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(54) **METHOD OF LIQUEFACTION OF INFLAMMABLE MINERALS**

(76) Inventors: **Chuluun Enkhbold**, Ulaanbaatar (MN); **Brodt Alexander**, Beer-Sheva (IL)

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(57) **ABSTRACT**

The method of inflammable minerals liquefaction is related to the technology of producing synthetic liquid fuel and can be applied to the processing of any kinds of fossil power-generating raw materials into various inflammable liquid products. The object of the present invention is to ensure a complex utilization of the initial power-generating raw material, to

intensify its processing, to decrease the power consumption of synthetic liquid fuel production and to weaken the harmful impact of the technological process on the natural environment. The method includes initial raw material milling, its hydrogenation in the presence of a catalyst and separation of non-liquefied solid residue from synthetic oil. However, milling of the initial raw material is carried out in two stages in an aqueous solution of mineral salts catalyzing hydrogenation, which is saturated to an intermediate density between those of inflammable mineral and waste rock. In the course of milling accompanied by the release of methane into gaseous phase, which is capped for its further utilization, inflammable minerals floating to the surface from the milling zone are separated from non-inflammable impurities. A suspension of the light product of the first milling stage is heated and fed to the second milling stage. The light product of the second milling stage is squeezed from excessive liquid phase, mixed with hot paste-forming agent, diluted with an organic diluent, ground to the colloidal size and fed to hydrogenation. After that, the remaining non-liquefied residue is washed with an organic diluent, dried and mixed with dehydrated heavy product of the first milling stage. The obtained mixture is washed with water and withdrawn from the process, whereas the remaining washing water discharge is evaporated and returned to the first milling stage. Meanwhile, the heavy product of the second milling stage is dehydrated, cooled and returned to the starting point of the process. Zinc and tin chlorides, iron sulfates, ammonium molybdates and their various mixtures can be used for preparing the solution, in which the initial raw material is milled, whereas various compositions containing tetraline, anthracene oil, methyl naphthaline and cresol can be used as paste-forming agents. Isopropyl alcohol can be used as a paste diluent, and light petroleum as an organic diluent.

[Fig. 1]

