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[54] PROCESS OF PRODUCING
HYDROCARBONS FROM COAL

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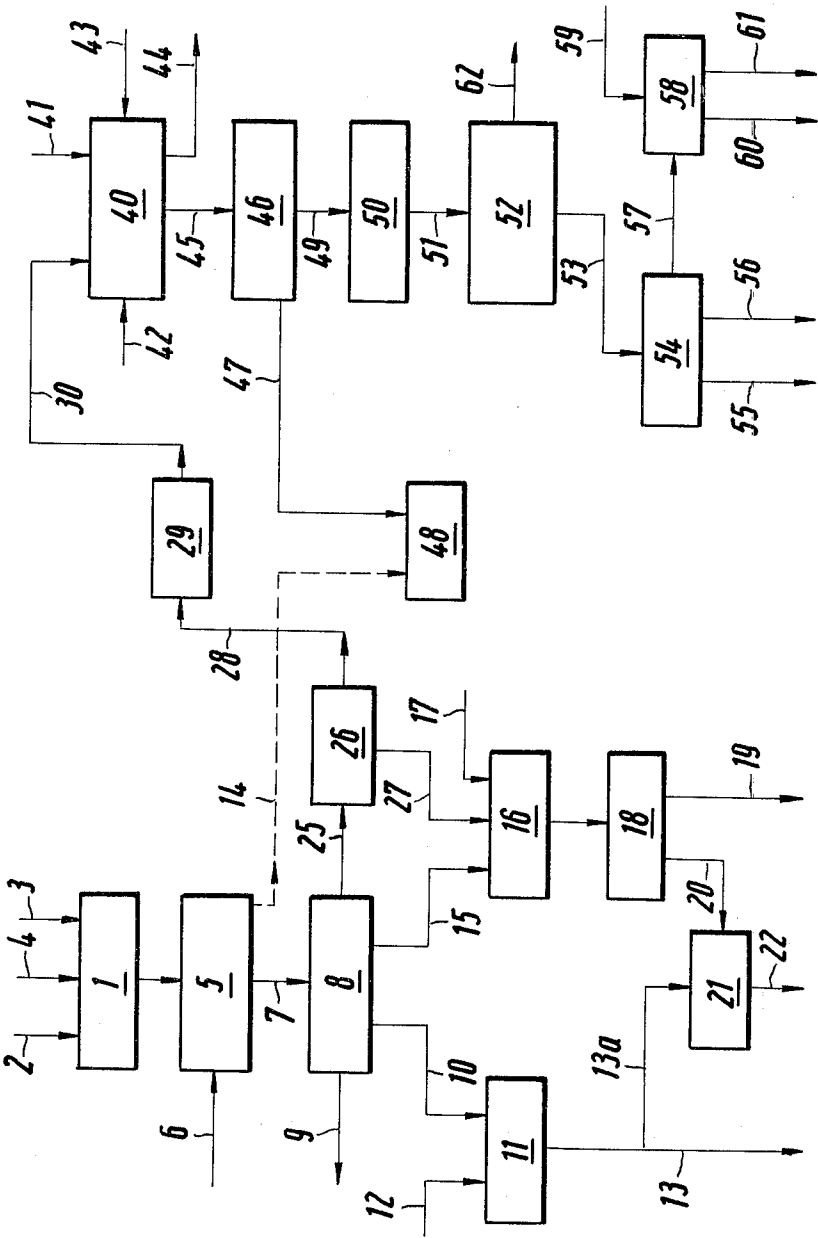
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[57] ABSTRACT

An improvement in a process for producing hydroca
bons which boil in the gasoline or diesel fuel range fro
coal where the coal is reduced to a particle size belo
2 mm, mixed with oil to form a pumpable pulp which
contacted with hydrogen in a hydrogenation zone r
sulting in a liquid fraction and a high melting residu
fraction is disclosed. The invention resides in removin
the high melting residue to a gasifying vessel to whic
is introduced granular coal of particle size of 3-50 mm
To the gasifying vessel is added a gasifying agent, e.g
water vapor alone or in admixture with oxygen whic
flows in countercurrent to the direction of the granula
coal. As a result of the process, a gasification product
obtained comprising hydrogen and carbon monoxide.

