Asphaltene und Pre-Asphaltene von der flüssigen Phase abgetrennt

werden und das Leichtöl zur Rückführung zu der im wesentlichen feststofffreien Träger-Lösung zurückgewonnen wird.

15. Verfahren nach irgendeinem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß die gewonnenen Asphaltene und Pre-Asphaltene, die im wesentlichen frei von Feststoffen sind, durch Hydrocracken in Destillate umgewandelt werden.

16. Verfahren nach irgendeinem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß die flüssigen Kohlenwasserstoffe aus wenigstens einem der folgenden Materialien bestehen:

Produkte, die aus der Verflüssigung einer Mischung aus von Kohle herrührenden Flüssigkeiten plus Kohle stammen;

Produkte, die aus der Verflüssigung einer Mischung aus nicht von Kohle herrührenden Flüssigkeiten plus Kohle stammen;

Produkte, die aus der Hydrierverarbeitung eines Gemischs von Kohle herrührender Flüssigkeiten stammen;

Produkte, die aus der Hydrierverarbeitung nicht von Kohle herrührender Flüssigkeiten stammen; der genannten Produkte, die mit Hilfe von Katalysatoren erhalten wurden; und Kombinationen der vorstehenden.

17. Verfahren nach irgendeinem der Ansprüche 1 bis 5, dadurch gekennzeichnet, daß ein Teil Träger-Lösung, aus der das Leichtöl und der größte Teil der Asphaltene und Pre-asphaltene abgetrennt worden sind, zur Rückgewinnung des flüchtigen Lösungsmittels einer Anlage für die Hydrierverarbeitung zugeführt werden.

Revendications

1. Procédé pour la séparation de matières solides insolubles à partir d'hydrocarbures liquides, contenant des huiles, des asphaltènes et des pré-asphaltènes, comprenant les étapes suivantes:

mise en contact des hydrocarbures liquides avec un solvant volatil compatible avec les hydrocarbures pour solubiliser les hydrocarbures liquides et former une solution mère;

séparation des matières solides insolubles à partir de la solution mère par décantation et déplacement de ladite solution mère des matières solides par addition de solvant volatil, lesdites matières solides insolubles étant transférées avec le solvant volatil interstitiel;

récupération dudit solvant volatil à partir des matières solides insolubles; et

récupération desdits hydrocarbures liquides pratiquement exempts de matière solide insoluble.

2. Procédé pour la séparation de matières solides insolubles à partir d'hydrocarbures liquides contenant des huiles, des asphaltènes et des pré-asphaltènes, caractérisé ce qu'il comprend les étapes suivantes:

mise en contact et mélange des hydrocarbures liquides et des matières solides insolubles avec

un mélange d'hydrocarbures liquides prétraité récupérable par évaporation, lequel est soluble avec lesdites huiles, asphaltènes et pré-asphaltènes dans toutes les étapes du procédé;

5 séparation des matières solides insolubles à partir de la solution par décantation et déplacement des asphaltènes et pré-asphaltènes des matières solubles en formant une phase solide compacte avec le minimum d'hydrocarbures liquides résiduels dans les espaces interstitiels;

10 récupération des huiles, asphaltènes et pré-asphaltènes pratiquement exempts de matière solide insoluble en une phase liquide transparente;

15 extraction des asphaltènes et pré-asphaltènes résiduels dans la phase solide compacte par contact et mélange de ladite phase solide compacte avec un solvant volatil et séparation des matières solides insolubles obtenues par

20 décantation en formant une phase solide compacte avec le minimum d'hydrocarbures liquides résiduels dans les espaces interstitiels jusqu'à ce que ledit mélange d'hydrocarbures liquides interstitiels dans la décantation final soit

25 pratiquement exempt desdits asphaltènes et pré-asphaltènes; et

récupération dudit mélange d'hydrocarbures liquides interstitiels résiduels à partir de matières solides insolubles de la séparation finale par

30 évaporation.

3. Procédé selon la revendication 2, dans lequel lesdites matières solides insolubles sont transférées avec le solvant interstitiel pratiquement exempt desdits asphaltènes et pré-asphaltènes.

35 4. Procédé selon l'une des revendications 2 ou 3, dans lequel le solvant volatil est récupéré à partir desdites huiles, asphaltènes et pré-asphaltènes.