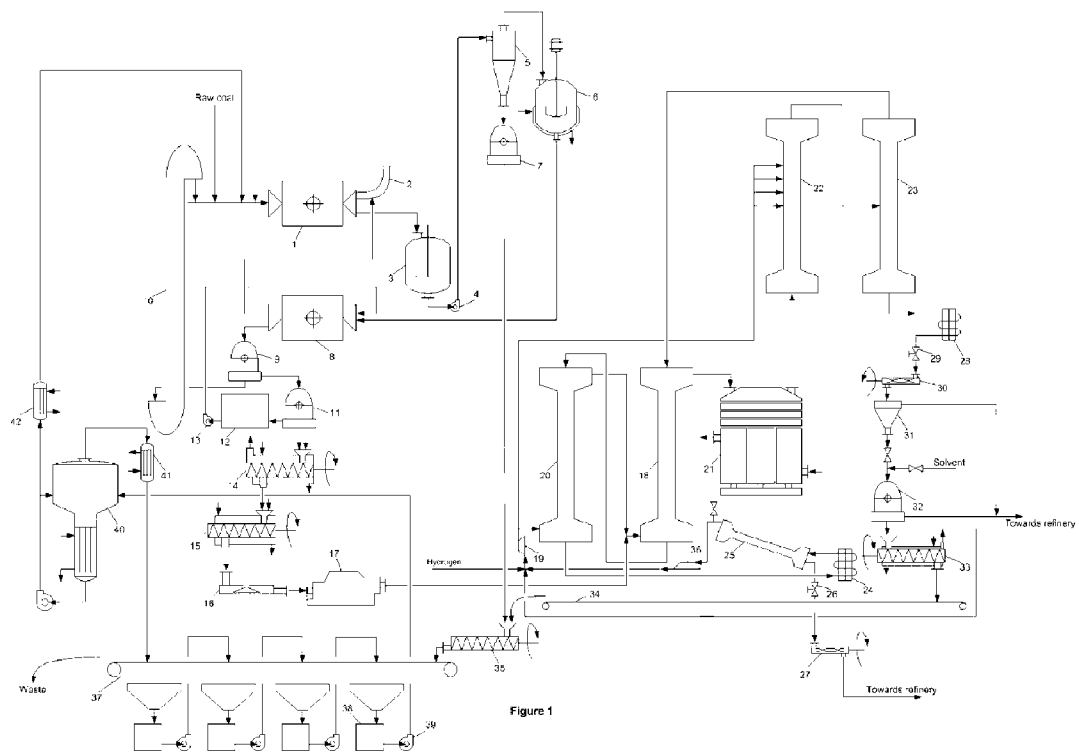


Figure 1

METHOD OF LIQUEFACTION OF INFLAMMABLE MINERALS

TECHNICAL FIELD

[0001] The invention is related to the technology of producing synthetic liquid fuel and can be applied to the processing of brown and bituminous coals, shale oil and other sapropelites, as well as lignites, peat, bituminous and sub-bituminous coals and other kinds of fossil power-generating minerals into synthetic petroleum products.

BACKGROUND ART

[0002] A method of coal liquefaction in the medium of lower aliphatic alcohols is known (see, for instance, Author's Certificate of the USSR No. 997441. According to this method, the initial dry raw material is milled, mixed with alcohol, the obtained suspension is heated to the necessary temperature, and coal liquefaction is carried out under an elevated pressure with a subsequent separation of the liquid phase from the remaining non-liquefied solid residue.

[0003] However, the mentioned method is characterized by a relatively low coal liquefaction efficiency and a significant non-productive alcohol consumption due to a considerable dehydration of the latter at high temperatures and elevated pressures.

[0004] A method of liquefaction of inflammable minerals, which is the closest to the claimed one in its technical idea and the achieved effect, includes preliminary milling of the initial power-generating raw material, its subsequent mixing with paste-forming substance representing a product of petroleum or coal origin, heating of the homogenized paste prepared in this way, and hydrogenation of coal substance forming a part of the processed inflammable mineral by its interaction with hydrogen under elevated pressure and at a high temperature in the presence of a catalyst. Then the reacted mixture is separated from the inert residue remaining in the solid state, and the obtained artificial petroleum is rectified into fractions, which results in the production of various kinds of liquid fuel and other mineral oil products.

[0005] Various oxides of iron, nickel, cobalt, molybdenum, aluminum, silicon, sulfides of sodium, nickel and iron with the addition of elemental sulfur, as well as various compositions on their basis and other chemical compounds, can be used as catalysts of inflammable minerals liquefaction according to the mentioned method (see the book of Goikhrakh I. M. and Pinyagin N. B. 'Chemistry and Technology of Synthetic Liquid Fuel', Moscow, Gostoptekhizdat, 1950, pp. 306-345).

[0006] Despite the more complete processing of the initial fossil power-generating raw material achieved by the mentioned method, the described technology does not ensure, nevertheless, its complex use, because in the course of dry coal milling, methane and other gaseous combustible hydrocarbons saturating the latter are released into the gaseous phase as its new solid surface becomes exposed. They get irreversibly lost volatilizing into the atmosphere and, moreover, destroy the stratospheric ozone.

[0007] Besides, the efficiency of liquefaction of power-generating raw material by the mentioned method sharply drops with its ash level growth. It leads to a considerable overconsumption of energy resources for the realization of this technological process and entails additional power consumption for the processing of rapidly growing hydrogenation

stage yield of nonutilizable sludge volume impregnated with synthetic petroleum. The necessity of a complete separation of the latter from the finely-dispersed solid phase involves an additional decrease in the technical and economic efficiency of this production process.

DISCLOSURE OF INVENTION

Technical Problem

Technical Solution

[0008] It is an object of the present invention to ensure a complex utilization of the initial power-generating raw material, to intensify its processing, to decrease the power consumption of synthetic liquid fuel production and to weaken the harmful impact of the technological process on the natural environment.

