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Claims 16-37 are deemed to be abandoned due to non-payment of the claims fees (Rule 45(3) EPC).

(54) **METHOD FOR PROCESSING HEAVY PETROLEUM FEEDSTOCK, SYSTEM FOR PROCESSING HEAVY PETROLEUM FEEDSTOCK, CONCENTRATED RESIDUE AND USE OF THE CONCENTRATED RESIDUE**

(57) The invention relates to the field of petroleum processing, in particular to a method for processing heavy petroleum feedstock allowing the production of valuable products from heavy residues, which are typically refractory products, wherein the method is characterized by greater stability and efficiency. The method for processing heavy petroleum feedstock comprises the steps of suspending solid granular material in the feedstock and subjecting the resulting slurry to hydrocracking in the presence of hydrogen in a slurry-phase hydrocracking reactor to obtain a heavy residue stream, wherein the heavy residue stream is a slurry of an un-

converted hydrocracking residue and a spent solid granular material; separating the spent solid granular material from the unconverted hydrocracking residue using a solvent in a washing section to obtain a mixture of the unconverted hydrocracking residue and the solvent; delivering the mixture to a vacuum column for separating the solvent to obtain a separated heavy residue; evaporating at least a portion of the separated heavy residue in an evaporator to obtain a concentrated hydrocracking residue and heavy vacuum gas oil (HVGO), wherein at least a portion of the HVGO is used to produce the solvent.

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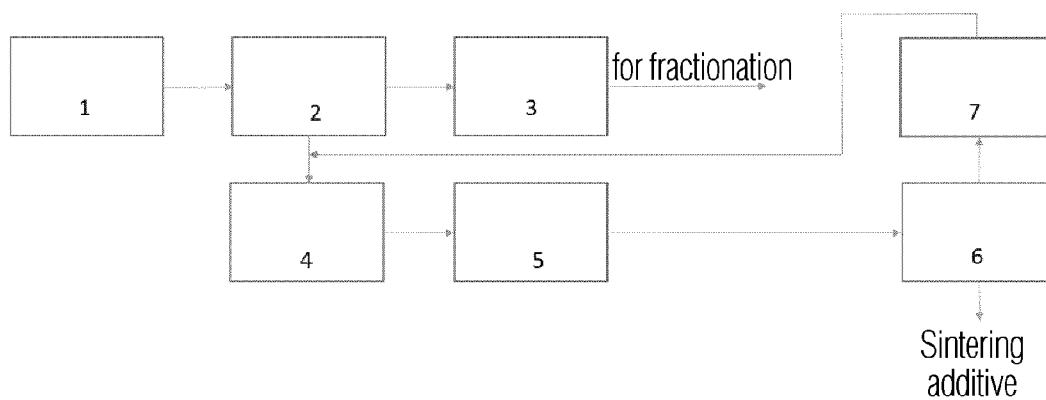


FIG. 1

Description

TECHNICAL FIELD

5 **[0001]** The invention relates to the field of petroleum processing, in particular to a method for processing heavy petroleum feedstock allowing the production of valuable products from heavy residues, which are typically refractory products, wherein the method is characterized by greater stability and efficiency, particularly, in the processes of thermal cracking and hydrocracking of heavy residues from petroleum processing.

10 BACKGROUND

[0002] In the state of the art there are many known processes for processing heavy hydrocarbons in the presence of special solid additives, adsorbents, and catalysts, for example, VCC, Uniflex, EST, GT-SACT, H-Oil, LC-Fining, etc. Among them, a combined hydrocracking process is most effective for processing heavy petroleum feedstocks such as tar obtained after the fractional distillation of heavy Urals crude oil.

15 **[0003]** However, each of these processes faces problems associated with processing residual hydrocracking products to obtain high-quality, demanded products.

[0004] For a combined hydrocracking process, document CA2157052 contemplates the use of a solidified residue from liquid-phase cracking as a binder added to coal charges for producing metallurgical coke, which residue comprises a carbon additive and has passed steps of separation and subsequent vacuum distillation.

20 **[0005]** However, this process is described for residues from the processing of Arabian light crude oil and is not applicable to residues from the processing of heavy crude oils since the high content of heavy hydrocarbons and asphaltenes in these residues will inevitably lead to coking of equipment and fail to provide necessary sintering properties.

[0006] The problem facing the present invention is the development of an effective and stable method for processing heavy petroleum feedstocks, such as heavy Urals crude oil, which makes it possible to derive valuable products from the residues formed through such processing, especially a sintering additive or bitumen products, and a stream of heavy vacuum gas oil, which can be converted into aromatic light gas oil through any known oil-refining and petrochemical process used to enhance aromatic hydrocarbon content, wherein the aromatic light gas oil can in turn be used in the processing of heavy petroleum feedstock, thereby increasing its efficiency and reducing its resource intensity.

30 SUMMARY OF THE INVENTION

[0007] The present invention relates to a method for processing heavy petroleum feedstock, comprising the steps of: suspending a solid granular material in said feedstock and subjecting the resulting slurry to hydrocracking in the presence of hydrogen in a slurry-phase hydrocracking reactor to obtain a heavy residue stream, wherein the heavy residue stream is a slurry of unconverted hydrocracking residue and spent solid granular material; separating the spent solid granular material from the unconverted hydrocracking residue using a solvent in a washing section to obtain a mixture of the unconverted hydrocracking residue and the solvent; delivering said mixture to a vacuum column for separating the solvent, thus obtaining a separated heavy residue; evaporating at least a portion of the separated heavy residue in an evaporator to produce a concentrated hydrocracking residue and heavy vacuum gas oil (HVGO); wherein at least a portion of the HVGO is used to produce the solvent.

[0008] Preferably, the solid granular material is an adsorbent or a catalyst.

[0009] Preferably, the adsorbent is a carbon material.

45 **[0010]** The slurry-phase hydrocracking additionally further a gaseous mixture of hydrocarbons, which is subjected to gas-phase hydrocracking, followed by fractionation of the hydrocracking products.

[0011] Preferably, at least a portion of the HVGO is subjected to catalytic cracking to produce the solvent.

[0012] Preferably, the HVGO is supplied to catalytic cracking in a mixture with at least one of the following components: straight-run vacuum gas oil, fuel oil from a gas condensate processing unit, and hydrotreated vacuum gas oil.

50 **[0013]** It is preferable that the mixture for catalytic cracking is characterized by the following percentage ratios based on the weight of the mixture:

- 10 to 80 hydrotreated vacuum gas oil and/or fuel oil; and
- 20 to 90 HVGO and, optionally, straight-run vacuum gas oil.

55 **[0014]** Additionally, the method comprises a step of recycling at least a portion of the HVGO in a mixture with the separated heavy residue into the evaporator.

[0015] Preferably, the heavy petroleum feedstock is characterized by an initial boiling point of at least 510°C and a density at 20°C of over 1000 kg/m³, in particular it is tar.

[0016] It is preferable that the concentrated hydrocracking residue has an ash content of not more than 1.0%, preferably not more than 0.6%.

[0017] Preferably, the carbon material consists of two fractions of particles, wherein the average particle size of a coarse fraction is greater than the average particle size of a fine fraction, and the ratio of the weighted average diameter of the coarse fraction particles to the weighted average diameter of the fine fraction particles varies from 2 to 7, wherein the coarse and fine fractions are characterized by different mesopore volumes.

[0018] Preferably, the mesopore volume of the fine fraction according to the Barrett-Joyner-Halenda (BJH) method is not less than 0.07 cm³/g and not more than 0.12 cm³/g, while the mesopore volume according to the BJH method for the coarse fraction is not less than 0.12 cm³/g and not more than 0.2 cm³/g.

[0019] Preferably, the carbon material has a BET specific surface area of not less than 230 m²/g and not more than 1250 m²/g, preferably not less than 250 m²/g and not more than 900 m²/g, most preferably not less than 270 m²/g and not more than 600 m²/g.

[0020] Preferably, the solvent is an aromatic light gas oil from catalytic cracking, comprising at least 80 wt. % of aromatic hydrocarbons having from 8 to 16 carbon atoms.

[0021] It is preferable that the evaporation is performed in a thin-film evaporator.

[0022] Preferably, the thin-film evaporator has a double jacket heated by flue gases.

