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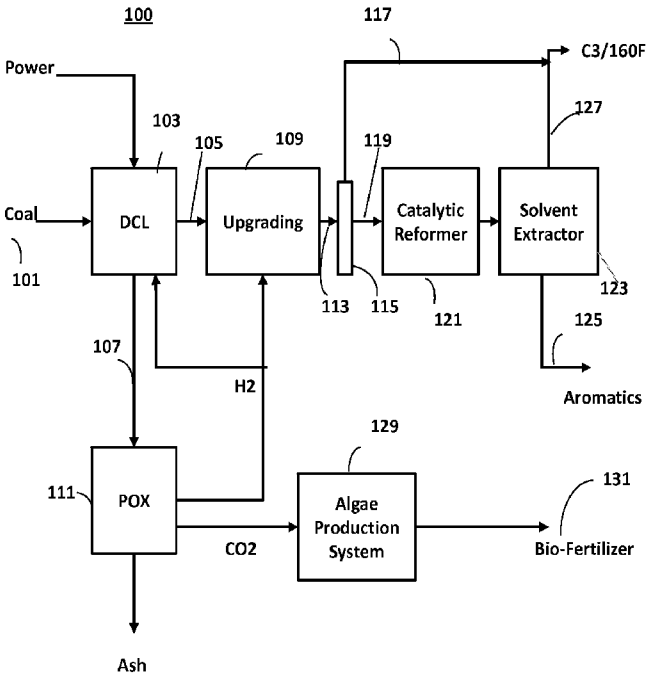
— *as to the identity of the inventor (Rule 4.17(i))*

[Continued on next page]

(54) Title: METHODS AND APPARATUS FOR PRODUCING AROMATICS FROM COAL

Figure 1



(57) Abstract: A method for converting coal into BTX in which feed coal is converted to a 600-700°F- product stream by direct liquefaction. This product stream is hydrocracked and hydroprocessed to produce a 350°F- stream which in turn is fractionated to produce a 160°F - stream and a 160/350°F stream that contains 85-90% naphthenes. The 160/350°F stream is catalytically reformed to produce an aromatic stream and a 160°F- paraffinic stream. The aromatics stream can be separated into benzene toluene and xylene streams by distillation.

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— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*

— *as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))*

Published:

— *without international search report and to be republished upon receipt of that report (Rule 48.2(g))*

METHODS AND APPARATUS FOR PRODUCING AROMATICS FROM COAL

FIELD OF THE INVENTION

The present invention relates to direct coal liquefaction processes for efficiently producing high-value aromatics from coal.

BACKGROUND OF THE INVENTION

Current and presently proposed methods for the production of high value aromatics (benzene, toluene, and xylene, i.e. BTX) from coal include either the capture of gaseous byproducts from the pyrolysis of coal in an airless environment, or the proposed conversion of coal to methanol followed by the conversion of the methanol to aromatics.

The pyrolysis method is of economic interest only as a byproduct of another process, e.g., producing coke from coal. The methanol to aromatics (MTA) process would involve the gasification of the coal feed, typically by partial oxidation (POX), to produce syngas as the feed for methanol synthesis, converts coal to methanol and then converts methanol to aromatics by processing over a fixed or fluid bed of catalyst. A published estimate stated that the required investment for a 1 million metric ton per stream day MTA plant in China would be about $4.6 billion. This high cost results in major part because of the need to use expensive POX for gasifying all of the coal. Available publications indicate that about 3 MT of coal would be required to produce 1 MT of methanol. A separate report states that 1.42 to 1.59 MT of methanol would be required to produce 1 MT of aromatics. If these two numbers are combined, it requires 4.26 to 4.77 MT of coal to produce 1 MT of aromatics. It is estimated that the thermal efficiency of the MTA process is about 40 to 45%.

SUMMARY OF THE INVENTION

In accordance with the invention, a highly efficient and lower cost method and system for producing high-value aromatics from coal is provided in which the feed coal is converted by direct coal liquefaction (DCL) to a 1000°F-, preferably an 800°F-, more preferably a 600 -750°F-

product, most preferably a 600-700°F- product, at least the 350°F+ portion of which is then hydrocracked to produce a 350° F- product stream. The DCL and hydrocracked 350°F- product streams are then hydroprocessed to remove sulfur, nitrogen and oxygen compounds and fractionated into approximately 160°F- and 160°F+ output streams. The approximately 160/350°F output stream is ideal for aromatics production. It typically contains 85 to 90% naphthenes, 5 to 10% paraffins, plus some single ring aromatics, and the naphthenes are easily converted into BTX in a catalytic reformer. The product from the catalytic reformer can be processed in a solvent extraction unit to produce a pure aromatics product and a paraffinic raffinate. Alternatively, after converting the naphthenes and heavier paraffins to aromatics in the catalytic reformer, the entire product can be passed over a bed of cracking catalyst in the same unit. This bed isomerizes lower value ethyl benzene to more valuable para-xylene and benzene, and cracks the remaining paraffins into an approximately 160°F- product that can be separated from the aromatics in the distillation tower, thereby producing a higher value aromatic product and eliminating the need for solvent extraction.