8 Claims, 1 Drawing Figure



PROCESS OF PRODUCING HYDROCARBONS FROM COAL

This is a continuation of application Ser. No. 931,349 filed Aug. 7, 1978, now abandoned.

This invention relates to a process of producing hydrocarbons which boil in the gasoline and diesel fuel ranges, from coal, in which the coal is reduced in size and in a particle size below 2 mm is mixed with oil to form a pumpable pulp and in a hydrogenating zone is reacted in the presence of hydrogen under a pressure of 100 to 400 bars and temperatures of 300° to 500° C., and the product formed in the hydrogenating zone is separated into fractions, which include liquid components, which consist mainly of hydrocarbons having 4 to 30 carbon atoms per molecule and are processed to form motor fuels, if desired, and a high-melting residue, which contains pitch and solids and is withdrawn.

It is known to hydrogenate various kinds of coal, including brown coal, and to process the hydrogenation products to form motor fuel. A comprehensive treatment of this technology is contained in the book by H. Krönig: "Die katalytische Druckhydrierung von Kohlen, Teeren und Mineral-ölen" (1950), Springer-Verlag, Berlin-Göttingen-Heidelberg. More recent developments have been described in U.S. Pat. Nos. 3,745,108; 3,660,269; and 3,635,814. In the known processes a catalyst is used or is not used in the hydrogenating zone. Processes using no catalyst utilize in most cases the catalytic activity of metallic constituents of the coal. For a hydrogenating of coal, it is important that the coal fed to the hydrogenating zone has been disintegrated to a very small particle size so that large surface areas are presented to the hydrogen. Any catalyst must also be used in a small particle size and mixed with the coal.

The product of the hydrogenating zone contains gaseous, liquid and solid constituents, which are usually subjected first to fractional condensation, which may be succeeded by a distillation. A high-melting residue, which contains pitch and solids, is thus formed and is not suitable for re-hydrogenation. On the other hand, the hydrocarbon-containing liquid can be processed in a hydrocracker and/or hydrotreater by known methods in the presence of hydrogen and possibly catalysts to produce motor fuel.

In this specification the term motor fuel includes mainly regular-grade gasoline (low-octane petrol), premium-grade gasoline (high-octane petrol), diesel fuel, and kerosene.

It is an object of the invention to convert available coal as completely as possible to valuable hydrocarbons. This is accomplished in that the residue together with granular coal having a particle size of 3 to 50 mm is gasified in a fixed bed under pressures of 10 to 100 bars by a treatment with gasifying agents flowing in a countercurrent to the coal, the gasification product gas, which contains hydrogen and carbon monoxide, is cooled, tar and oil are withdrawn as condensate, and the gas is purified.

In the known hydrogenating processes it has always been difficult to dispose of the high-melting residue. It has already been considered to process that residue by coking, dry distillation or solvent extraction. The process according to the invention, in which the residue is gasified together with granular coal, is much simpler than the processes suggested before.

After purification, the gasification product can be used for various purposes. It can be processed further to produce a synthesis gas from which a gas having a high calorific value can be produced, e.g., by methanation. The purified gas may also be shift-converted and used as a source of hydrogen for hydrogenating the coal.

The high-melting residue may be subjected to gasification in a liquid state together with the coal. It is particularly advantageous, however, to granulate the residue and to subject a residue having particle sizes in the range from 3 to 50 mm to gasification. Surprisingly it has been found that the granules are not melted, would be expected, but retain their shape when they are heated during the gasification in the fixed bed. If the residue is granulated it is not necessary to find out how a solidification of the residue in the hydrogenating plant can be avoided in a reliable manner.