[0023] The separated heavy residue is preferably fed into the thin-film evaporator using a manifold made in the form of a hollow circular tube having supply holes evenly distributed along the diameter of the tube.

[0024] Preferably, the evaporation is performed from a constant-thickness film, wherein the thickness of the film is not more than 1.5 mm, preferably not more than 1.3 mm, and even more preferably from 1.1 to 1.15.

[0025] Preferably, stream redistributors are provided along the height of the thin-film evaporator, which are circle-shaped metal plates installed along the height of the reactor.

[0026] Preferably, the circulation of a bottom product in the thin-film evaporator with a tangential input.

[0027] The evaporation is preferably performed in the presence of atmospheric oxygen.

[0028] Preferably, the evaporation from the constant-thickness film is carried out for a predetermined time at a temperature and an evaporation pressure which ensure the evaporation of volatile components to a volatile component mass fraction of at most 60% in the concentrated residue and to a ring-and-ball softening point of the concentrated residue of at least 105°C.

[0029] Preferably, HVGO is produced by condensing vapors of the evaporator using a refrigerator, followed by collection of the resulting distillate.

[0030] According to another aspect of the invention, a system is provided for processing heavy petroleum feedstock, comprising a slurry-phase hydrocracking section; a separation section; a washing section for separating spent solid material from a slurry; a vacuum column; and an evaporator.

[0031] Preferably, the slurry-phase hydrocracking section comprises at least one slurry-phase hydrocracking reactor.

[0032] Preferably, the washing section comprises at least one mixing tank and at least one separating tank.

[0033] Preferably, the mixing tank is configured to mix a slurry of the spent solid material in an unconverted hydrocracking residue with a solvent.

[0034] Preferably, a separation vessel is configured to separate the spent solid material from the mixture of the unconverted hydrocracking residue and the solvent, in particular, using centrifugal forces, gravitational forces, or flotation, preferably centrifugal forces.

[0035] Preferably, the vacuum column is configured to separate the mixture of the unconverted hydrocracking residue and the solvent to produce a recovered solvent, vacuum gas oil, and a recovered heavy residue.

[0036] Preferably, the evaporator is configured to concentrate the separated heavy residue by evaporation to produce a concentrated hydrocracking residue and a heavy vacuum gas oil.

[0037] The evaporator is preferably a thin-film evaporator.

[0038] Preferably, the solvent is a product of the processing of heavy vacuum gas oil, wherein the processing is aimed at increasing the content of aromatic hydrocarbons, in particular of catalytic cracking.

[0039] According to another aspect of the invention, a concentrated hydrocracking residue obtained by the method according to the invention is provided, wherein the residue is characterized by an ash content of not more than 1.0% and ring-and-ball softening point of not less than 105°C.

[0040] According to another aspect of the invention, the use of the claimed concentrated residue is provided as a sintering additive in charge to produce a coke, the coke being metallurgical coke, foundry coke, in particular molded coke.

[0041] According to another aspect of the invention, the use of the claimed concentrated residue is provided as a sintering additive in charge to produce carbon electrodes, wherein the carbon electrodes are an anode or cathode for galvanic processes, in particular, for the production of aluminum.

[0042] According to another aspect of the invention, the use of the claimed concentrated residue is provided as a sintering additive in charge to produce self-sintering electrodes.

[0043] According to another aspect of the invention, the use of the claimed concentrated residue is provided to produce

petroleum coke, wherein the petroleum coke may be anode coke.

BRIEF DESCRIPTION OF THE DRAWINGS

5 [0044]

Fig. 1. Flow diagram of the process according to the claimed method.

Fig. 2. Schematic illustration of separation section 2;

Fig. 3. Cross-sectional view of the casing of the thin-film evaporator.

10 Fig. 4. General view of the rotor with installed scrapers in the thin-film evaporator.

Fig. 5. General view of the feedstock distributor in the thin-film evaporator.

Fig. 6. Illustration of the feedstock redistributor.

DETAILED DESCRIPTION OF THE INVENTION

15 [0045] The method and system according to the present invention make it possible to process feedstocks, which are traditionally very difficult to process into useful and marketable products. Suitable feedstock includes tar, bottom products of atmospheric columns, bottom products of vacuum columns, heavy gas oil from catalytic cracking, shale oil, coal oil, crude oil bottoms, stripped crude oil and heavy bituminous crude oil from petroleum-bearing sandstones.

20 [0046] The system includes a slurry-phase hydrocracking (SPH) section 1, a separation section 2, a washing section 4 for separating a solid phase of the slurry, a vacuum column 5, and an evaporator 6. A gas-phase hydrocracking section 3 can be provided.

[0047] In the method for processing heavy petroleum feedstock according to the present invention, as shown in FIG. 1, a solid granular material is mixed with heavy hydrocarbon feedstock to produce a homogeneous slurry. In one aspect, 25 various solid particles can be used as the granular material to inhibit the coking process, such as catalysts or adsorbents, provided that the solid particles are capable of withstanding the hydrocracking process. As a catalyst, particles of iron(II) sulfate, metal naphthenate or metal octanoate may be contemplated, wherein the metal may be molybdenum, tungsten, ruthenium, nickel, cobalt, or iron. As an adsorbent, non-metallized carbon-containing additives, unmodified or modified, can be used. Non-metallized additives can be modified, for example by depositing metals, such as FeOOH and Fe_2O_3 , on 30 its surface to impart catalytic properties to a carbon-containing additive. In another embodiment of the invention, the carbon-containing additive is modified to change its structure, in particular to increase the volume of mesopores in order to increase the probability of asphaltene molecules accessing the internal pores of the carbon-containing additive, which will lead to the removal of asphaltenes, which are precursors of coke formation, from the reaction zone and hydrocracking products, thereby improving the quality of the residual hydrocracking products. In the present invention, mesopores 35 include pores with a diameter of 10 to 200 nm, preferably 20 to 50 nm.

[0048] At step 1 of SPH, hydrocarbons are cracked and saturated in a hydrogen environment, wherein asphaltenes, and along with them metals such as Ni, V, Fe, etc., which are catalytic poisons for gas-phase hydrocracking, are adsorbed on a carbon additive.

[0049] In particular, carbon additives, which adsorb heavy hydrocarbons of the asphaltene series, are effective for tar 40 obtained by vacuum distillation of heavy oil. In a particular case, the additive comprises porous carbon material of two different grain compositions, namely a coarse fraction and a fine fraction, wherein the ratio of the weighted average diameter of the coarse fraction particles to the weighted average diameter of the fine fraction particles varies from 2 to 7. The coarse and fine fractions are preferably characterized by different mesopore volumes. Thus, the mesopore volume of the fine fraction according to the Barrett-Joyner-Halenda (BJH) method is preferably not less than $0.07 \text{ cm}^3/\text{g}$ and not more 45 than $0.12 \text{ cm}^3/\text{g}$, while the mesopore volume of the coarse fraction according to the BJH method is preferably not less than $0.12 \text{ cm}^3/\text{g}$ and not more than $0.2 \text{ cm}^3/\text{g}$. For greater efficiency of the asphaltene adsorption, it is preferable that the carbon material has a BET specific surface area is not less than $230 \text{ m}^2/\text{g}$ and not more than $1250 \text{ m}^2/\text{g}$, preferably not less than $250 \text{ m}^2/\text{g}$ and not more than $900 \text{ m}^2/\text{g}$, most preferably not less than $270 \text{ m}^2/\text{g}$ and not more than $600 \text{ m}^2/\text{g}$. Carbon materials that can be used to produce carbon additives for combined hydrocracking are known in the art. They include, for 50 example, lignite, activated brown coal, and activated long-flame coal. In addition, the carbon additive can be modified with metal salts (Mo, W, Fe, etc.) to enhance the cracking function and by attaching hydrogen to improve the conversion of asphaltenes and heavy hydrocarbons.

[0050] Slurry-phase hydrocracking section 1 comprises at least one slurry-phase hydrocracking reactor. The number of 55 reactors can vary depending on a desired yield. The prepared slurry, together with heavy hydrocarbon feedstock, is heated in a flame heater and fed into a slurry-phase hydrocracking reactor (SPH reactor). Heated hydrogen is supplied to the same reactor. Thus, the feedstock entering the SPH reactor comprises three phases: solid particles, liquid hydrocarbons, hydrogen gas and evaporated hydrocarbons. A solid granular material is used to reduce coke formation by decomposition or adsorption of coke precursors, in particular heavy hydrocarbons such as asphaltenes, carbenes, and carboids. The

solid material is added in an effective amount adjusted to strike a balance between the effectiveness of the solid material in performing its function without a significant increase in the risk of erosive wear. Moreover, these amounts also vary for different types of initial heavy petroleum feedstock and solid materials of different nature and can be adjusted by a skilled person based on known methods and/or experiments. In particular, for tar obtained by vacuum distillation of heavy oil, the carbon additive is usually added in an amount of 1 to 2.5% by weight of the feedstock.