The preferred DCL system includes a slurry DCL reactor containing a molybdenum or iron, preferably molybdenum, microcatalyst and is operated at high conversion with the product boiling above the 600-700°F range preferably being recycled and mixed with the DCL feed coal as a non-donor stream in a ratio of non-donor stream to coal at the input to the reactor (on a moisture free weight basis) of between 1.6 and 3.5:1. By “non-donor” is meant that the recycle stream has not been processed in a hydrotreater to partially hydrogenate multi-ring aromatic compounds in the stream in order to produce compounds that can donate hydrogen during liquefaction.

In order to provide the additional hydrogen required for the DCL and hydrocracking processes, the bottoms from the DCL reactor can be gasified in a POX reactor. As an alternative, the additional hydrogen may be provided by processing 160°F- product of the upgrading and the catalytic reformer by liquid POX or steam naphtha reforming (SNR). If natural gas is available, it can be used as the feed to the liquid POX or SNR instead of the 160°F- product.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a diagram of the flow stream of an embodiment of the method and apparatus of the invention in which the additional hydrogen is provided by POX.

FIG. 2 is a schematic diagram of a preferred direct coal liquefaction system suitable for use in the illustrated embodiments of the invention.

FIG. 3 is a diagram of the flow stream of an embodiment of the method and apparatus of the invention in which the additional hydrogen is produced from the 160°F- product stream.

FIG. 4 is a diagram of the flow stream of an embodiment of the method and apparatus of the invention in which the additional hydrogen is produced from natural gas feed.

DETAILED DESCRIPTION OF ILLUSTRATED EMBODIMENTS

Referring now to FIG. 1 of the drawings, there is illustrated a schematic of the overall flow scheme of a first embodiment of a coal to aromatics plant 100 according to the invention. The coal feed 101 is supplied to the DCL unit 103 that is preferably operated at a high conversion of 80+% on a moisture and ash free (MAF) basis. In the DCL unit 103, coal is hydrogenated to produce an approximately a 1000°F-, preferably an 800°F-, more preferably a 600 -750°F- product, most preferably a 600-700°F- product stream 105 and an effluent stream 107 that consists of ash, unconverted coal, and liquids boiling above 1000°F. The product stream 105 flows to the upgrader 109, in which at least the 350°F+ portion of the stream is hydrocracked to produce a 350° F- product stream. The 350° F- product stream from the hydrocracker, or, if only the 350°F+ portion of the DCL product was hydrocracked, the combined 350° F- DCL and hydrocracked streams are hydroprocessed to remove heteroatoms, and to convert most of the aromatics present into naphthenes to produce a 350°F- product stream 113. This stream is then fractionated in the atmospheric fractionator 115 into approximately160°F- and 160°F+ outputs 117 and 119, respectively. Light gases produced in the DCL unit 103 and the upgrader 109 may be used to supply a portion of the fuel for the plant. Excess light gases (C2-) may be sent to a steam methane reformer to supply a portion of the hydrogen required by the DCL unit 103. The

bottoms stream 107 from the DCL unit 103 is gasified in the POX unit 111 for supplying additional hydrogen to the DCL unit 103 and the upgrader 109.

The approximately 160/350°F stream 119 from the fractionator 115 is typically made up of 85% to 90% naphthenes, 5 to 10% paraffins, and some single ring aromatics. This stream is fed to the catalytic reformer 121 where the naphthenes are converted into aromatics. The output of the catalytic reformer 121 is fed to the solvent extractor unit 123 where it is separated into a pure aromatics stream 125 and a lower boiling point paraffinic raffinate stream 127. The aromatics stream 125 can be separated into its benzene, toluene and xylene components by distillation.

As attractive alternative to the use of the extraction unit 123 is that described in U.S. Patent No. 5,472,593 in which, after converting the naphthenes and heavier paraffins to aromatics in the catalytic reformer, the entire product is passed over a bed of catalyst in the same unit comprising a medium-pore molecular sieve having a pore size of from 5 to 6.5 .ANG., a refractory inorganic oxide, a platinum-group metal component and a lead or bismuth metal attenuator. This bed converts the lower value ethyl benzene to more valuable para-xylene and benzene, and cracks the remaining paraffins into a 160°F- product that can be separated from the aromatics by distillation, thereby producing a higher value aromatic product and eliminating the need for solvent extraction. This alternative does, however, require additional hydrogen for cracking. The disclosure of U.S. Patent No. 5,472,593 is hereby Incorporated by reference in its entirety.

CO_2 produced by the POX unit 111, and optionally, by the DCL reactor system 103 and/or other components of the liquefaction and upgrading system, is fed to the algae production system 129, which includes a photo-bio reactor (PBR) in which the CO_2 is used to produce preferably blue-green algae through photosynthesis. The DCL reactor system 103 and especially the upgrading system 109 also produce NH_3, which can be fed to the algae production system 129 as a nutrient. The algae from the algae production system 129 is preferably used to produce a biofertilizer 131. Methods and

apparatus suitable for use in the present invention for producing algae and biofertilizer are disclosed in U.S. Patent Application No. 13/316,546 that was filed on December 11, 2011, the disclosure of which is hereby Incorporated by reference in its entirety.