[0009] The object is achieved according to the present invention as follows. Before the hydrogenation, the initial raw material treatment is started with its preliminary milling in two stages realized in mineral salts solution. Only inorganic compounds catalyzing the hydrogenation process and possessing a high solubility in water can be used as such mineral salts, because the initial raw material milling according to the invention is carried out in a solution saturated to the density exceeding that of inflammable mineral, but inferior to that of waste rock. In this way, besides a simple size reduction, mineral components of the initial raw material are separated, namely, a light product representing an inflammable mineral floats up from the zone of the impact of milling bodies, whereas a heavy product consisting mainly of waste-rock minerals sinks in such medium. At the same time with size reduction, methane discharge from the disintegrated material into the gaseous phase takes place; the former is capped for external consumers and serves as a raw material for producing hydrogen to be used in the process of hydrogenation. A suspension of the light product of the first milling stage is heated before the second milling stage, and a repeated milling of this material is carried out in the hot regime. The light product of the second, hot milling stage is mixed after its dehydration with paste-forming agent, the obtained mixture is diluted with diluent, the solid phase of this paste is levigated to the colloidal size and fed to hydrogenation. After that, non-liquefied solid residue is rinsed with organic diluent, dried and mixed with dehydrated heavy product of the first milling stage. The obtained mixture of inert minerals is rinsed with water and removed from the production cycle. Washing water left after rinsing this production waste is evaporated to the original density, and the regenerated aqueous solution is returned to the first milling stage. The heavy product of the second milling stage is squeezed from excessive liquid phase, cooled down and also returned to the process starting point.

[0010] Only water-soluble inorganic compounds, whose water solubility allows the preparation of aqueous solutions with the density sufficient for floating of inflammable components of the initial raw material, can be used as mineral salts catalyzing the hydrogenation process according to the invention.

[0011] Both individual mineral salts with a sufficiently high water solubility, such as zinc or tin chlorides, ammonium molybdates or iron sulfates, and their various mixtures can be used as water-soluble compounds catalyzing the hydrogenation process. Water solubility of these hydrogenation catalysts is sufficient for preparation on their basis aqueous solu-

tions with the density sufficient for floating of inflammable components of the initial raw material.

[0012] Various compositions consisting of products of oil-refining, by-product-coking industry or fine organic synthesis containing organic substances of aromatic series capable of acting at their heating as atomic hydrogen donors (for instance, a mixture of tetraline with anthracene oil diluted with isopropyl alcohol) can be used as a paste-formative substance for realizing the method of the present invention.

[0013] In contrast to known solutions in the field of synthetic liquid fuel production using dry milling, the preliminary two-stage wet milling of the initial raw material according to the present invention, before its hydrogenation in aqueous-mineral medium, whose density is intermediate between those of inflammable mineral and its non-inflammable components, leads to automatic slipping of inflammable minerals

[0014] from under the impacts with milling bodies and, thus, to a reduction of ball loads of the mills. In combination with a narrowing of the technological flow be heated to the reaction temperature, which is fed to the hydrogenation stage, and minimization of power inputs connected with further separation of liquid products from the solid impurities carried out with the latter from the hydrogenation process, the realization of the method of the invention ensures an essential decrease in power consumption of this industrial process.

[0015] Hence, all distinctive features of the invention are organically interconnected, and the achievement of the object of the invention is ensured only in such combination of said features. The analysis of distinctive features of known technological processes described in scientific and technical literature, as well as the revealed main points of engineering solutions forming the basis of inventions found in the process of patent search have shown a total lack of any methodological similarity between the technological features of the proposed method and distinctive features of known methods of inflammable minerals liquefaction.

[0016] Thus, the invention possesses not only novelty, but also essential distinctive features imparting a number of obvious technical and economical advantages to the method of the invention, which favorably distinguish the latter from known engineering solutions in the area of synthetic liquid fuel production.