The process according to the invention may be applied to various kinds of coal, including pit coal and brown coal. In a preferred embodiment of the process the product gas obtained by the gasification of granular coal and high-melting residue is purified and then fed to a Fischer-Tropsch synthesis section for producing hydrocarbons which at least in part contain 4 to 30 carbon atoms per molecule. The production of hydrocarbons along two interconnected routes comprising the hydrogenation of coal and the Fischer-Tropsch synthesis constitutes a versatile overall process. If one of the two routes fails entirely or in part for certain time, the production can be continued on the route which is still intact. For instance, when the coal-gasifying section must be shut down, the operation of the coal hydrogenating section may be continued. In that case the high-melting residue is preferably dumped in a granulated state. A further advantage resides in that a high ash coal can be used, which is fed to the coal-gasifying section, whereas low-ash coal is desired for the hydrogenating section.

By the Fischer-Tropsch synthesis, synthesis gas containing mainly CO and H₂ is reacted at pressures of 5 to 30 bars and temperatures in the range from 150° to 350° C. to produce hydrocarbons. The synthesis is suitably effected in the presence of catalysts which contain, e.g., cobalt, manganese or iron as active component or activators. The Fischer-Tropsch synthesis is known in itself and has been described, e.g., in the book by Storck, Golumbic, Anderson: "The Fischer-Tropsch and Related Synthesis", (1951) John Wiley and Sons, New York.

The Lurgi process may be used for the gasification of coal. Details of that process have been explained in U.S. Pat. Nos. 2,667,409; 3,930,811; 3,937,620; 4,032,030; and 4,033,730. The gasification of granular coal provides for a processing of coal having a suitable particle size and for a processing of the coal dust, which is always present and is subjected to hydrogenation.

In addition to the ash contained in the coal, the process according to the invention does not result in solid residues because all residues formed in the coal hydrogenating section can be fed to the gasifying section. The primary product of the Fischer-Tropsch synthesis can readily be converted to motor fuel. For the production of high-grade motor fuel from the product of the hydrogenation of coal, said product must be catalytically refined and at least part of the refined products must be cracked in the presence of hydrogen and catalysts. These treatments can be effected in accordance with processes which are known in refinery technology.

ogy. The refining stages may also be fed with tar and which become available as condensates when the gas produced by the gasification of coal is cooled in 35.

Tail gases which become available in the hydrogenation section and/or the Fischer-Tropsch synthesis section can be converted to a high-hydrogen gas, from which the hydrogen is separated and fed to the hydrogenating stage. For instance, the tail gas may be catalytically or noncatalytically reformed by a treatment with hydrogen so that the product gas contains mainly CO and H₂. This gas can be shift-converted by a treatment with water vapor so that the CO content of the gas is converted to H₂ and CO₂ and CO₂ can then be scrubbed. Alternatively, H₂S, CO₂, and NH₃ may be removed from the tail gases and the latter can then be separated at low temperatures to recover the hydrogen for the hydrogenating. C₃ and C₄ hydrocarbons become available at the same time as liquefied gas.

Further details of the process will now be explained with reference to the drawing, which is a flow scheme. The stage 1 for pretreating the coal is fed by a conveyor 2 with pre-crushed coal having substantially a particle size below 2 mm and is fed through conduit 3 with oil having a boiling range of about 250° to 450° C. In the pretreating stage 1, the coal is further disintegrated and intensely mixed with oil. For this reason the treating stage is suitably fed also with fine-grained catalyst material through conduit 4. The catalyst rate is about 10% by weight of the coal feed rate. The catalyst accelerates the hydrogenation in the hydrogenating zone 5.

A pumpable pulp consisting of coal, oil and catalyst leaves the pretreating stage 1 and is fed to the hydrogenating zone 5. In addition to oil, the pump contains about 30 to 60% solids. Hydrogen is fed to the hydrogenating zone 5 through conduit 6. The pressure in the hydrogenating zone is 100 to 400 bars, preferably 120 to 150 bars, and the temperatures are in the range from 150° to 500° C. and preferably at 400° to 475° C. The major reactants consisting of coal, hydrogen catalyst material are intensely contacted with each other in the hydrogenating zone 5.