Referring now to the embodiment of a DCL system illustrated in FIG. 2 of the drawings, the coal feed is dried and crushed in a conventional gas swept roller mill 201 to a moisture content of 1 to 4 %. Crushed and dried coal is fed into a mixing tank 203 where it is mixed with a stream constituted by a 600 to 700°F+ fraction, preferably a 650°F+ fraction, of the output of the liquefaction reactor to form a slurry stream. The catalyst precursor in the illustrated embodiment preferably is in the form of an aqueous water solution of phosphomolybdic acid (PMA) in an amount that is equivalent to adding between 50wppm and 2% molybdenum relative to the dry coal feed. In the slurry mix tank 203, typical operating temperature ranges from 300 to 600°F and more preferably between 300 and 500°F. From the slurry mix tank, the catalyst containing slurry is delivered to the slurry pump 205. The selection of the appropriate mixing and temperature conditions is based on experimental work quantifying the rheological properties of the specific slurry blend being processed.

Most of the remaining moisture in the coal is driven off in the mixing tank due to the hot atmospheric fractionator bottoms feeding to the mixing tanks. Residual moisture and any entrained volatiles are condensed out as sour water (not shown in Fig. 2). The coal in the slurry leaving the mixing tank 203 has about 0.1 to 1.0% moisture. The slurry formed by the coal, 600 to 700 to 1,000°F stream from the vacuum fractionator 221, and the 600 to 700°F+ stream fraction from the atmospheric fractionator 219 is pumped from the mixing tank 203 and the pressure is raised to about 2,000 to 3,000 psig (138 to 206 kg/cm^2 g) by the slurry pumping system 205. The resulting high pressure slurry may be preheated in a heat exchanger (not shown), mixed with a treat gas consisting of recycled and makeup treat gas containing over 80% hydrogen, and then further heated in furnace 207.

The coal slurry and hydrogen mixture is fed to the input of the first stage of the series-connected liquefaction reactors 209, 211 and 213 at between 600 to 700°F (316 to 371°C) and 2,000 to 3,000 psig (138 to 206 kg/cm^2 g). The reactors 209, 211 and 213 are simple up-flow tubular vessels, the total length of the three reactors being 40 to 200 feet. The temperature rises from one reactor stage to the next as a result of the highly exothermic coal liquefaction reactions. In order to maintain the maximum temperature in each stage below about 800 to 900 °F (427 to 482 °C), a portion of the hydrogen based treat gas is preferably injected between reactor stages. The hydrogen partial pressure in each stage is preferably maintained at a minimum of about 1,000 to 2,000 psig (69 to 138 kg/cm^{2}g).

The effluent from the last stage of liquefaction reactor is separated into a gas stream and a liquid/solid stream, and the liquid/solid stream let down in pressure, in the separation and cooling system 215. The gas stream is cooled to condense out the liquid vapors of H2O, naphtha, distillate, and solvent. The remaining gas is then processed to remove H_2S, NH_3 and CO_2

Most of the processed gas is then sent to a hydrogen recovery system, not shown, for further processing by conventional means to recover the hydrogen contained therein, which is then recycled to be mixed with the coal slurry. The remaining portion of the processed gas is purged to prevent buildup of light ends in the recycle loop. Hydrogen recovered therefrom can be used in the downstream hydro-processing upgrading system.

The depressurized liquid/solid stream and the hydrocarbons condensed during the gas cooling are sent to the atmospheric fractionator 219 where they are separated into light ends and in the preferred embodiment, a 600 to 700°F- fraction, and a 600 to 700°F+ fraction. The light ends are processed to recover hydrogen and C_1-C_2 hydrocarbons that can be used for fuel gas and other purposes. The 600 to 700°F- fraction is sent to upgrading for aromatics production. Alternatively, the fractionator 219 could be arranged to produce 1000°F- and 1000°F+ fractions or 800°F- and 800°F+ fractions in which the 1000°F- or 800°F- fraction would be sent to the upgrading step.

In the preferred embodiment, a portion of the 600 to 700°F+ (316 to 371°C+) is recycled to the slurry mix tank. The remaining 600 to 700° F+ fraction produced from the atmospheric fractionator 219 is fed to the vacuum fractionator 221 wherein it is separated into a 1000°F- fraction and a 1000°F+ fraction. The 1000°F- fraction is added to the 600 to 700°F+ stream being recycled to the slurry mix tank 203.

In the preferred embodiment illustrated in FIG. 2, the 1000° F+ fraction from the vacuum fractionator 221 is sent to be gasified by the POX system 223 (shown in FIG. 1 as POX 111) to generate hydrogen for use in the liquefaction and upgrading. Alternatively, instead of the POX system 223, the 1000° F+ bottoms from the vacuum fractionator 221 may be processed in a Circulating Fluid Bed boiler, a cement plant, or sold as a feed for asphalt paving or electrode manufacture. G.E., Shell, and others offer commercial processes for gasification (partial oxidation) of the 1000°F+ bottoms and Circulating Fluid Bed boiler manufactures such as Foster-Wheeler and Alstom offer technology for combusting the 1000°F+ bottoms.