[0051] In one aspect, the method according to the present invention can be carried out in an SPH reactor at a pressure ranging from 18 to 24 MPa. The temperature in the reactor typically ranges from 350 to 600°C, preferably from 400 to 500°C. The rate at which hydrogen is supplied ranges from 674 to 3370 Nm³/m³ of oil product. The slurry-phase hydrocracking section may comprise one or more SPH reactors arranged in parallel or series. In addition, cold hydrogen can be introduced into the reactor to cool (quench) the reactor.

[0052] Preferably, the hydrocracking-subjected stream, when passing through separation section 2, is divided into a heavy residue stream, which is a slurry of the spent solid granular material in the unconverted hydrocracking residue, and a stream of a gaseous hydrocarbon mixture, which is supplied to fractionation with or without preliminary gas-phase hydrocracking. Preferably section 2 shown in Fig. 2 comprises a high-pressure hot separator 21 maintained at a separation temperature of 400 to 470°C and, in one aspect, at an SPH reaction pressure. In the high-pressure hot separator, the effluent from the SPH reactor is separated into a gaseous stream and a liquid stream. The gaseous stream is a product of flash distillation at the temperature and pressure of the high-pressure hot separator and comprises from 35 to 95% by volume, preferably from 70 to 95% by volume of hydrocarbon product from the SPH reactor. Similarly, the liquid stream is a liquid phase at the temperature and pressure of the high-pressure hot separator. The gaseous stream is withdrawn from the top of the high-pressure hot separator, and the liquid stream, including the granular material, is withdrawn from the bottom of the high-pressure hot separator. The liquid stream is delivered to a low-pressure hot separator 22 operating at the same temperature as the high-pressure hot separator, but at a pressure between 690 kPa and 3447 kPa. The upper gaseous fraction can be delivered to further separation, gas-phase hydrocracking, or fractionation, and the liquid fraction, which is a slurry of the solid granular material in the unconverted residue, flows out of the low-pressure hot separator 22 and enters the washing section 4.

[0053] As a result of slurry-phase hydrocracking, from 70 to 95% of hydrocarbons were converted into a gaseous mixture of partially hydrogenated hydrocarbons, which are lighter components of liquid-phase hydrocracking products: H₂S, NH₃, H₂O, C₁, C₂, C₃, C₄, C₅ hydrocarbons, naphtha, diesel fraction, and vacuum gas oil.

[0054] The remainder is a slurry of solid granular material in the unconverted residue, which is a mixture of predominantly high-boiling hydrocarbons with an initial boiling point above 525°C.

[0055] In the embodiment of the invention where a carbon additive is used, it is preferable that the additive has a sufficiently high volume of mesopores, i.e., pores with a size exceeding 10 nm, such as more than 25% of the total pore volume, for more efficient adsorption of asphaltenes. Such pores allow large, heavy hydrocarbon molecules to pass into them and precipitate on the pore surface.

[0056] A developed specific surface area (at least 230 m²/g), especially if it is provided by a large number of mesopores, additionally contributes to a large "liquid-solid" phase boundary at which cracking reactions occur, and, in addition, a more developed surface facilitates the entry of asphaltenes into pores without the risk of "flying out" due to the complex geometry of the pores, i.e., they act as a kind of pore "lock" for asphaltenes.

[0057] However, about 10 wt.% of asphaltenes in the feedstock, as well as carbenes and carboids resulted from side condensation reactions during slurry-phase hydrocracking, remain in the form of a dispersed phase surrounded by a dispersion medium at any type of the used solid granular material, which leads to an imbalance between asphaltenes and, on the one hand, aromatic hydrocarbons which disperse asphaltenes, and, on the other hand, saturated hydrocarbons which promote the precipitation of asphaltenes. As a consequence, such an unconverted residue is aggregatively unstable, which leads to its delamination and the appearance of difficult-to-control deposits in the form of asphaltene sediment. Such deposits negatively affect the operation of equipment, leading to wear, shutdowns and difficulties in cleaning and replacing deposit-prone equipment.

[0058] In this regard, it will be desirable to increase the content of aromatic hydrocarbons in the dispersion medium, thereby preventing the precipitation of the asphaltenes remaining in the unconverted residue.

[0059] In addition, the unconverted residue is a fairly viscous liquid, and its flow can entrain the solid granular material to further processing. Therefore, effective reduction of the viscosity of the unconverted residue is necessary to separate solid granular material from it. Effective reduction of the viscosity means, in this case, the creation of a viscosity and density gradient between the unconverted residue and the solid granular material so that the created gradient facilitates the separation of said solid material. In view of the above, in order to reduce the viscosity, while eliminating delamination, an aromatic solvent free of paraffins, which are natural precipitants of asphaltenes, is suitable.

[0060] The process of separating the spent solid material from the unconverted residue occurs in separation step 4, in which in the washing section, the slurry is mixed with a solvent to wash the solid material and separate it from the unconverted residue.

[0061] The washing section is preferably a paired section consisting of a mixing tank and a separation tank. The number

of paired sections can vary depending on a desired productivity and a required efficiency of the separation of spent additive. In the mixing tank, the slurry of the spent solid material in the unconverted hydrocracking residue is mixed with a solvent. In the mixing tank, the slurry of the unconverted high-boiling residue together with the spent solid additive is mixed at a flow rate of 15-20 tons/h at a temperature of about 400-450°C and a pressure of 0.15 to 0.35 MPa(g) with a solvent supplied at a flow rate of 30-35 tons/hour and a temperature of about 220-260°C. In the separation vessel, for example, equipped with a cyclone unit, a decanter, or a flotation apparatus, the spent solid granular material is separated from the unconverted residue in the mixture with the solvent, for example, using centrifugal forces, gravitational forces, or flotation.

[0062] In the separation tank, which in particular is a vertical cylindrical apparatus with a conical bottom, the spent solid additive is separated from the mixture of the unconverted high-boiling residue with the solvent due to the hydrocyclone operation. A gas cushion is provided at the top of the separation tank to control the level of fluid in the tank and to regulate drainage from the tank. The spent solid additive, if necessary, can be pushed further downstream by increasing the pressure of the gas cushion, thus reducing the risk of equipment clogging.

[0063] The hydrocyclone generally consists of a short cylindrical (upper) part with a pipe for tangential input of the mixture (tangentially to the surface of the cylinder) and a conical (lower) part with a hole at the top of the cone to discharge solid fractions.

[0064] Tangential input of the mixture and axial discharge of the separation products lead to the rotation of the mixture, its axial and radial movement from the walls of the apparatus to the drain and discharge holes. The rotating flow in the hydrocyclone has several zones: external (wall) - downward; internal - ascending; middle - circulation, occupying the main volume of the hydrocyclone. Heavy and large solid particles coming with the initial pulp are thrown by centrifugal force onto the inner surface of the cylinder and are carried down by the rotating flow. Under the action of the radial component of the flow (from the walls to the center) and the turbulent nature of its movement, light and small grains are carried into the inner zone. Part of the downward wall vortex flow in the lower zone of the cone turns upward, forming a drain. The hydrocyclone is the preferred embodiment of separators because it contains no moving parts, which increases its reliability while providing high efficiency in separating heavier and lighter fractions. The pressure in the separation tank should be maintained in the range of 0.25 to 0.27 MPa, and the temperature should be maintained at 220-260°C.

[0065] The mixture entering the separation tank is separated in a hydrocyclone into an upper light stream and a lower heavy stream. The upper light stream, which includes predominantly a liquid phase, is sent to a vacuum stripping column. In case of multiple sections, the upper flow is directed to the mixing tank of the second section.

[0066] The lower heavy stream from the hydrocyclone, which includes predominantly wet solids, is removed from the process.

[0067] Suitable solvents for the section of washing the solid granular material can include heavy reformat, light or heavy catalytic cracking gas oil, and toluene.