The resulting main product is transferred from the hydrogenating zone 5 through a conduit 7 to a separator in which the liquid and vapor phases are separated at temperatures of about 400° to 450° C. The liquefiable hydrocarbons are separated from the gas stream by fractional condensation. Uncondensed tail gas is withdrawn in conduit 9. The condensable components separated by distillation in the separator and a fraction to boiling in the range of 30° to 250° C., preferably to 200° C., under standard pressure, is transferred in conduit 10 to a hydrotreater 11, which is fed also with hydrogen from conduit 12 and in which a catalytic conversion and desulfurization are effected by processes known per se. As a result, gasoline which is suitable as a motor fuel becomes available in conduit 13. To raise its octane rating, all or part of that gasoline may be fed in conduit 13a to an aromatizing stage 21, the product of which becomes available in conduit 22.

A higher-boiling fraction, which has a boiling point at least 230° C. at normal pressure, is withdrawn from separator 8 through conduit 15 and is cracked in a hydrocracker 16 in known manner in contact with hydrogen from conduit 17. The cracking is preferably accelerated by catalysts. The product formed in the hydrocracker 16 is separated in a distillation stage 18

and the heavier fraction is withdrawn in conduit 19 as diesel fuel. The lighter fraction is fed in conduit 20 to the aromatizing stage 21. Gasoline is withdrawn in conduit 22.

In the separator 8, hot sludge becomes available at temperatures of about 400° to 450° C. That sludge contains unreacted coal, high-boiling oil and any catalyst material used for the hydrogenation. That sludge is fed through conduit 25 to a vacuum distillation stage 26 and is further separated therein. The lower-boiling fraction is fed in conduit 27 also to the hydrocracker 16. A high-melting residue is left, which contains pitch and solids and is fed in conduit 28 to a granulator 29. The granulated residue is fed by a conveyor 30 to a pressure gasifier 40, which is also fed by the conveyor 41 with granular coal having a particle size in the range from 3 to 50 mm. In the gasifier 40, the coal to be gasified is in a fixed bed and flown through in a countercurrent by gasifying agents fed from below. The gasifying agents consist of water vapor fed through conduit 42 and oxygen fed through conduit 43. In addition to these gasifying agents, CO₂ may be used. This is not shown on the drawing.

The coal gasifier 40 operates under pressures in the range from 10 to 100 bars, preferably 15 to 50 bars. Ash is withdrawn from the pressure gasifier through conduit 44. That ash contains also the catalyst which has been used in the hydrogenating zone 5. Raw product gas at a temperature of about 300° to 800° C. leaves the gasifier 40 through conduit 45. The raw gas is first cooled in a cooler 46, in which it is preferably prescrubbed at the same time with circulating condensate. Surplus condensate is fed in conduit 47 contains tar and oil.

The cooled gas is fed in conduit 49 to a fine purification stage 50, in which sulfur compounds as well as NH₃ are removed so that the gas has the purity required for the synthesis. The fine purification may be effected, e.g., by the known Rectisol process, in which liquid methanol is used as absorbent. Depending on its composition, the gas is suitably shift-converted before or after the fine purification in stage 50. By this shift conversion, the CO and H₂ contents of the gas are controlled as desired. Because such shift conversion is not always required, it has not been shown on the drawing for the sake of clearness.

The gas which leaves the fine purification stage 50 consists mainly of CO, H₂, and CH₄ and is fed in conduit 51 to the Fischer-Tropsch synthesis section 52, in which hydrocarbons are produced at a pressure in the range of about 5 to 30 bars and at temperatures of 150° to 350° C., preferably in the presence of catalysts. The catalysts may contain, e.g., cobalt manganese or iron as active components. The catalyst in the synthesis section 52 is preferably contained in a fixed bed and the reaction is isothermal and adiabatic. The primary product of the synthesis is separated from the gas stream by a heat exchange and cooling and flows in conduit 53 to a separator 54. Gasoline is withdrawn in conduit 55 and diesel fuel in conduit 56. A higher-boiling residue flows in conduit 57 to a wax cracker 58, in which additional gasoline (in conduit 60) and diesel fuel (in conduit 61) are produced by a treatment with hydrogen in conduit 59. This gasoline and diesel fuel are suitably refined to remove oxygen-containing compounds. This is not shown on the drawing.