Catalysts useful in DCL processes also include those disclosed in U.S. Patents Nos. 4,077,867, 4,196,072 and 4,561,964, the disclosures of which are hereby incorporated by reference in their entirety. Other DCL reactor systems suitable for use in the process of the invention are disclosed in U.S. Patents Nos. 4,485,008, 4,637,870, 5,200,063, 5,338,441, and 5,389,230, and U.S. Patent Application No. 13/657,087,the disclosures of which are hereby incorporated by reference in their entirety.

The preferred DCL Process combines several elements that contribute to maximum BTX Product production and maximum thermal efficiency. These include, very importantly, the recycle of a non-donor 600 to 700°F+ stream, preferably including atmospheric fractionator bottoms, to maintain a ratio of the recycle stream to coal at the input to the reactors 209, 211, 213 that is between 1.6:1 and 3.5:1 on a moisture free weight basis; the use of a microcatalyst in the form of finely divided molybdenum; and the use of a much lower treat gas rate than in previous systems. Also, the use of bottoms recycle, and multiple slurry reactors in series contribute to the benefits of the process.

Use of a microcatalyst, which is either a compound of molybdenum or iron, more preferably molybdenum, and added at 100 to 1,000 wppm, more preferably 100 to 500 wppm, and most preferably 100 to 300 wppm, eliminates several disadvantages to the use of a donor solvent such as required by prior DCL systems. First, energy is lost during preparation of the donor solvent. Second, energy is required to preheat the donor solvent in the solvent hydrotreater and hydrogen must be compressed and circulated around the hydrotreater. Thirdly, the heat release during partial hydrogenation of the donor solvent is lost during cooling prior to separation of hydrogen for recycle. In comparison, all of the heat release occurs in the liquefaction reactors during operation with a 600 to 700°F+ recycle stream, which minimizes the preheat requirement prior to liquefaction. These factors contribute to the higher thermal efficiency of the microcatalytic coal liquefaction process. Moreover, the use of a microcatalyst and the consequent elimination of the need for a donor solvent also eliminates the need for an expensive solvent hydrotreater to generate the donor solvent, thereby substantially reducing the capital and operating cost of the system. It also permits the use of coals having substantially higher ash contents, from 6 to 20 wt % or more on a moisture free basis, and the recycle of a substantially higher portion of bottoms than were possible with donor solvent systems. Examples of microcatalysts and their method of preparation are described in U.S. Patent No. 4,226,742, the contents of which are hereby incorporated by reference in their entirety.

The 600 to 700° F+ fraction recycled from the atmospheric fractionator 219 and the 1000° F - fraction from the vacuum fractionator 221 as the non-donor stream being recycled to the slurry mix tank 203 provides preheat for the coal and solvent in the slurry mix tank 203. This raises the temperature in the mix tank to 300°F to 500°F, more preferably 350°F to 500°F, and most preferably about 400 to 500°F. This further reduces the energy requirement for preheating the slurry prior to liquefaction. A significant portion of the of the microcatalyst is entrained in the 600 to 700° F+ fraction recycled from the atmospheric tower 219, so that recycling a larger portion of such fraction increases the catalyst concentration in the DCL reactors 209, 211, 213, thereby decreasing the requirement for the addition of fresh catalyst precursor and increasing the conversion efficiency of the DCL process.

Use of the non-donor 600°F to 700°F+ stream, more preferably 630°F to 670°F+, and most preferably a 650°F+, process derived recycle solvent in the DCL process reduces cracking, relative to donor solvent, and produces a 650°F- product with a greater fraction of diesel and less light gases and naphtha. The 650°F- product can be selectively upgraded to finished products in fixed bed upgrading reactors.

The much lower treat gas rate of 600 to 900 NL per kg of slurry has a significant impact on thermal efficiency, plant investment, and operating cost. The required recycle treat gas rate for the DCL process of the invention is up to three times lower than the preferred gas rate in the NEDOL program (without taking into account the treat gas rate to the solvent hydrotreater, which makes the difference even larger). This has an important impact on power requirements for the compressor and fuel requirements for slurry preheat furnace 207 and solvent hydrotreater preheat.

The use of two to four, more preferably three slurry reactors in series approaches a plug flow reactor and hence has as little as two thirds of the required volume of one or two ebullated bed reactors such as used in some prior DCL systems. Since all of the heat is released in the three liquefaction reactors, the temperature profile can be also maintained to maximize selectivity to liquids. Operation of the initial reactor at a somewhat lower temperature has been reported in previous patents as a route to increase conversion and liquid yields.

An exemplary process for upgrading the liquid product of the DCL reactors 209, 211, 213 is disclosed in U.S. Patent number 5,198,099, the disclosure of which is hereby incorporated by reference in its entirety. Other processes and systems suitable for upgrading the liquid products are commercially available from vendors such as UOP, Axens, Criterion and others.