[0068] Preferably, the solvent is aromatic light gas oil from petroleum processing and petrochemical process to increase the aromatic hydrocarbon content, in particular, of catalytic cracking due to the content of C₆₋₁₈ aromatic hydrocarbons exceeding 80% by weight.

[0069] The function of the solvent is to effectively reduce the viscosity of the unconverted residue and eliminate the precipitation of asphaltenes. Aromatic light gas oil is advantageous because it increases the aromatics proportion in a dispersed system and is free of paraffins, which are natural precipitants for asphaltenes. Thus, the group composition provided in aromatic light gas oil comprising more than 80 wt. % aromatic hydrocarbons provides better separation of solid material from the unconverted residue.

[0070] The purification degree of the unconverted residue from solid material plays an important role when the unconverted residue is processed into a petroleum sintering additive, which must have low reactivity and low ash content to improve its performance properties. Catalytic or carbon particles of the solid granular material, if present in the additive composition, negatively affect these parameters.

[0071] Light aromatic gas oil, resulting from petroleum processing, is usually used to produce diesel fuels and, as a consequence, its use as a solvent is impractical and unprofitable. Therefore, in order to provide additional light aromatic gas oil, it is proposed to use heavy vacuum gas oil produced by the method according to the present invention, as described below. This additional amount can be used as a solvent in the separation step, which will additionally increase the efficiency and reduce the resource intensity of the method according to the invention. Thus, the present invention provides an additional source of feedstock for the production of light aromatic gas oil, at least a portion of which can be used as a solvent according to the present invention. The features of providing said feedstock source will be clear from the further description of the method.

[0072] It should be noted that the smaller the amount of asphaltene compounds remains in the unconverted residue, i.e. the more efficient the solid granular material, the less aromatic solvent is required in step 4 of separating spent carbon additive from the unconverted residue. In addition, the more efficiently the solid granular material is separated from the unconverted residue in separation step 4, the more stable the unconverted residue in terms of a petroleum dispersed system.

[0073] After the washing section, the granular material can be removed from the process or recycled to the SPH step,

and the separated unconverted residue mixed with the solvent passes to step 5 to a vacuum column. The vacuum at the top of the vacuum column is from 10 to 150 mmHg, preferably from 10 to 70 mmHg, even more preferably from 10 to 30 mmHg. The pressure difference between the bottom part of the vacuum column and the lower packing bed, including a "blind" plate, is not more than 15 mmHg, preferably not more than 10 mmHg, even more preferably from 5 to 7 mmHg. The temperature of the vacuum still in the vacuum column is not more than 305°C, preferably from 250 to 295°C, even more preferably about 270°C. In the vacuum column, the mixture of the solvent is separated from the separated unconverted residue.

[0074] The products resulted from the vacuum distillation process are:

- a regenerated solvent;
- light vacuum gas oil (LVGO) and purified vacuum gas oil (VGO), and
- a separated heavy hydrocracking residue.

[0075] The composition of the separated heavy residue is consistently homogeneous, viscous, low-ash, with a low sulfur content, and free of benzopyrene, which is important for the environment. Since the step of hydrocracking heavy petroleum feedstock is carried out in a hydrogen environment, the products of this process, in particular residual products, comprise a reduced content of sulfur and are free of benzopyrenes. In the washing section, an aromatic solvent is preferable to use because the contained aromatic compounds allow the asphaltene compounds to be dispersed, and the absence of paraffin compounds prevents their precipitation. The solvent advantageously has a composition that effectively reduces viscosity to maximize the removal of the solid granular material to ensure stability and uniformity, as well as reactivity and ash content of both the separated heavy residue and the petroleum sintering additive based thereon. In the case of using a carbon additive, it is preferable to comply with the requirement for a mesopore content of more than 25% of the total pore volume since this allows an additional increase in the adsorption efficiency and removal of asphaltene compounds from the unconverted residue.

[0076] The separated heavy residue has properties and composition which facilitate its use as a feedstock to prepare a sintering additive used for the production of metallurgical or foundry coke or electrode mass in the manufacture of carbon anodes, for example, for the aluminum industry. In addition, the concentrated residue can be used to produce petroleum coke or anode coke, for example, in a delayed coking unit.

[0077] To obtain a sintering additive, the separated heavy residue must be subjected to concentration step 6 to remove heavy vacuum gas oil (VG). Such a concentration apparatus can be a traditional vacuum column, or it can have a special function for stripping VG from the separated heavy residue by forming a film of evaporated material, which facilitates the evaporation of low-boiling components from the separated heavy residue. Special film-forming evaporators are capable of stimulating the evaporation of VG quickly enough to avoid coking. Film evaporators can be a stripping evaporator, a thin-film evaporator, a film evaporator, a falling film evaporator, a rising film evaporator, and a scraper evaporator. Some of these film-forming evaporators can include a movable part for resurfacing the separated heavy residue in the concentration apparatus. Other types of thin-film evaporators are also applicable. For example, a thin-film evaporator (TFE) heats the separated heavy residue on the inner surface of a heated pipe until the VG begins to evaporate. The separated heavy residue is kept in the form of a thin film on the inner surface of the pipe using a rotating blade with a fixed gap. VG vapors are then liquefied on cooler pipes of a condenser. A film evaporator (WFE) differs from a thin-film evaporator in that it uses a hinged blade with minimal gap clearance from the internal surface that agitates the flowing heavy residue to promote separation. In both the thin-film evaporator and the WFE, the heavy residue enters the device tangentially above a heated inner tube and is evenly distributed around the inner periphery of the tube by a rotating blade. The heavy residue material spirally moves down along the wall, and the VG evaporates. The VG can condense in a condenser located outside the evaporator, but as close as possible to it. Another type of evaporator is a molecular distillation device that has an internal condenser. A scraper evaporator works on a principle similar to the WFE's principle. However, the scraper evaporator is designed not only to maintain a thin film on the internal heated surface, but also to protect the film on the heated surface from overheating by frequent scraping.

[0078] In a falling film evaporator, the separated residue enters the evaporator from the top and is evenly distributed over heating pipes. A thin film enters the heating pipes and moves downward at the boiling point, partially evaporating. An inert gas, such as water vapor, can be used to heat the pipes from the outside. Both the heavy residue and the VG vapor move down through the pipes to a lower separator, where the vaporous VG is separated from the heavy residue.

[0079] A rising film evaporator operates on the thermosyphon principle. The heavy residue enters heating pipes from below, which are heated by water vapor supplied to the outer surface of the pipes. As the heavy residue heats up, VG vapors begin to form and rise upward. The ascending force of this evaporating VG causes liquid and vapors to flow upwards in parallel flow. At the same time, the production of VG vapors increases and the rising heavy residue is compressed into a thin film on the walls of the pipes. This co-current upward movement against gravity has a beneficial effect of creating a high degree of turbulence in the heavy residue material, which promotes heat transfer and coke inhibition.

[0080] In one aspect, the evaporator can be a specially designed TFE, as described below, configured to concentrate the separated heavy residue by evaporation to produce a concentrated hydrocracking residue (CHR) and a heavy vacuum gas oil (HVGO). At the same time, for the quality of CHR and HVGO, it is important to prevent local overheating of TFE because this leads to local coking of a film with the risk of formation of larger volumes of coke deposits inside the apparatus. Such coking-prone inclusions in CHR, when used as a sintering additive, reduce its sintering properties due to a solid carbon fraction remained in the coked material that loses its sintering properties and acts as ballast in the sintering additive. The main elements of the TFE according to the present invention, as generally illustrated in Fig. 3, are a tubular casing with a vertical wall forming a chamber, a distribution device located in the upper part of the casing and configured to supply hydrocracking residue into the chamber, a rotor mounted coaxially to the casing, and blades mounted on the rotor. To prevent local coking, the TFE was equipped with a double jacket heated by flue gases which are fed into the outer jacket and then distributed into the inner one. This feature is illustrated in Fig. 3. The presence of two jackets makes it possible to evenly distribute flue gases over the outer surface of the reactor vessel and avoid local overheating.

[0081] A film is created on the vertical inner wall of the chamber using rotor blades. To ensure a constant film thickness, the blades are distributed along the height of the rotor, forming a row in the form of a fragment of a spiral, with adjacent blades in the row located partially overlapping each other, as shown in Fig. 4.