[0017] The method is realized by a consecutive realization of the following principal operations:

[0018] preparation of aqueous medium for the initial raw material milling by a preliminary water dissolution of mineral salts catalyzing the hydrogenation process so that the density of the prepared aqueous solution were intermediate between those of the inflammable mineral and contaminating mineral impurities;

[0019] the first (cold) stage of milling lumps of initial freshly-mined (or just delivered to the processing place by hydrotransport) power-generating raw material at the ambient temperature in aqueous solution saturated to the density value, which is intermediate between those of inflammable and non-inflammable mineral components, with a simultaneous capping of methane released into the gaseous phase and subsequent separate withdrawal of light and heavy milling products from this process;

[0020] heating of the suspension of the light product of the first milling stage up to the temperature that does not cause aqueous solution boiling;

[0021] the second stage of milling carried out in a heated aqueous solution with a simultaneous (as at the first stage of milling) withdrawal of methane released in the course of size reduction of the milled material and a subsequent separate withdrawal of light product directed later to the paste preparation and heavy product returned, respectively, to the first stage of milling;

[0022] squeezing of light product of the second milling from excessive aqueous-salty medium and its mixing with hot paste-forming agent with a simultaneous removal of moisture residues from the solid phase and subsequent dilution of the obtained mixture with an organic diluent;

[0023] additional grinding of the solid phase of the prepared paste to a colloidal particle size;

[0024] heating of the homogenized paste to the reaction temperature and its subsequent feeding to hydrogenation;

[0025] hot catalytic hydrogenation of coaly ingredients of the paste by their interaction with hydrogen under an elevated pressure;

[0026] separation of vapor/gaseous phase from the liquid phase of the reacted mixture with a subsequent condensation of the light hydrogenate and recirculation of non-condensed gases into the hydrogenation system by cooling the heavy hydrogenate and individual throttling of liquid products of hydrogenation;

[0027] separation of the heavy hydrogenate from the remaining solid impurities and its feeding to further processing;

[0028] cleaning of the solid residue extracted from the heavy hydrogenate from the infiltrating liquid phase with an organic diluent with its subsequent desiccation and withdrawal from the process;

[0029] mixing of the dry solid hydrogenation residue with the dehydrated heavy product of the first stage of the initial raw material milling;

[0030] cleaning of the obtained moist mechanical mixture of inert non-inflammable minerals with water and subsequent squeezing of such non-utilizable product from the excessive liquid phase;

[0031] evaporation of rinse water remaining after washing the mixture of solid non-utilizable products until the evaporated solution reaches the initial density intermediate between those of the inflammable mineral and mineral impurities, and return of the regenerated aqueous/mineral medium to the starting point of the process.

Advantageous Effects

DESCRIPTION OF DRAWINGS

[0032] The idea of the method is clarified by a technological schematic diagram of the apparatus of liquid-phase coal hydrogenation according to the invention shown in FIG. 1.

[0033] Freshly-mined (or delivered to the processing site by hydrotransport) initial lump coal is charged into tumbling ball mill 1 flooded with aqueous solution of hydrogenation catalyst representing a 40% aqueous solution of zinc chloride with the density 1.417 g/cm³. Here the finest particles of the combustible mineral, which are not contaminated by waste rock and not conjoined with it, do not sink and remain afloat on the surface of such liquid medium, while the rest of the initial raw material components submerge into the zone of milling bodies impact. As a result, as the initial raw material

is refined by steel balls permanently rolling inside the mill drum, coal lumps exposed in the course of this process, irrespective of the attained refinement degree, float to the surface of such liquid automatically avoiding, in this way, energy over-consumption for their additional re-refinement. Meanwhile, methane released into the gaseous phase (as well as other gaseous hydrocarbons) bubbles through the layer of aqueous-salt solution and then is either fed by manifold 2 to catalytic conversion in order to obtain hydrogen for the hydrogenation stage, or delivered to outside customers as a combustible gas.

[0034] The discharge of mill 1 is fed to sump 3 equipped with a stirrer, wherefrom it is fed by pump 4 to hydrocyclone 5, where a more intense separation of coal (after milling in mill 1) from contaminating mineral impurities representing heavier components of the initial power-generating raw material takes place. The light product of the first milling stage discharged from the cylindrical part of hydrocyclone 5 is fed to container 6 with an agitator heated by steam. As a result of heating, the density of the liquid phase of such suspension drops from 1.417 g/cm³ (at 20° C.) to 1.344 g/cm³ (at 100° C.). For a more complete opening of the aggregations, this hot technological flow is fed to the second milling stage, which is realized in a heated (but not reaching the boiling temperature) solution of zinc chloride in water, in tumbling ball mill 8, which is also connected by its end face to methane manifold 2 and covered with a thick layer of thermal insulation. Meanwhile, the heavy product of the first milling stage discharged from the conical part of hydrocyclone 5 is squeezed from the excessive liquid phase on centrifugal filter 7 and fed to mixer 35 for mixing with the remaining solid phase extracted out of the heavy hydrogenate in the end of the flow chart. The fugate of centrifuge 7 is returned to the first milling cycle for mixing with the initial as-received coal fed to mill 1.