Referring now to FIG. 3 of the drawings, there is illustrated a second embodiment of the coal to aromatics flow scheme of the invention. Elements of the flow scheme that are the same as corresponding elements of the embodiment of FIG. 1 are identified with the same reference numbers as the corresponding elements of FIG. 1. The primary difference between the embodiments of FIG's. 1 and 3 is that, in FIG. 3, the additional hydrogen required for the DCL

system 103 and the upgrader 109 is produced by the H_2 Plant 303 rather than by the POX unit 111 used in FIG. 1. The H_2 Plant 303 can be implemented as a steam naptha reformer (SNR) or a liquid POX unit, both of which are well known standard equipment in the art. For instance, SNR's are available from sources such as Akzo Nobel N.V. Similarly, liquid POX units are available from sources such as Haldor-Topsoe, Inc. or Lurgi GmbH. In either case, at least the C4- portion and if needed, part of the lighter portion of the C5+ portion of the 160°F- stream from the solvent extractor 123 is used as the feed to the H_2 Plant 303.

Referring now to FIG. 4 of the drawings, there is illustrated a third embodiment of the coal to aromatics process and system of the invention in which components that are the same as corresponding components in FIG's. 1 or 3 are labeled with the same reference numbers as such corresponding components in FIG's. 1 or 3. The primary difference between the embodiments of FIG's. 4 and 3 is that, in FIG. 4, the feed to the H_2 Plant 303 that produces the additional hydrogen required for the DCL system 103 and the upgrader 109 is supplied by natural gas rather than by a the C1-C4 portion of the output from the solvent extractor 123. In this case the H_2 Plant 303 is implemented as an SMR. The feed to the H_2 Plant 303 can also be from sources such as shale gas, or coal mine methane. The SMR technology is utilized worldwide in refineries and is offered by many commercial vendors such as Haldor-Topsoe.

The embodiment of the invention illustrated in FIG. 1 is less expensive to implement and has a substantially higher thermal efficiency (between 60 and 65%) than the prior MTA systems (40-45%). In the configuration of FIG. 1, POX (of the DCL bottoms) is still required, but the quantity of material being processed in the POX unit is greatly reduced. Since the principle product is aromatics, the remaining exported byproduct is a C3/160°F stream from the solvent extractor 123. This stream would be converted to lighter paraffins if the alternative approach disclosed in U.S. Patent No. 5,472,593 is utilized, but will also require additional hydrogen for cracking.

The embodiment of the invention illustrated in FIG. 3 requires the lowest investment per ton of aromatics and also has the simplest flow scheme. Instead of bottoms POX, the lighter portion of the low value C1/160°F product is utilized as a feed for the production of H_2. This allows use of

lower cost H_2 generating technologies including liquid POX (no ash) or SNR. A plant implementing the flow scheme of FIG. 3 consumes much of the lower value products that it produces, and therefore has a lower thermal efficiency (about 51.5%) than the embodiment of FIG. 1. Thus, there is a trade-off between higher thermal efficiency (FIG. 1), and lower investment, simpler, easier to operate plant (FIG. 3).

The embodiment of FIG. 4 is preferable where inexpensive natural gas is available. In this embodiment, H_2 is generated via SMR and the DCL bottoms are preferably sent to a CFB power plant for generation of power. Given the lower cost of shale or natural gas, this embodiment would be economically preferable to the production of H_2 from the 160°F- stream. This stream can better be sent to a Steam Cracking system for the production of olefins or aromatics. This embodiment also has a substantially higher thermal efficiency (about 70%).

CLAIMS

1. A method for producing aromatics from coal comprising the steps of:
    a. producing a 1000F- product stream from feed coal by direct coal liquefaction (DCL);
    b. hydrocracking the 1000° F- product stream to produce a 350°F- product stream;
    c. fractionating the 350°F- product stream into 160/350°F and lower boiling point streams; and
    d. converting the 160/350°F stream to aromatics and a lower boiling point stream by catalytic reforming.

2. The method of Claim 1 wherein the fraction produced in step a is an 800° F- fraction.

3. The method of Claim 1 wherein the fraction produced in step a is a 600-700° F- fraction.

4. The method of Claim 1 further including gasifying bottoms produced in the direct coal liquefaction to produce hydrogen for supply to the direct coal liquefaction and hydrocracking steps.