[0082] All other things being equal, the higher the heating temperature of the feedstock, the better the quality of the sintering additive in terms of "ring and ball softening temperature (R&B)", but the lower its yield. The maximum temperature in the chamber is limited by the possibility of coke formation and the residence time of the mixture in the evaporator. Preferably the temperature is 400-450°C.

[0083] The vacuum in the system can significantly reduce the temperature at which light hydrocarbons begin to evaporate and can reduce the risk of coking of the separated heavy residue. A decrease in pressure promotes reducing the content of volatile components in the sintering additive due to improved conditions for evaporation of intermediate products (or secondary resins). Preferably, the process occurs at a residual pressure of minus 85 kPa to minus 99 kPa relative to atmospheric pressure (i.e., from 2.325 kPa to 16.325 kPa).

[0084] The residence time of the feedstock in the apparatus is calculated based on the condition required to obtain a product with a residual mass fraction of volatile substances of not more than 60%, and preferably ranges from 20 to 30 seconds.

[0085] It is desirable to carry out the process from a film with a thickness of not more than 1.5 mm, most preferably not more than 1.2 mm, and in the range of 1.1 to 1.15. The evaporation of a substance from a thin film of the specified thickness on the evaporator surface provides high rates of heat and mass transfer. In addition, the film thickness directly impacts the quality of the resulting sintering additive, ensuring fewer volatile substances and enhanced sintering capability. In addition, a film with the specified thickness according to the claimed method reduces the risk of coking. In a larger film thickness, there is a risk of coking on the walls, and the scrapers can become ineffective, which can lead to jamming of the rotor. If the thickness is less than the specified one, then evaporation will occur too intensely, the residue will not have time to drain, which will also lead to local build-up, which in turn will lead to coking.

[0086] The stream of the separated heavy residue after the vacuum stripping column is supplied to the upper part of the TFE by a distribution device. The distribution device comprises a manifold made in the form of a hollow circular tube having supply holes evenly distributed along the diameter, as shown in Fig. 5. Preferably, the distribution device further comprises a circular distribution plate configured to receive hydrocracking residue coming from the supply holes. This input ensures additional prevention of equipment from coking over time and the elimination of coking inclusions in the resulting sintering additive.

[0087] Stream redistributors are provided along the height of the apparatus, which are circle-shaped metal plates installed along the height of the apparatus. The plates have grooves for blades. They are aimed at ensuring uniform application of the feedstock stream to the walls along the height of the reactor, thereby eliminating stagnant zones. This feature is shown in Fig. 6.

[0088] To intensify the process, evaporation can be conducted in the presence of oxygen. For this purpose, an air supply can be provided to the lower part of the TFE at a rate of 40-50 L/hour, preferably 44-47 L/hour, even more preferably 45 L/hour, depending on the composition of the feedstock, as well as the necessary requirements for the quality of the sintering additive. In this case, the process temperature can be reduced to 210-240°C.

[0089] The concentrated hydrocracking residue (CHR) is removed from TFE's still. In some embodiments, constant circulation of the CHR is provided in the TFE's still by tangential input into the lower part of the TFE.

[0090] The upper product of the TFE, which is distillate vapor, is removed from the reactor and condensed in a refrigerator. The condensed distillate, which is heavy vacuum gas oil (HVGO), is collected in a distillate collection tank. A portion of the resulting HVGO can be recycled into the TFE in a mixture with the separated heavy residue. At least a portion of the HVGO is involved in processing step 7 to increase the content of aromatic hydrocarbons, in particular, of catalytic cracking to obtain an aromatic solvent for the additive washing section.

[0091] HVGO after processing in the TFE has a composition and properties suitable for processes such as, for example, catalytic cracking, namely:

- a low content of catalytic poisons, in particular nickel (not more than 0.006 wt. %);
- a kinematic viscosity at 50°C of not more than 60 mm²/s;
- a fractional composition close to the fractional composition of straight-run vacuum gas oil;
- a low coking capacity and asphaltenes content; and
- a low sulfur content (not more than 3 wt. %).

[0092] The HVGO is supplied to catalytic cracking in a mixture with one or more components of straight-run vacuum gas oil, hydrotreated vacuum gas oil from a combined hydrocracking unit, and fuel oil. The ratio between these four feed streams of a catalytic cracking unit can vary over wide ranges, wt. %:

- hydrotreated feedstock (hydrotreated vacuum gas oil from the combined hydrocracking unit and/or fuel oil from a gas condensate processing unit) of 10 to 80; and
- non-hydrotreated feedstock (HVGO and, optionally, straight-run vacuum gas oil) of 20 to 90.

[0093] It is important to recognize that increasing a non-hydrotreated feedstock fraction leads to a higher yield of light gas oil from catalytic cracking. However, to prolong catalyst life, it is advisable to dilute non-hydrotreated feedstock with hydrotreated one. In addition, a non-hydrotreated feedstock fraction should not be increased, as this could adversely affect the quality of the main product - catalyst, which is then used in the production of motor gasoline.

[0094] In the case of using fuel oil, it is important to note that straight-run fuel oil obtained by distillation from petroleum cannot be used for catalytic cracking in the classical sense. For catalytic cracking, fuel oil obtained from a gas condensate processing unit (GCPU) is used since in this case its properties are similar to vacuum gas oil obtained by distillation from petroleum, i.e. the GCPU fuel oil lacks heavy fractions (tar).

[0095] The TFE bottom product is a concentrated hydrocracking residue. The concentrated residue obtained according to the present invention is characterized by a low ash content, in particular, not more than 1.0 wt. %, preferably not more than 0.6 wt. %, an R&B softening point of at least 105°C, and a volatile component content of not more than 60 wt. %. The characteristics of the resulting concentrated residue allow its use as a sintering additive for the production of various types of products in the coke industry. Suitable products can be, for example, coke, more particularly metallurgical coke, foundry coke, in particular molded coke, where sintering additive is used as part of charge for their production. In addition, sintering additive can be used as part of charge for the production of carbon electrodes, such as an anode or cathode for galvanic processes, in particular, for the production of aluminum. Sintering additive can also be used as part of charge for the production of self-sintering electrodes. The concentrated residue may undergo additional processing, for example, in a delayed coking unit, to yield petroleum coke or anode coke.

[0096] The invention provides stable non-stop operation of the combined hydrocracking unit without coking of the equipment, the resulting products with advantageous performance characteristics, and the resolves the issue of processing residual hydrocracking products into marketable products.

[0097] According to the present invention, the stable operation of the combined hydrocracking unit means continuous operation in established modes with a given productivity.

Example

[0098] Heavy petroleum feedstock, which was tar obtained after distillation of low-boiling fractions from heavy Urals crude oil and had an initial boiling point of at least 510°C and a density at 20°C of more than 1000 kg/m³, was mixed with 1.5 wt. % (based on the tar weight) of carbon additives of two granulometric compositions: coarse fraction with a particle diameter of about 1 mm and a fine fraction with a particle diameter of about 0.3 mm. The coarse and fine fractions were characterized by different mesopore volumes: the BJH mesopore volume of the fine fraction was at least 0.07 cm³/g, and the BJH mesopore volume of the coarse fraction was at least 0.12 cm³/g for more efficient adsorption of asphaltenes with a molecule size of 40 to 90 nm for tar from Urals crude oil. The carbon additive had a BET specific surface area of not less than 230 m²/g and not more than 1230 m²/g.

[0099] The feedstock in the form of slurry was supplied to SPH, where hydrogen was supplied at a temperature of 450°C and a pressure of 20 MPa. A mixture of the carbon additive, tar and gas passed through three SPH reactors. The resulting mixture consisted of gaseous products and slurry comprising a spent carbon additive and an unconverted high-boiling residue. This mixture was delivered to the separation step, after which a gaseous stream was subjected to gas-phase hydrocracking, and the slurry was sent to the additive washing section consisting of a mixing tank and a cyclone separation tank.

[0100] The slurry of the unconverted high-boiling residue together with the spent solid additive at a flow rate of 15-20 tons/h, a temperature of about 420°C, and a pressure of not more than 0.3 MPa was mixed in a mixing tank with an aromatic light gas oil from catalytic cracking supplied at a flow rate of 30-35 tons/h, a temperature of about 220-260°C. The pressure in the mixing tank was from 0.15 to 0.35 MPa(g) and was regulated by a system of control valves to avoid excessive

evaporation of the solvent.