Tail gases from the Fischer-Tropsch synthesis section 52 are fed in conduit 62 to a suitable use. Because tail gases which contain hydrocarbons become available

elsewhere too, it is economical to process such gases. This may be accomplished by a separation at low temperatures so that the synthesis gas components CO and H₂ are separated and recycled to the synthesis section 52. Alternatively, the tail gases in conduit 52 and the tail gases from the hydrogenating section may be cracked jointly to form a high-hydrogen gas.

As has been explained before, condensate which contains oil and tar is fed in conduit 47 to a tar separator 48 and is separated therein by gravity. That tar separator 48 may also be fed in the conduit 14, represented by a dotted line, with tar products from the hydrogenating zone 5. For the sake of clearness, the disposal of the fractions which become available in the tar separator has not been shown. For instance, the solids-containing heavy tar as well as distillates and condensates boiling above 250° C. may be fed through conduit 3 to the pretreating stage 1. A fraction boiling in the gasoline range may be fed to the hydrotreater 11. Higher-boiling condensates or distillates, excepting solids-containing condensates or residual oils, may be fed to the hydrocracker 16. The dust-containing heaviest fraction from the tar separator 48 may be recycled to the pressure gasifier 40.

EXAMPLE 1

Pit coal is processed by a method as shown on the drawing. The raw coal fed to the hydrogenating section has an ash content of 4.65% by weight and a water content of 2.78% by weight. The pure coal, which is assumed to be free from water and ash, has the following elementary analysis in % by weight:

C —80.44
H —4.76
O —12.37
N —1.27
S —1.16

To facilitate the explanation, all statements made hereinafter refer to pure coal. 1000 tons having a particle size below 2 mm are mixed to form a pulp in the pretreating stage 1 with 1500 tons of a hydrogenating oil fraction, which boils between 250° and 450° C. In the pretreating stage 1 the coal is further reduced in size to have a particle size below 500 microns and at least 50% of particles below 100 microns 20 tons of catalyst having the same particle size are fed to the pretreating stage 1 in the form of red mud obtained by the processing of bauxite. That red mud consists mainly of ferric hydroxide. The pulp consisting of coal, oil and catalyst is fed to the hydrogenating zone 5 and pressurized to 350 bars and heated to the reaction temperature of 460°C. The pulp is heated in the presence of hydrogen, which is supplied as fresh gas to the hydrogenating zone 5 in an amount of 65 tons through conduit 6.

55 tons of H₂ are chemically combined in the hydrogenating zone 5. 10 tons of H₂ are included in the tail gas which is withdrawn in conduit 9 from the separator and contains also the noncondensable low-boiling hydrocarbons which have been formed by the hydrogenation as well as CO, CO₂, H₂S, and NH₃.

The first stage of the separator 8 is operated at a temperature of 450° C. and at the operating pressure of the hydrogenating zone, amounting to 350 bars. In that first stage, 480 tons of a sludge are formed, which consists of nonevaporated hydrocarbons, unreacted coal, ash, and catalyst. This sludge is pressure-relieved in several steps and then fed to a vacuum distillation stage 26, in which 260 tons of distillate and 220 tons of a

high-melting residue are formed. The latter consists 50 tons of ash, 50 tons of unreacted coal, 20 tons catalyst and 100 tons of compounds of carbon, hydrogen, oxygen, sulfur and nitrogen. The distillate is fed directly to the pretreating stage 1 through conduit 5 After the cooling in separator 3, 1740 tons of liquid products become available, which are further separated by distillation, 1240 tons of separated heavy oils are also fed to the pretreating plant 1 in conduit 3. 530 tons liquid products having a final boiling point of 440° also become available in the distillation stage and are fed in part to the hydrotreater 11 and in part to the hydrocracker 60.

The high-melting residue from the vacuum distillation stage 26 has a melting point above 100° C. and is fed to a granulator 29, in which the molten material is extruded onto a water bath through nozzles which are about 10 mm in diameter. The resulting extrusions are then crushed to a length of about 5 to 10 mm. The molten granules are mixed with coal in a ratio of 220 metric tons of granules to 897 tons of coal, calculated as pure coal. The mixture is fed to a Lurgi system pressure gasifier 40. 125 metric tons of ash, which is in lump form and contains 20 tons of hydrogenation catalyst, are withdrawn from the gasifier 40. This amount of catalyst is discharged.