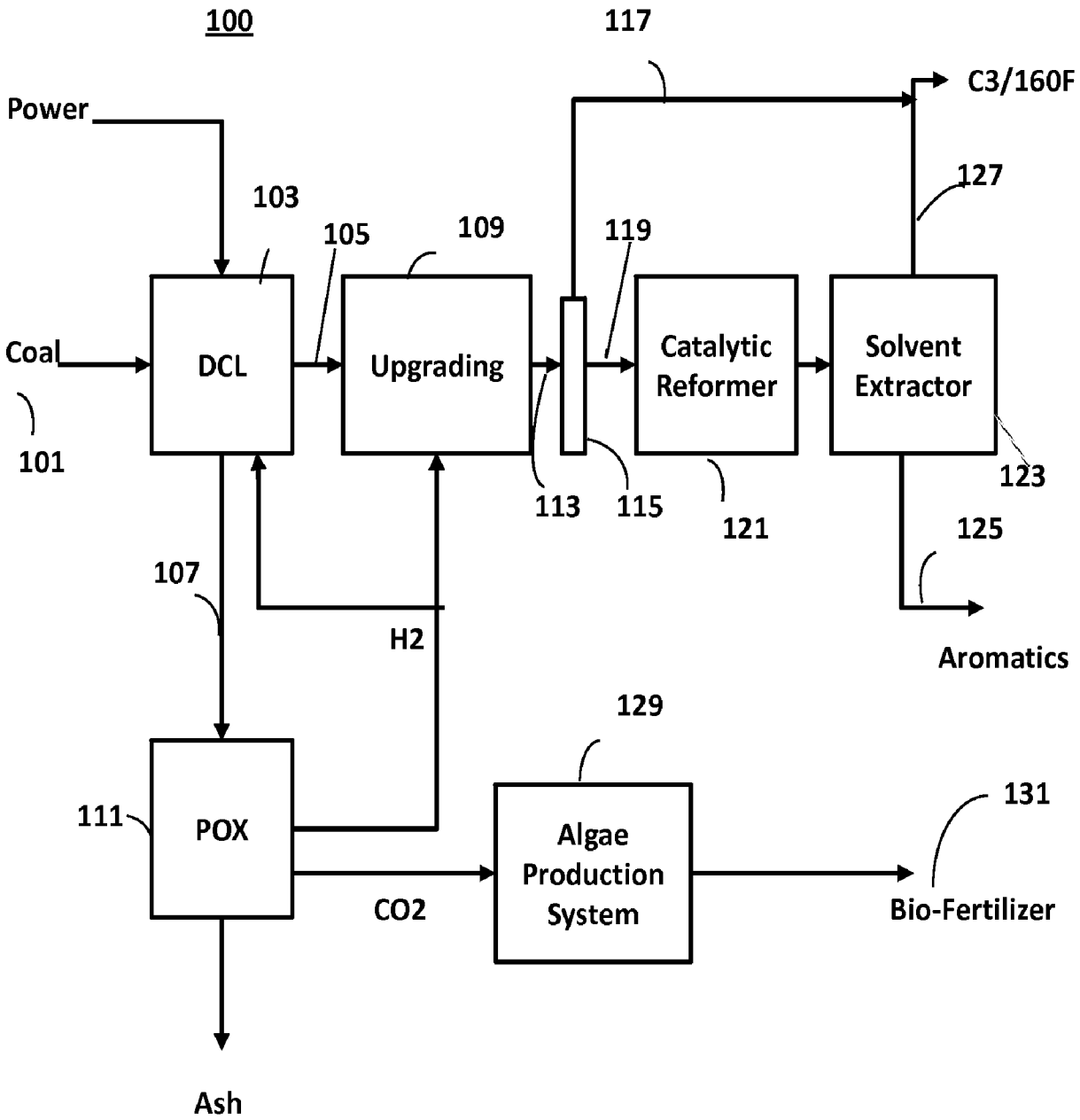
5. The method of Claim 1 or 3 further including processing at least a portion of the lower boiling point streams to produce hydrogen for supply to the direct coal liquefaction and/or hydrocracking steps.

6. The method of Claim 1 further including processing natural gas feed to produce hydrogen for supply to the direct coal liquefaction and hydrocracking steps.

7. The method of Claim 1 further including separating aromatics produced in step d. into benzene toluene and xylene streams.