[0101] Further, the stream was fed into a separation tank equipped with a cyclone unit, where the spent additive was separated from the unconverted high-boiling residue mixed with the aromatic light gas oil from catalytic cracking under the action of centrifugal forces.

[0102] After the washing section, the spent carbon additive was extracted from the process, and a separated unconverted high-boiling residue heated to a temperature of not more than 385°C and mixed with the aromatic light gas oil from catalytic cracking was delivered to a vacuum column. At the top of the vacuum column, the vacuum was about 20 mmHg, the pressure difference between the bottom part of the vacuum column and the lower packing bed, including a "blind" plate, was about 7 mmHg, and the temperature of the still of the vacuum column was about 290°C.

[0103] The products obtained from the vacuum distillation process were:

- light vacuum gas oil (LVGO) and vacuum purified gas oil (VPGO); and
- separated heavy residue, which was a tar-hydrocracking residual product (THRP).

[0104] The heavy residue (bottom residue) obtained by the above method had the following physical and mechanical properties:

Table 1

1	Density at 15°C, kg/m ³	1.054
3	Flash point in open cup, °C	195
4	Mass fraction of sulfur, % by weight	1.945
5	Coking capacity, % by weight	21.21
6	Dynamic viscosity, sPa at 200°C at 240°C	221 45
7	Fractional composition, % by weight	
	Initial boiling point, °C	340
	130-180°C Fraction	
	180-200°C Fraction	
	200-340°C Fraction	
	340-460°C Fraction	22.98
	Residue, more than 460°C	77.02
	460-480°C Fraction	7.60
	480-500°C Fraction	7.60
	500-540°C Fraction	14.80
	Residue, more than 540°C	47.02
8	Asphaltenes, % by weight	20.69
9	Carbenes, % by weight	1.01
10	Carboids, % by weight	2.27
11	Setting point, °C	plus 30

[0105] The above bottom residue (the separated heavy residue) was fed through a manifold comprising discrete feed points into a thin-film evaporator (TFE) for concentration.

[0106] In the TFE, the temperature was maintained at 400°C. The pressure in the TFE was maintained at minus 95 kPa relative to atmospheric pressure (6.325 kPa vacuum).

[0107] The film thickness was 1.12 mm and was constant along the height of the apparatus.

[0108] The residence time of the feedstock in the TFE for the above-mentioned bottom residue and the specified film thickness was 20 seconds.

[0109] The distillate obtained by the method according to the present invention had the following characteristics:

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Table 2

No.	Parameter	Test method	Test results (average data)
1	Density at 20°C, kg/cm ³	GOST 3900	982.1
2	Mass fraction of sulfur, %	GOST P 51947	1.93
3	Coking capacity, % by weight	EN ISO 10370	1.55
4	Fractional composition:		
	- initial boiling point, °C	ASTM D 86	302
	- distilled at 400°C, %		37
5	Kinematic viscosity at 50°C, mm ² /s	GOST 33	56,12
6	Setting temperature, °C	GOST 20287 (Method B)	23.4
7	Flash point in closed cup, °C	ASTM D 93	175.4
8	Asphaltenes content, mg/kg	Total 642	710.6
9	Metal content		
	Sodium, mg/kg	ASTM D 5863	1.02
	Iron, mg/kg		20.32
	Nickel, mg/kg		2.51
	Vanadium, mg/kg		1.05

[0110] These parameters allow the resulting HVGO to be used as a feedstock for catalytic cracking.

[0111] The concentrated residue from tar hydrocracking produced by the proposed method has the characteristics shown in Table 3.

Table 3

Determined parameters	Unit of meas.	Test results	ND for test method
Ash content, dry state, A ^d	%	0.6	GOST 22692-77
Mass fraction of volatile substances, dry state, V ^d	%	52.4	GOST 22898-78
Mass fraction of total sulfur, dry state, S _t ^d	%	2.23	GOST 32465-2013
Mass fraction of total carbon, dry state, C ^d	%	87.3	GOST 32979-2014
Mass fraction of water, W	%	0.1	GOST 2477-2014
Mass fraction of insoluble substances in toluene, α	%	25	GOST 7847-2020
Mass fraction of substances insoluble in quinoline, α ₁	%	5	GOST 10200-2017
R&B softening point (melting point), T	°C	113	GOST 9950-2020
B&R Softening point (melting point), T	°C	128	GOST 11506-1973
Softening (melting) temperature according to Mettler, T	°C	131	GOST 32276-2013
Gray-King coke type	type	G ₁₃	GOST 16126-91 (ISO502-82)
Coking index, G (1:5)	unit	80	GOST ISO 15585-2013
Coking index, G (1:7)	unit	68	GOST ISO 15585-2013

[0112] These parameters enable the use of THRP as a sintering additive in the production of metallurgical coke, foundry coke, or anodes for the aluminum industry, which have excellent sintering properties similar to the sintering properties of coal tar pitches.

[0113] As a result of industrial tests of the claimed method, the achieved productivity of feedstock, in particular tar, was at least 2,600,000 tons per year.

Claims

1. A method for processing heavy petroleum feedstock, comprising the steps of:

- suspending solid granular material in the feedstock and subjecting the resulting slurry to hydrocracking in the presence of hydrogen in a slurry-phase hydrocracking reactor to obtain a heavy residue stream, wherein the heavy residue stream is a slurry of an unconverted hydrocracking residue and a spent solid granular material;
- separating the spent solid granular material from the unconverted hydrocracking residue using a solvent in a washing section to obtain separated spent solid granular material and a mixture of the unconverted hydrocracking residue and the solvent;
- delivering the mixture of the unconverted hydrocracking residue and the solvent to a vacuum column for separating the solvent to obtain a separated heavy residue;
- evaporating at least a portion of the separated heavy residue in an evaporator to obtain a concentrated hydrocracking residue and heavy vacuum gas oil (HVGO); and
- using at least a portion of the HVGO to produce the solvent.

2. The method according to claim 1, wherein the solid granular material is an adsorbent or a catalyst.

3. The method according to claim 2, wherein the adsorbent is a carbon material.

4. The method according to claim 1, wherein the slurry-phase hydrocracking further produces a gaseous mixture of hydrocarbons which is subjected to gas-phase hydrocracking, followed by fractionation of the hydrocracking products.

5. The method according to claim 1, wherein at least a portion of the HVGO is subjected to catalytic cracking to produce the solvent.

6. The method according to claim 5, wherein the HVGO is supplied for catalytic cracking in a mixture with at least one of the following components: straight-run vacuum gas oil, fuel oil from a gas condensate processing unit, and hydrotreated vacuum gas oil.

7. The method according to claim 6, wherein the mixture for catalytic cracking is **characterized by** the following percentage ratios based on the weight of the mixture:

- 10 to 80 hydrotreated vacuum gas oil and/or fuel oil; and
- 20 to 90 HVGO and, optionally, straight-run vacuum gas oil.

8. The method according to claim 1, additionally comprising a step in which at least a portion of the HVGO is fed for recycling in a mixture with the separated heavy residue into the evaporator.

9. The method according to claim 1, wherein the heavy petroleum feedstock is **characterized by** an initial boiling point of at least 510°C and a density at 20°C of over 1000 kg/m³, in particular the heavy petroleum feedstock is tar.

10. The method according to claim 1, wherein the concentrated hydrocracking residue has an ash content of not more than 1.0%, preferably not more than 0.6%.

11. The method according to claim 3, wherein the carbon material consists of two fractions of particles, wherein the average particle size of a coarse fraction is greater than the average particle size of a fine fraction, and the ratio of the weighted average diameter of the coarse fraction particles to the weighted average diameter of the fine fraction particles varies from 2 to 7, wherein the coarse and fine fractions are **characterized by** different mesopore volumes.

12. The method according to claim 11, wherein the volume of mesopores for the fine fraction according to the Barrett-Joyner-Halenda (BJH) method is not less than 0.07 cm³/g and not more than 0.12 cm³/g, while the mesopore volume according to the BJH method for the coarse fraction is not less than 0.12 cm³/g and not more than 0.2 cm³/g.

13. The method according to claim 11, wherein the carbon material has a BET specific surface area of not less than 230 m²/g and not more than 1250 m²/g, preferably not less than 250 m²/g and not more than 900 m²/g, most preferably not less than 270 m²/g and not more than 600 m²/g.