40 5. Procédé selon l'une des revendications 1 à 4, dans lequel lesdits hydrocarbures liquides sont des produits de la liquéfaction du charbon et ledit solvant volatil est un extrait de charbon.

45 6. Procédé selon l'une quelconque des revendications précédentes appliquant ladite décantation dans une centrifugeuse où lesdites matières solides insolubles sont comprimées pour réduire au minimum le volume de solution mère interstitielle ou de solvant volatil.

50 7. Procédé selon les revendications 4, 5 ou 6, dans lequel ledit solvant volatil est ajouté en une quantité en poids de 10 à 250% des hydrocarbures liquides.

55 8. Procédé selon l'une quelconque des revendications 1 à 5, dans lequel les hydrocarbures liquides sont mis en contact avec le solvant volatil dans un prémélangeur avant la décantation et pendant une durée suffisante pour réaliser une solubilisation importante des hydrocarbures liquides.

60 9. Procédé selon l'une quelconque des revendications 1 à 5, dans lequel les hydrocarbures liquides sont mis en contact avec le solvant volatil dans un dispositif à un seul étage ou à plusieurs étages, à courant croisé ou à contre-courant.

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10. Procédé selon l'une quelconque des revendications 1 à 5, dans lequel la décantation est mise en oeuvre sous l'action de forces centrifuges de façon séquentielle avec addition et mélange du solvant volatil et des hydrocarbures liquides, ledit solvant volatil étant ajouté en une quantité en poids dans l'intervalle de 10 à 250% hydrocarbures liquides, suivant un courant croisé ou à contre-courant du courant des hydrocarbures liquides où les matières solides insolubles dans un mélange final et à l'étage de séparation sont mises en contact avec le solvant volatil frais pour transfert des matières solides insolubles compactes contenant le solvant volatil interstitiel pratiquement exempt dudit hydrocarbure liquide.

11. Procédé selon l'une quelconque des revendications 1 à 5, dans lequel la décantation est mise en oeuvre à une température d'environ 50 à 350°C, à une pression comprise entre la pression subatmosphérique et environ 3,5 MPa.

12. Procédé selon la revendication 4, 5 ou 6, dans lequel au moins 80% en volume du solvant volatil a une température de distillation allant de 205 à 535°C.

13. Procédé selon l'une quelconque des revendications 1 à 12, dans lequel la solution mère pratiquement exempte de matières solides récupérée à partir de la décantation est introduite dans un hydrocraqueur à la sortie duquel les liquides sont séparés dans une colonne de distillation en produits, huile recyclée pour délayer le charbon et solvant recyclé compatible avec les asphaltènes et pré-asphaltènes; une colonne de distillation pour la séparation dudit surnageant par gamme d'ébullition; ou un procédé de déasphaltage par solvant dans lequel des asphaltènes et pré-asphaltènes sont séparés du solvant.

14. Procédé selon l'une quelconque des revendications 1 à 5, dans lequel la solution mère pratiquement exempte de matières solides récupérée à partir de la décantation est mise en contact avec une huile légère incompatible avec les asphaltènes et pré-asphaltènes en quantité suffisante pour précipiter une fraction desdits asphaltènes et pré-asphaltènes, avec séparation desdits asphaltènes et pré-asphaltènes précipités à partir de la phase liquide, et récupération de l'huile légère pour recyclage vers la solution mère pratiquement exempt de matières solides.

15. Procédé selon l'une des revendications précédentes, dans lequel les asphaltènes et pré-asphaltènes récupérés, pratiquement exempts de matières solides insolubles sont transformés par hydrocraquage en distillats.

16. Procédé selon l'une quelconque des revendications précédentes, dans lequel les hydrocarbures liquides sont au moins choisis parmi:

les produits dérivés de la liquéfaction d'un mélange de liquides dérivés du charbon plus du charbon; les produits dérivés de la liquéfaction de liquides ne dérivant pas du charbon plus du charbon; les produits dérivés du traitement hydrothermique d'un mélange de liquides dérivés du charbon; les produits dérivés du traitement hydrothermique de liquides ne dérivant pas du charbon; lesdits produits dérivés avec un catalyseur; et leurs combinaisons.

17. Procédé selon l'une des revendications 1 à 5, dans lequel une partie de la solution mère dont on a séparé l'huile légère et la majeure partie des asphaltènes et pré-asphaltènes, est introduite dans un hydro-processeur pour régénérer le solvant volatil.

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