[0035] The flow discharged from mill 8, similarly to the discharge of mill 1, is also fed to separation. However, the latter is realized in sedimentation centrifuge 9 (and not in a hydrocyclone), which is thermally insulated, just as mill 8, in order to prevent the return of the liquid phase density to its initial value, at which the first stage of milling was carried out. Thus, a high-purity coaly material representing the light product of the second milling stage, which is wholly free from visible mechanical impurities, is carried out together with the fugate of sedimentation centrifuge 9. Meanwhile, the discharged cake representing the heavy product of the second milling stage and containing residues of the combustible substance, is returned to mill 1 for regrinding in a cold aqueous salt solution of the initial density by means of elevator 10 equipped with a cooling jacket and blown by a fan for cooling the transported material to the ambient temperature.

[0036] The final deep squeezing of the light product of the second milling stage is realized by feeding the fugate of sedimentation centrifuge 9 to centrifugal filter 11. After that, the obtained hot transparent water-mineral medium is returned by pump 13 from sump 12 to mill 8, while wet coal cake impregnated with aqueous zinc chloride solution is fed to screw mixer 14 heated with indirect steam in order to mix with hot paste-forming agent. The latter represents tetraline (1,2,3,4-tetrahydronaphthalene C₁₀H₁₂) with an admixture of anthracene oil (coal-tar resin fraction boiling within the limits from 270 to 360° C.). At this stage, not only paste formation takes place, but also deep coal desiccation from moisture residues, and as a result, mixer 14 operates in rarefaction conditions. Water vapors released in the course of said pro-

cess are diverted for condensation, and the uniform paste-like mass obtained in screw mixer 14 is further diluted in screw mixer 15 (equipped with a cooling jacket) with a diluent representing isopropyl alcohol (isopropanol CH₃CHOHCH₃) and fed to disperser 16. In the latter, the solid phase of such diluted paste is levigated to the colloidal size in a strong centrifugal field.

[0037] The coal deeply purified from mineral impurities and levigated in disperser 16 to ultrafine state is fed, as a liquid homogenized paste, into recuperative heat exchanger 18 by plunger pump 17 equipped with a hydraulic drive. At the inlet of this heat exchanger, hydrogen preheated in heater 20 is added to the paste fed into said heat exchanger for heating. Heated reaction mixture is fed for final heating to 400° C. into high-pressure pipe furnace 21 heated by gas burners. From this furnace, it is fed into hydrogenation reactor 22, where chemical interaction of carbon-containing substances forming a part of the initial raw material with ordinary molecular hydrogen entering the system from outside (from a facility of conversion of methane obtained during the initial raw material milling) occurs, side by side with the reaction of coaly substance hydrogenation by atomic hydrogen, which is cleaved from paste-forming agent components and reveals, in this connection, an elevated reaction activity. They occur with a catalytic participation of zinc chloride under the pressure of 10 . . . 12 MPa, at the temperature 390 . . . 425° C. Fresh cold hydrogen is additionally fed to said reactor 22 through several connecting pipes located at different levels for a fine adjustment of thermal regime of hydrogenation.