14. The method according to claim 1, wherein the solvent is an aromatic light gas oil from catalytic cracking, comprising at least 80 wt.% of aromatic hydrocarbons having from 8 to 16 carbon atoms.

15. The method according to claim 1, wherein the evaporation is performed in a thin-film evaporator.

16. The method according to claim 15, wherein the thin-film evaporator has a double jacket heated by flue gases.

17. The method according to claim 15, wherein the separated heavy residue is fed into a thin-film evaporator using a manifold made in the form of a hollow circular tube having supply holes evenly distributed along the diameter of the tube.

18. The method according to claim 15, wherein the evaporation is performed from a constant-thickness film, wherein the thickness of the film is not more than 1.5 mm, preferably not more than 1.3 mm, and even more preferably from 1.1 to 1.15.

19. The method according to claim 15, wherein stream redistributors are provided along the height of the thin-film evaporator, which are circle-shaped metal plates installed along the height of the reactor.

20. The method according to claim 15, wherein the circulation of a bottom product in the thin-film evaporator with a tangential input is provided.

21. The method according to claim 1, wherein the evaporation is performed in the presence of atmospheric oxygen.

22. The method according to claim 18, wherein the evaporation from a constant-thickness film is carried out for a predetermined time at a temperature and an evaporation pressure which ensure the evaporation of volatile components to a volatile component mass fraction of at most 60% in the concentrated residue and to a ring-and-ball softening point of the concentrated residue of at least 105°C.

23. The method according to claim 1, wherein the HVGO is produced by condensing vapors of the evaporator using a refrigerator, followed by collection of the resulting distillate.

24. A system for processing heavy petroleum feedstock, comprising:

a slurry-phase hydrocracking section intended for slurry-phase hydrocracking heavy petroleum feedstock, the slurry-phase comprising a slurry of the heavy petroleum feedstock and a solid granular material, to produce a hydrocracking-subjected stream;

a separation section designed to receive the hydrocracking-subjected stream from the slurry-phase hydrocracking section intended for separating said hydrocracking-subjected stream to obtain a heavy residue stream, wherein the heavy residue stream is a slurry of an unconverted hydrocracking residue and a spent solid granular material;

a washing section designed to receive the heavy residue stream from the separation section and intended for separating spent solid material from the unconverted hydrocracking residue of the slurry to produce a separated spent solid granular material and a stream of a mixture of the unconverted hydrocracking residue and a solvent; a vacuum column designed to receive the stream of the mixture of the unconverted hydrocracking residue and the solvent from the washing section and intended for separating the mixture of the unconverted hydrocracking residue and the solvent to produce a regenerated solvent, vacuum gas oil and a separated heavy residue of hydrocracking; and

an evaporator designed to receive the stream of the separated heavy residue of hydrocracking from the vacuum column and intended for removing a heavy vacuum gas oil to obtain a low-ash concentrated heavy residue of hydrocracking and a stream of the heavy vacuum gas oil.

25. The system according to claim 24, wherein the slurry phase hydrocracking section comprises at least one slurry-phase hydrocracking reactor.

26. The system according to claim 25, wherein the washing section comprises at least one mixing tank and at least one separating tank.

27. The system according to claim 26, wherein the mixing tank is configured to mix a slurry of the spent solid material in an

unconverted hydrocracking residue with a solvent.

28. The system according to claim 26, wherein the separating tank is configured to separate the spent solid material from the mixture of the unconverted hydrocracking residue and the solvent, in particular, using centrifugal forces, gravitational forces, or flotation, preferably centrifugal forces.

29. The system according to claim 28, wherein the evaporator is a thin-film evaporator.

30. A concentrated slurry-phase hydrocracking residue obtained by the method according to any one of claims 1 to 22, **characterized by** an ash content of not more than 1.0% and a ring-and-ball softening point of not less than 105°C.

31. Use of the concentrated residue according to claim 30 as a sintering additive in charge to produce a coke.

32. The use according to claim 31, wherein the coke is metallurgical coke, foundry coke, in particular molded coke.

33. Use of the concentrated residue according to claim 30 as a sintering additive in charge to produce carbon electrodes.

34. The use according to claim 33, wherein the carbon electrodes are an anode or cathode for galvanic processes, in particular, for the production of aluminum.

35. Use of the concentrated residue according to claim 36 as a sintering additive in charge to produce self-sintering electrodes.