77 tons of tar and oil are condensed as the raw gas from the pressure gasifier is cooled. The dust-containing heavy tar fraction is also fed to the pretreating stage through conduit 3. Medium-range distillate from the separator 48 is fed to the hydrocracker 16. The low-boiling fraction is refined in the hydrotreater 11. 43 tons of gas naphtha become available as a result of the purification of the gas and are also treated in the hydrotreater 11. The hydro-refined product from the hydrotreater 11 and the gasoline which has been produced by the hydrocracker 16 and the succeeding distillation stage 18 is fed to the aromating stage 21 for an increase of its octane rating. Hydrogen is also released in the aromating stage.

284 tons of motor gasoline and 314 tons of diesel fuel are produced by the hydrogenation of coal and the processing of the products which become available during the gasification (without the Fischer-Tropsch synthesis).

Any tail gases from the parts 5, 8, 9, 11, 16, and 18 of the plant are freed from CO₂ and desulfurized and together with the sulfur-free tail gas from aromating stage 21 are separated into their components at low temperature and under pressure. Separated hydrogen is recycled to the stages in which it is used. Methane and ethane are used in known manner to produce hydrogen. Propane can be delivered as liquefied gas. Butane is used to adjust the vapor pressure of the gasoline product.

The synthesis gas from the fine purification stage consists virtually only of CO and H₂ and some methane. In a Fischer-Tropsch synthesis section 52, the synthesis gas is reacted to form hydrocarbons at temperatures 220° C. and a pressure of 30 bars at an iron catalyst forming a multi-stage fixed bed. The conversion amounts to 90%, based on CO and H₂. The reaction product is condensed from the gas stream in that the latter is first cooled to ambient temperature. Naphtha is removed by a cooling below 0° C. at the operating pressure of the synthesis section. In a separator 54, gasoline fraction and a diesel fuel fraction are distilled from the condensates. Before these products are del

ed as motor fuels, they are slightly refined to increase the octane rating of the gasoline and to remove oxygen-containing components.

The distillation residue from the separator 54 consists of a mix of waxlike hydrocarbons and is fed to the wax cracker 58 and catalytically cracked therein in the presence of hydrogen to produce a larger amount of diesel fuel and a smaller amount of gasoline.

A total of 142 tons of motor gasoline and 72 tons of diesel fuel are produced in the Fischer-Tropsch synthesis section.

The tail gas from the synthesis section becomes available in the conduit 62 and together with the tail gas from the hydrocracker 16 is further cooled and separated under pressure. A mixture of the synthesis gas components CO and H₂ is recycled to the synthesis section 52. Methane and ethane are reformed to produce hydrogen. The C₃ fraction is delivered as liquefied gas after its olefins have been hydrogenated. The C₄ fraction is admixed to the gasoline product.

A total of 1897 tons of pure coal, 426 tons of motor gasoline and 386 tons of diesel fuel are produced in the hydrogenation section and the Fischer-Tropsch synthesis section. In addition, 110 tons of propane are delivered as liquefied gas. The yield corresponds to a thermal efficiency of 65% if the consumption of coal required to produce energy is neglected and sulfur, ammonia, and C₁ to C₄ alcohols which become available as by-products of the Fischer-Tropsch synthesis are not taken into account.

Example 2

The object underlying this example is to produce twice as much Fischer-Tropsch product as in Example 1 and to change also the ratio of diesel fuel to gasoline. The process is similar to that of Example 1.

1000 tons of pure coal are processed too, but the gasifier 40 is fed with 1794 tons rather than 897 tons of pure coal and with 220 tons of high-melting residue. As a result, additional 64 tons of tar are fed to the hydrocracker 16 and additional 16 tons of gas naphtha are fed to the hydrotreater 11 so that the output of the hydrogenation section is increased to 284 tons of motor gasoline and 364 tons of diesel oil.

The quantity of primary product of the Fischer-Tropsch synthesis of Example 1 is almost doubled.