[0038] The mixture leaving hydrogenation reactor 22 passes through hot separator 23, where vapor/gaseous phase is separated from liquid reaction products carrying out solid phase residues. Vapor/gaseous mixture is removed from hot separator 23 from above and then is fed for heat recuperation into heat exchangers 18 and 20. After that, it is fed to condenser 24 for cooling and to cold separator 25—for subsequent separation, with a release of circulation gas returned into the process and light condensed hydrogenate. Meanwhile, a mixture of high-boiling liquid hydrogenation products contaminated with solid particles residues is carried out from the lower part of hot separator 23 into refrigerator 28, throttled in valve 29 and, after the final pressure release in expander 30, is accumulated in thickener 31. Thickened sludge is fed from the latter to a batch centrifugal filter 32 for deep squeezing from the infiltrating liquid phase. The cake squeezed on centrifuge 32 is cleaned on it from the impregnating residues of heavy hydrogenate with an organic solvent, for instance, light petroleum (a mixture of light hydrocarbons, predominantly paraffin hydrocarbons with 5 and 6 carbon atoms), passes through drier 33 and is reloaded to belt conveyor 34 transporting this industrial waste into mixer 35 for mixing with wet heavy product removed from the initial power-generating raw material already at the first stage of its milling. Meanwhile, the discharge of thickener 31 is combined with the fugate of centrifuge 32, and the resulting heavy hydrogenate, which is completely free from solid particles, is fed to further processing realized within the system of synthetic petroleum processing.

[0039] Organic diluent vapors lost by desiccated cake released from drier 33 are diverted for mixing with the gaseous phase leaving cold separator 25. Then, jointly with the total circulating gas flow, they are fed to the suck-in of compressor 19 for adiabatic compression together with the initial

hydrogen. The complete expansion of gas leaving cold separator **25** is accomplished in turboexpander **36**.

[0040] Light hydrogenate separated from non-condensing gases consisting of low-boiling fractions of synthetic petroleum, which is carried out from the lower part of cold separator **25**, is throttled in valve **26**, passed through expander **27** and then fed, together with the heavy hydrogenate, to a subsequent distillation for reprocessing into various kinds of petrol and other synthetic oil products.

[0041] The wet mixture of solid industrial wastes obtained in mixer **35** is cleaned with hot water from zinc chloride residues carried out of the technological process by means of countercurrent multi-stage washing on band vacuum filter **37** and withdrawn from the production cycle to storage or utilization.

[0042] Washing water resulting from the countercurrent washing process, which represents a dilute aqueous solution of zinc chloride, is fed from sump **38** by pump **39** into evaporator system **40** for evaporation. Water steam lost by such solution in the course of evaporation is condensed in condenser **41**, and the obtained fresh condensate is returned to the first step of countercurrent washing of solid industrial waste on band vacuum filter **37**. Aqueous solution of zinc chloride in water recovered, in this way, to the required initial density of 1.417 g/cm^3 is continuously discharged from its internal circulation circuit into the starting point of the process by pump **41** with its simultaneous cooling in heat exchanger **42**, and fed to the input of ball mill **1**.

[0043] Thus, the production cycle of zinc chloride used as a mineral salt catalyzing the process of hydrogenation is completely closed, and at adequate production standards, the former is not practically consumed.

Example 1

[0044] Freshly-mined coarse coal (ash level 8.9%, volatile substances content 45.2%, sulfur content 0.8%) is charged into a steel mortar vessel filled with 40% aqueous zinc chloride solution with the density 1.417 g/cm^3 . Then the cleanest coal particles in the initial raw material, which are not contaminated with waste rock, remain afloat in such liquid medium, while the remaining rock mass sinks to the bottom of said vessel. The sunk material is manually ground into smaller fragments using a steel tamper. Meanwhile, bubbles of methane insoluble in said aqueous medium continuously bubble through the zinc chloride solution layer, and as coal is liberated from its accretions with waste rock, pure particles of this inflammable mineral automatically slip from under the impacts with said milling body and float to the liquid surface, thus avoiding additional irrational energy consumption for their further destruction. Respectively, less and less solid material remains on the bottom of the mortar vessel, and each time the efforts aimed at its destruction are lightened.

[0045] When the emersion of inflammable minerals with the density below 1.417 g/cm^3 to the surface of the liquid medium is ceased, the contents of the mortar vessel are poured over into a container with an agitator. By means of a centrifugal pump, roiled suspension from said container is passed through a hydrocyclone, where coal is more rapidly separated from the waste rock. Heavy product of the first milling stage representing a mixture of waste-rock minerals, which leaves the conical part of hydrocyclone, is dehydrated on a nutsch filter. Meanwhile, a suspension of the light product of the first milling stage collected into a bucket at the outlet of the cylindrical part of the hydrocyclone is heated to

100° C. and fed to a thermally insulated ball mill rotating on two rollers of a roll-table, which is equipped with methane-removing manifold with end-face sealing and flooded with hot (100° C.) 40% aqueous zinc chloride solution. Thus, at the second stage of wet milling, even more purified coal is released from its accretions with waste rock (which is also accompanied by methane emission), since only coal that practically does not contain any mineral impurities is capable of floating to the surface of liquid medium contained in thermally insulated mill with decreasing density of said liquid phase.