8. The method of Claim 1 or 3 wherein the lower boiling point stream in step c is a 160° F- stream.

Figure 1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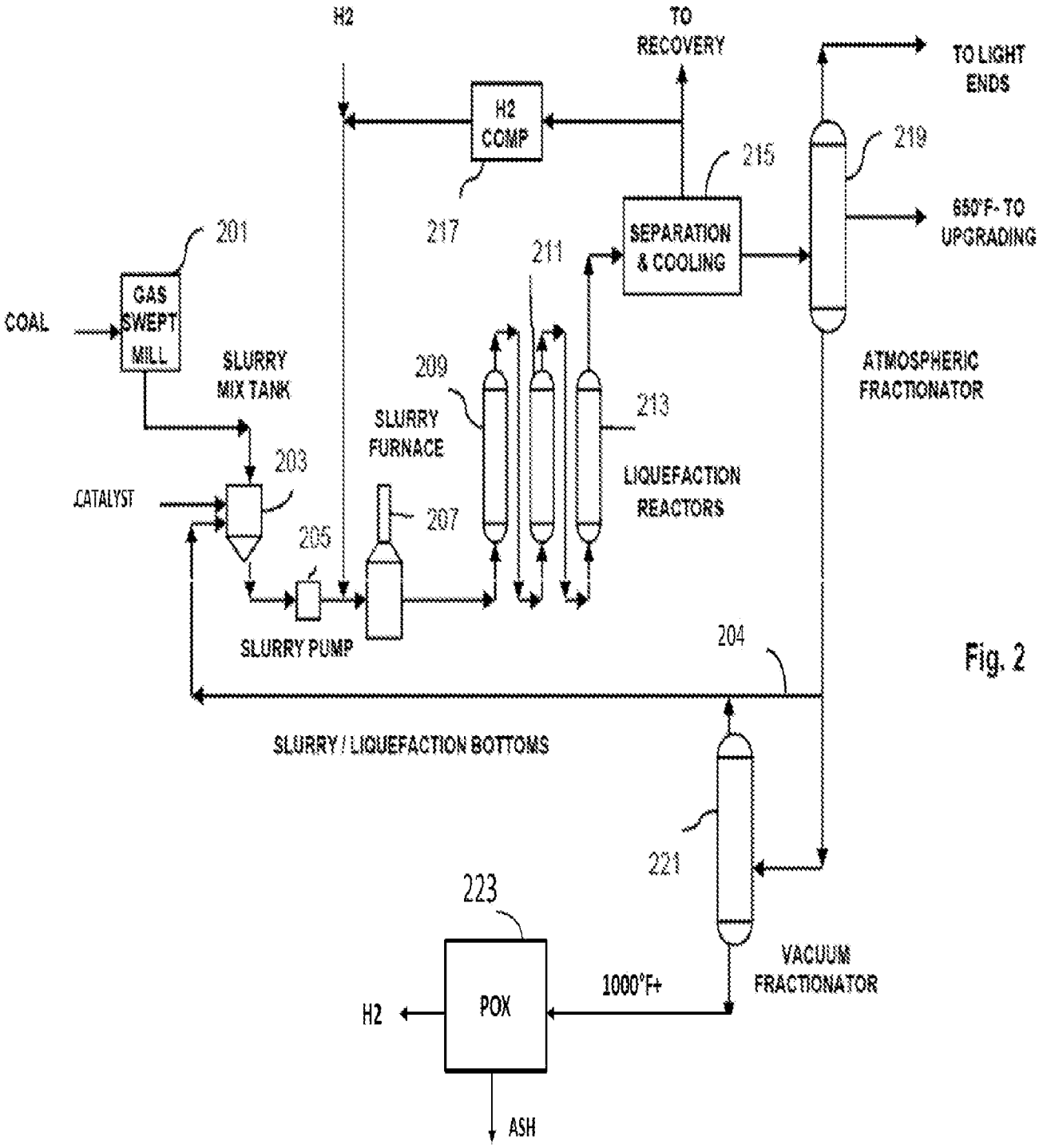
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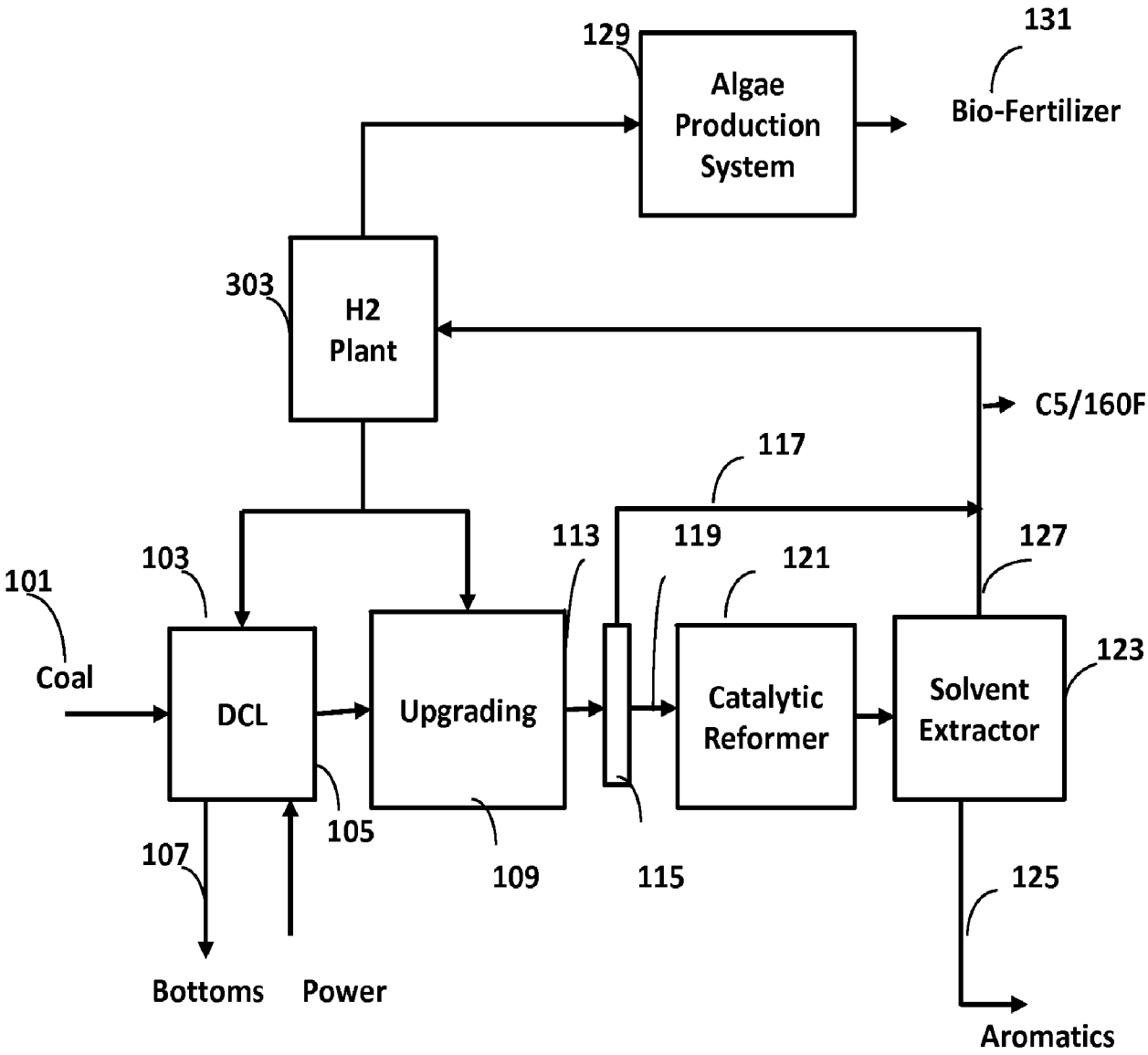
ASH