36. Use of the concentrated residue according to claim 30 to produce petroleum coke.

37. The use according to claim 36, wherein the petroleum coke is anode coke.

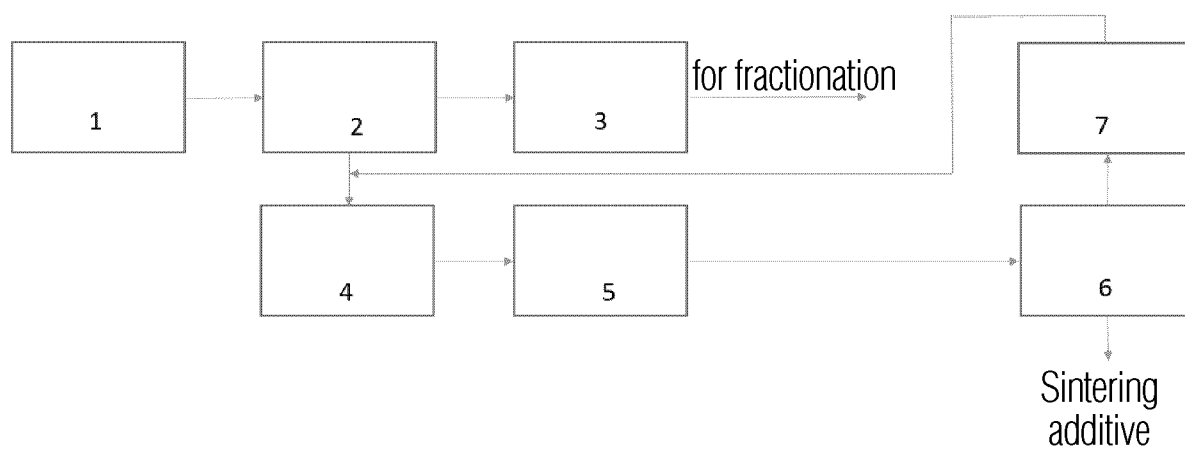


FIG. 1

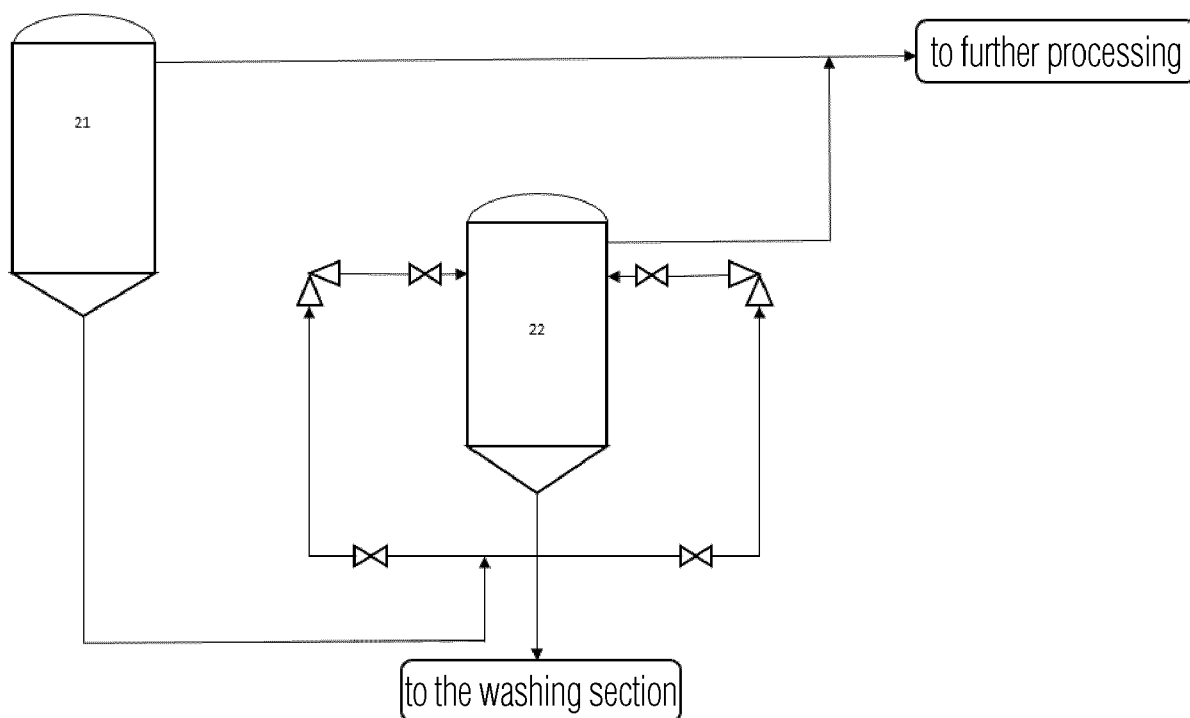


FIG. 2

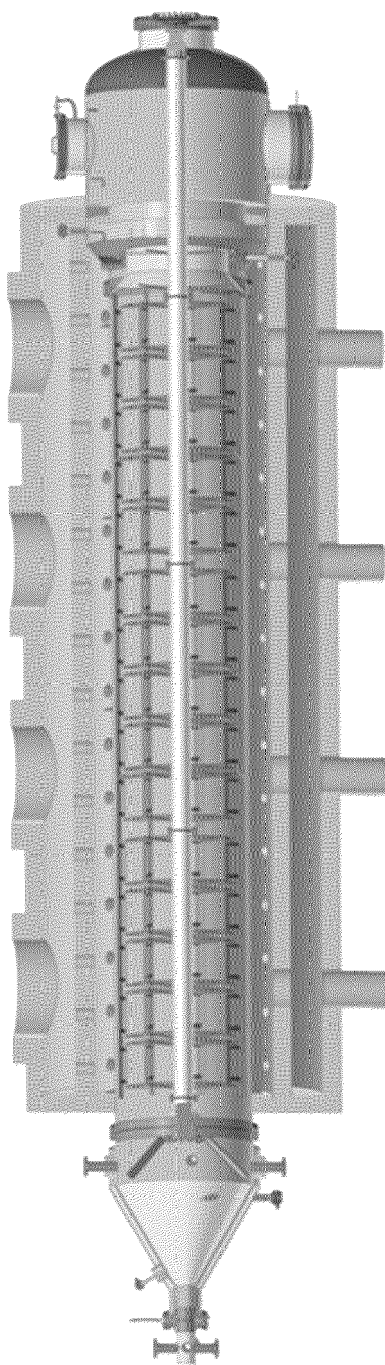


FIG. 3

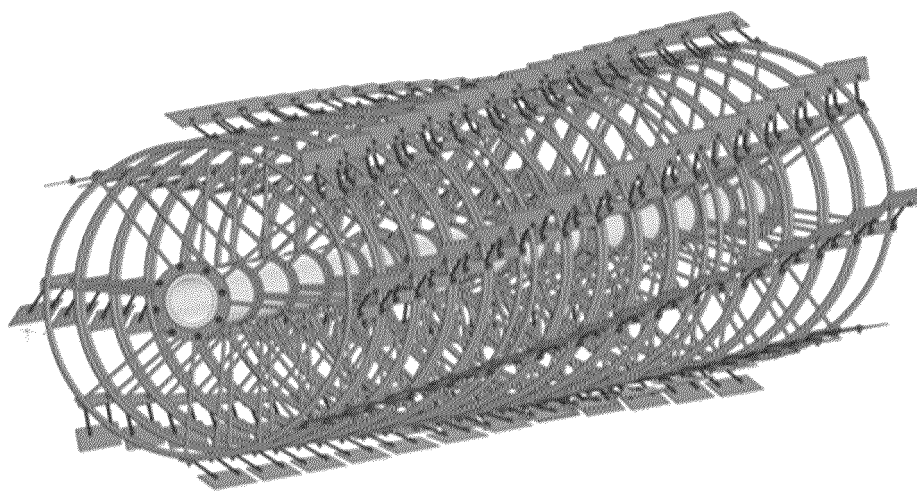


FIG. 4

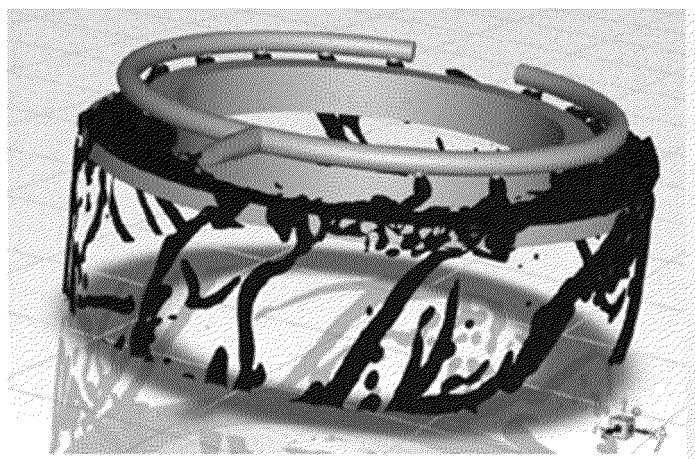


FIG. 5

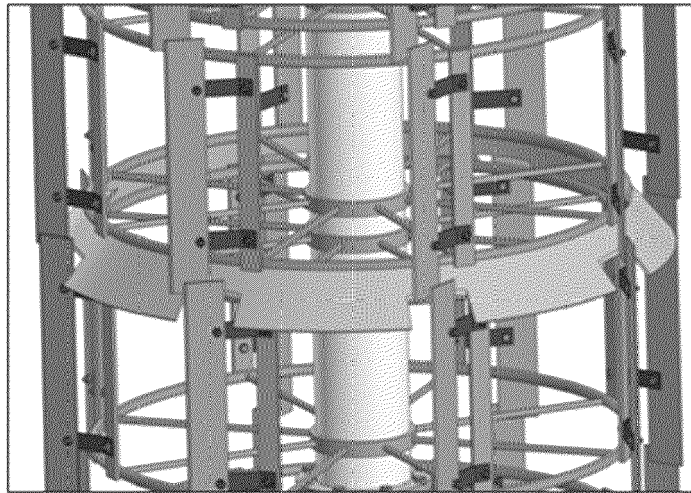


FIG. 6



EUROPEAN SEARCH REPORT

Application Number

EP 24 17 7294

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	US 2016/130511 A1 (GOVINDHAKANNAN JAGANNATHAN [US] ET AL) 12 May 2016 (2016-05-12) * paragraph [0008] - paragraph [0038] * * figure 1 *	1 - 15	INV. C10G7/06 B01J35/61 B01J35/63 C10G11/00 C10G21/00 C10G47/26
A	US 2017/369796 A1 (LE COZ JEAN-FRANCOIS [FR] ET AL) 28 December 2017 (2017-12-28) * figures 1-2 * * paragraph [0053] - paragraph [0188] *	1 - 15	
A	US 2017/128929 A1 (KUMAR BRIJESH [IN] ET AL) 11 May 2017 (2017-05-11) * figures 1-3 * * paragraph [0020] - paragraph [0048] *	1 - 15	
			TECHNICAL FIELDS SEARCHED (IPC)
			C10G B01J
The present search report has been drawn up for all claims			
Place of search		Date of completion of the search	Examiner
The Hague		11 February 2025	Bernet, Olivier
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

EPO FORM 1503 03.82 (P04C01)



Application Number

EP 24 17 7294

CLAIMS INCURRING FEES

The present European patent application comprised at the time of filing claims for which payment was due.

☐ Only part of the claims have been paid within the prescribed time limit. The present European search report has been drawn up for those claims for which no payment was due and for those claims for which claims fees have been paid, namely claim(s):

☒ No claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for those claims for which no payment was due.

LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

☐ All further search fees have been paid within the fixed time limit. The present European search report has been drawn up for all claims.

☐ As all searchable claims could be searched without effort justifying an additional fee, the Search Division did not invite payment of any additional fee.

☐ Only part of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the inventions in respect of which search fees have been paid, namely claims:

☐ None of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims, namely claims:

☐ The present supplementary European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims (Rule 164 (1) EPC).

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 24 17 7294

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
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11 - 02 - 2025

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

REFERENCES CITED IN THE DESCRIPTION

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