[0046] Then the hot suspension of finely ground coal poured out of the thermally insulated ball mill is separated on a fast (4000 rpm) sedimentation centrifuge. Sediment pressed to the internal surface of the rotating rotor by centrifugal forces, which is discharged from the centrifuge by means of a rotating screw, is cooled and returned to the steel mortar vessel for mixing with the next batch of the initial rock mass. Meanwhile, the fugate representing a suspension of coal finely cleaned from mineral impurities in aqueous solution of zinc chloride is separated on a centrifugal filter.

[0047] Before the wet cake deeply squeezed on the centrifugal filter gets cooled, 108 grams of such material are taken, which corresponds to net 100 grams of dry demineralized (i.e. containing no ZnCl_2) solid coaly substance, and mixed with 200 ml of hot (160° C.) paste-forming agent representing a mixture (90:10 by volume) of tetraline with anthracene oil. In the course of said mixing, water vapor sucked out by a vacuum pump is practically totally completed from the prepared paste.

[0048] Then 100 ml of isopropyl alcohol are added to the prepared paste cooled down to 80° C. , and the obtained mixture is diluted with such diluent in a fast mixer.

[0049] Then the paste diluted with isopropyl alcohol is fed to a centrifugal disperser, where the solid phase of this system is additionally disintegrated to a colloidal size. After that, the disperser content is reloaded into a steel autoclave installed afterwards in a muffle furnace with a tangential input of compressed hydrogen (for more efficient mixing), and coal hydrogenation is realized in this medium. For this purpose, the mixture in the autoclave is gradually heated from 80° C. and initial hydrogen pressure of 2 MPa to the temperature 405° C. and the pressure of 11 MPa.

[0050] After two hours of hydrogenation, the autoclave is taken out of the muffle furnace, cooled, depressurized, and the synthetic oil formed therein is separated from solid particles in a centrifugal filter. Solid sediment extracted from said liquid is cleaned from synthetic oil residues by light petroleum, dried with hot air and then rinsed with plenty of warm distilled water, dried and weighed. The obtained dry residue weighs 2.74 gram. This material is mixed with cake obtained by squeezing the heavy product of the first milling stage on a nutsch filter. The obtained waste mixture of solid minerals is washed on the nutsch filter with hot fresh water and withdrawn from the process as waste. The remaining washing water is mixed with the discharge of washing the solid residue extracted from the synthetic oil and evaporated to the density of aqueous zinc chloride solution equal to its initial value 1.417 g/cm^3 .

[0051] Since the autoclave was charged with 100 grams of pure original raw material forming a part of the paste prepared

according to the described procedure, the efficiency of liquefaction of such material fed for hydrogenation amounts to 97.26%.

Example 2

[0052] The same freshly-mined coal containing 8.9% of non-inflammable mineral impurities, 45.2% of volatile substances and 0.8% of sulfur is roiled in 40% water-mineral solution prepared by dissolving a mixture of dry nine-water sulfate of ferric iron $\text{Fe}_2(\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}$ with ammonium par-amolybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ taken in the ratio 92:8 (by weight) in water and delivered in such medium (with the density 1.403 g/cm^3) to the liquefaction place.

[0053] After the accomplishment of a complete cycle of this raw material processing in aqueous solution of a mixture of said mineral salts according to the method of example 1, the weight of dry clean residue extracted from the synthetic oil obtained in the autoclave is 1.39 gram. Thus, in this case, the efficiency of liquefaction of raw material impregnated before its hydrogenation in the autoclave with a mixture of iron sulfate and ammonium molybdate reaches 98.61%.