Fig. 2

Figure 3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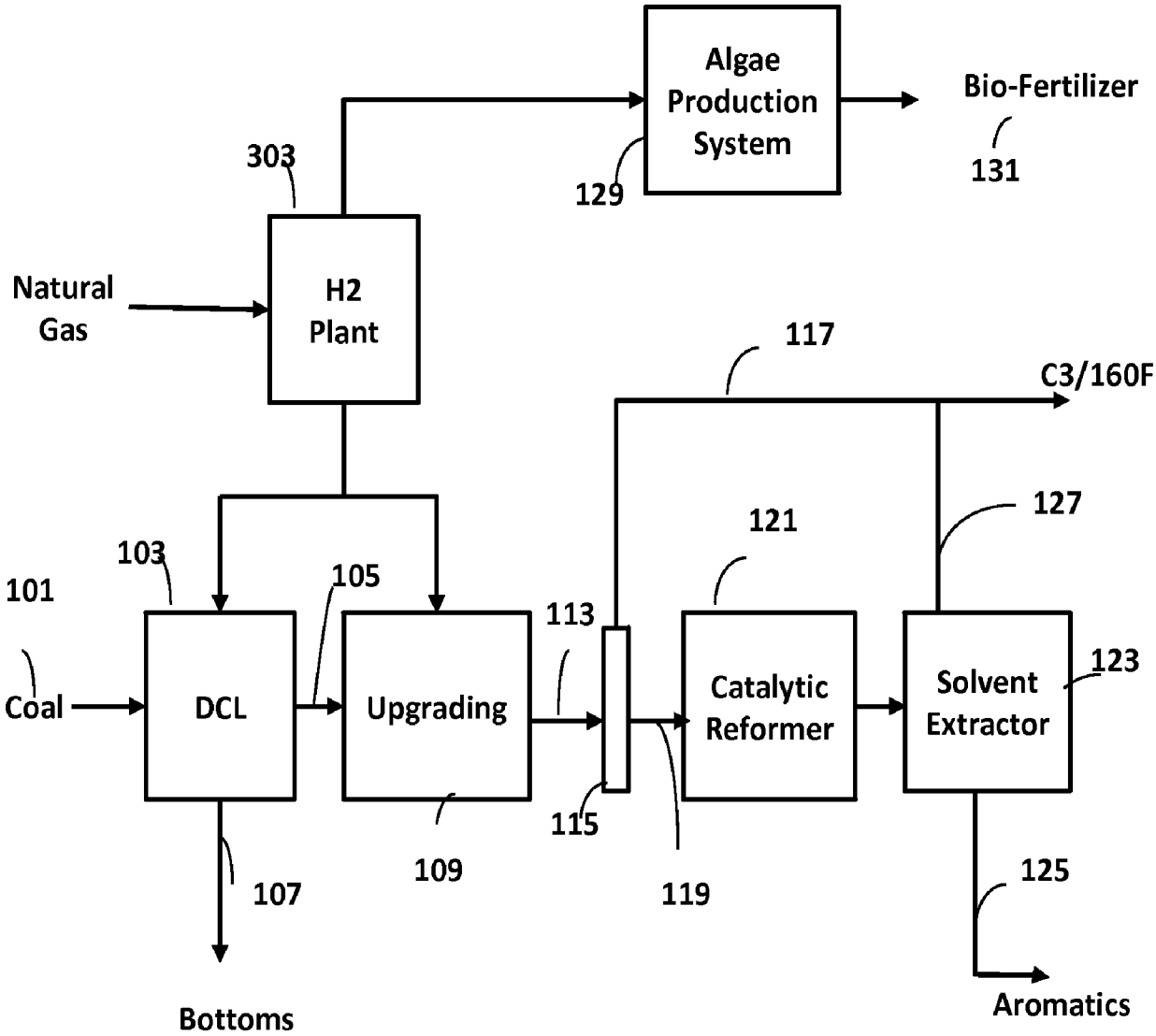
DCL
Upgrading
Catalytic Reformer
Solvent Extractor
105
107
109
115
125
Bottoms
Power
Aromatics

Figure 4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Upgrading
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