Because more diesel fuel is desired, the wax cracker 58 is operated at somewhat lower temperatures so that more liquid products are produced, namely, a total of 18 tons of motor gasoline and 217 tons of diesel fuel. The propane production is increased in 150 tons.

From the larger Fischer-Tropsch synthesis section, twice as much tail gas becomes available for utilization. Because the amount of hydrogen consumed for hydrogenation is only slightly changed, the surplus tail gas can be used for power production, e.g., as gas for supply over long distances or in a gas turbine.

EXAMPLE 3

In a process which is similar to that of Example 1 the Fischer-Tropsch synthesis is omitted. As has been explained in Example 1, about 70 to 75 tons of hydrogen are required to hydrogenate 1000 tons of pure coal. Part of that hydrogen is produced from about 60 tons of ethane produced by the hydrogenation. A high-pressure hydrogen gas is produced from said methane by the steam reforming process. 220 tons of high-melting residue and 200 tons of granular coal are fed to the

gasifier 40. The gasification product gas is purified and shift-converted and then separated at low temperature to provide a high-hydrogen gas, which contains at least 95% H₂ by volume and can also be used for hydrogenation.

Condensate from the gasification product gas is fed to the tar separator 48, 43 tons of tar are separated in the separator 48 and fed to the hydrocracker 16. 23 tons of gas naphtha are also recovered in the tar separator 48 and are fed to the hydrotreater 11. In this process, a total of 264 tons of motor gasoline and 284 diesel fuel are produced.

What is claimed is:

1. In a process for producing hydrocarbons which boil in the gasoline and diesel fuel ranges from coal, wherein a first portion of coal is reduced to a particle size below 2 mm, mixed with oil to form a pumpable pulp, the pumpable pulp is contacted with hydrogen in a hydrogenating zone under a pressure of 100-400 bars at a temperature of 300°-500° C., the resultant hydrogenated product separated into fractions including a liquid fraction comprising mainly hydrocarbons of 4 to 30 carbon atoms per molecule and a high melting residue fraction containing pitch and solids,

A. a second portion of coal having a particular size of 3 to 50 mm is fed to a gasifying vessel and is gasified in a fixed bed under pressure of 10 to 100 bars by contacting the composition with at least one gasifying agent selected from the group consisting of water vapor, oxygen, and carbon dioxide, said gasifying agent passing in countercurrent flow to the direction of movement of said second portion of coal,

B. withdrawing gasification product gas comprising hydrogen and carbon monoxide from said vessel and cooling the same and withdrawing therefrom tar and oil as condensate, the gasification product gas is fed after purification to a Fischer-Tropsch synthesis section for production of primary product hydrocarbons which at least in part contain 4 to 30 carbon atoms per molecule, said primary product hydrocarbons being at least partially converted to motor fuel,

the improvement which comprises withdrawing said high melting residue fraction, granulating said residue fraction to a particle size of 3 to 50 mm and feeding it into said gasifying vessel for gasification together with said second portion of coal.

2. A process according to claim 1 wherein following step B the product gas which has been freed of tar and oil is further purified.

3. A process according to claim 1 wherein the tar and oil removed are used at least in part to form said pumpable pulp.

4. A process according to claim 1 wherein the liquid fraction comprising hydrocarbons of 4 to 30 carbon atoms per molecule is thereafter treated with hydrogen in the presence of a catalyst to convert the same to a motor fuel component.

5. A process according to claim 1 wherein the tar products from said hydrogenating zone are admixed with oil and tar condensate obtained as a result of cooling said gasification product and the resulting composition is subjected to cracking.

6. A process according to claim 1 wherein the residual gases from the hydrogenating section and/or the Fischer-Tropsch synthesis section are withdrawn and

converted to high hydrogen gas and the hydrogen is separated and fed to the hydrogenating section.

7. A process according to claim 1 wherein the pressure maintained within the gasifying vessel is 15-50 bars.

8. A process according to claim 1 wherein the condi-

tions within the gasifying vessel are maintained such that the gasification product gas withdrawn from the gasifying vessel is at a temperature, prior to the cooling of step B, of 300°-800° C.

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