Example 3

[0054] A mixed water-mineral medium is prepared by dissolving a dry mixture of ammonium tetramolybdate $(\text{NH}_4)_2 \cdot 4\text{MoO}_3 \cdot 2\text{H}_2\text{O}$ with iron vitriol $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ taken in the weight ratio 84:16 in water. The solution density is brought to 1.296 g/cm^3 by water saturation with said composition of mineral salts at room temperature. After that, freshly-mined coal with the composition as in example 1 is charged into this solution, and liquefaction is performed according to the method described above in example 1.

[0055] As a result, dry residue output remaining non-liquefied in the synthetic oil decreases to 1.18 gram. Hence, the efficiency of liquefaction of the original raw material in case of its processing using ammonia salt of molybdenum acid increases up to 98.82%.

Example 4

[0056] The initial raw material for liquefaction and experimental conditions are the same as in Example 1. However, 40% solution of ferric iron sulfate $\text{Fe}_2(\text{SO}_4)_3$ with the density of 1.448 g/cm^3 is used for coal milling. Here the dry clean residue output that has not transformed into synthetic oil amounts to 3.12 grams. Hence, the efficiency of coal processing in this case equals 96.88%.

Example 5

[0057] The initial raw material for liquefaction and experimental conditions are the same as in Example 1. However, 40% solution of divalent tin chloride SnCl_2 with the density of 1.414 g/cm^3 is used as a medium for wet coal milling. As a result of such processing, dry residue remaining non-liquefied is 2.89 grams. Hence, the efficiency of coal liquefaction in this case equals 97.11%.

Example 6

[0058] Raw material for liquefaction and experimental conditions are the same as in Example 1. The solution for wet coal grinding with the density 1.392 g/cm^3 is prepared by

dissolving a mixture of nine-water ferric iron sulfate $\text{Fe}_2(\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}$ with five-water quadrivalent tin chloride $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ taken in the weight ratio 75:25 in water. The weight of the residue remaining non-liquefied is 2.41 grams; hence, the efficiency of coal liquefaction in this case equals 97.59%.

BEST MODE

Mode for Invention

INDUSTRIAL APPLICABILITY

[0059] Thus, the use of the method of the invention for the production of synthetic liquid fuel, undoubtedly, contributes to an essential increase in the technico-economical efficiency of the processing of fossil power-generating raw material, especially that with elevated mineral impurities content, and, at the same time, ensures its complex utilization and increase in liquefaction efficiency, and considerably weakens the harmful impact of the synthetic liquid fuel production on the natural environment.

Sequence List Text

1. A method of liquefaction of inflammable minerals involving a preliminary grinding of the initial raw material with further mixing of the ground material with paste-forming agent, hydrogenation of its carbon-containing components by attaching hydrogen in the presence of a catalyst and separation of liquid products from non-liquefied solid residue, wherein to ensure a complex utilization of the initial raw material, to increase liquid products output, to decrease the power consumption of the process and to weaken the harmful impact of synthetic liquid fuel production on the natural environment, the preliminary grinding of the initial raw material is carried out in two stages with simultaneous capping of methane released from the destroyed material, in an aqueous solution of mineral salts catalyzing hydrogenation, said solution being saturated to the density intermediate between those of inflammable mineral and waste rock, and separate extraction of light and heavy products after every grinding stage, wherein the suspension of the light product of the first milling stage is heated before the second milling stage, and after the latter, the light product of the second milling stage is dehydrated, mixed with paste-forming agent, diluted with diluent, ground to the colloidal size and fed to hydrogenation; non-liquefied hydrogenation residue is washed with an organic diluent, dried and mixed with dehydrated heavy product of the first milling stage, and the produced mixture is washed with water, whereas the remaining washing water is evaporated to the initial density of the original solution and returned to the first milling stage, while the heavy product of the second milling is squeezed from excessive liquid phase, cooled and returned to the starting point of the process.

2. A method according to claim 1, wherein both individual inorganic compounds and their mixtures forming aqueous solutions with the density sufficient for floating of inflammable components of the processed raw material, such as zinc or tin chlorides, iron sulfates and ammonium molybdates, are used as mineral salts catalyzing the hydrogenation process, whereas organic compounds of the aromatic series representing donors of atomic hydrogen, such as tetraline, methyl naphthalene, cresol and anthracene oil components are used as paste-forming agents.

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