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(54) **PROCESS FOR REMOVING ASH AND HEAVY HYDROCARBONS FROM COAL TAR**

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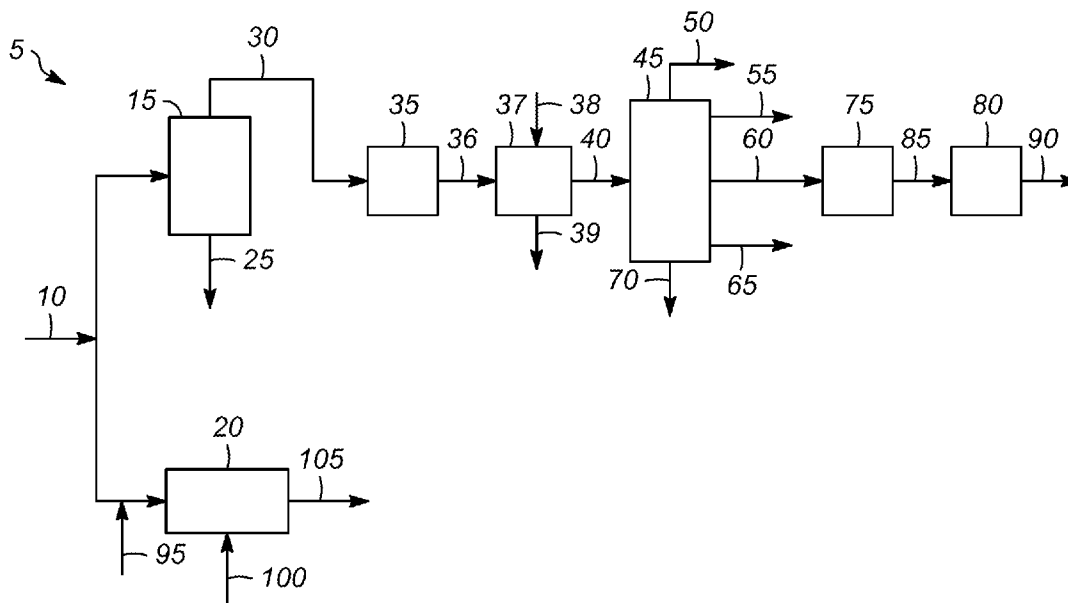
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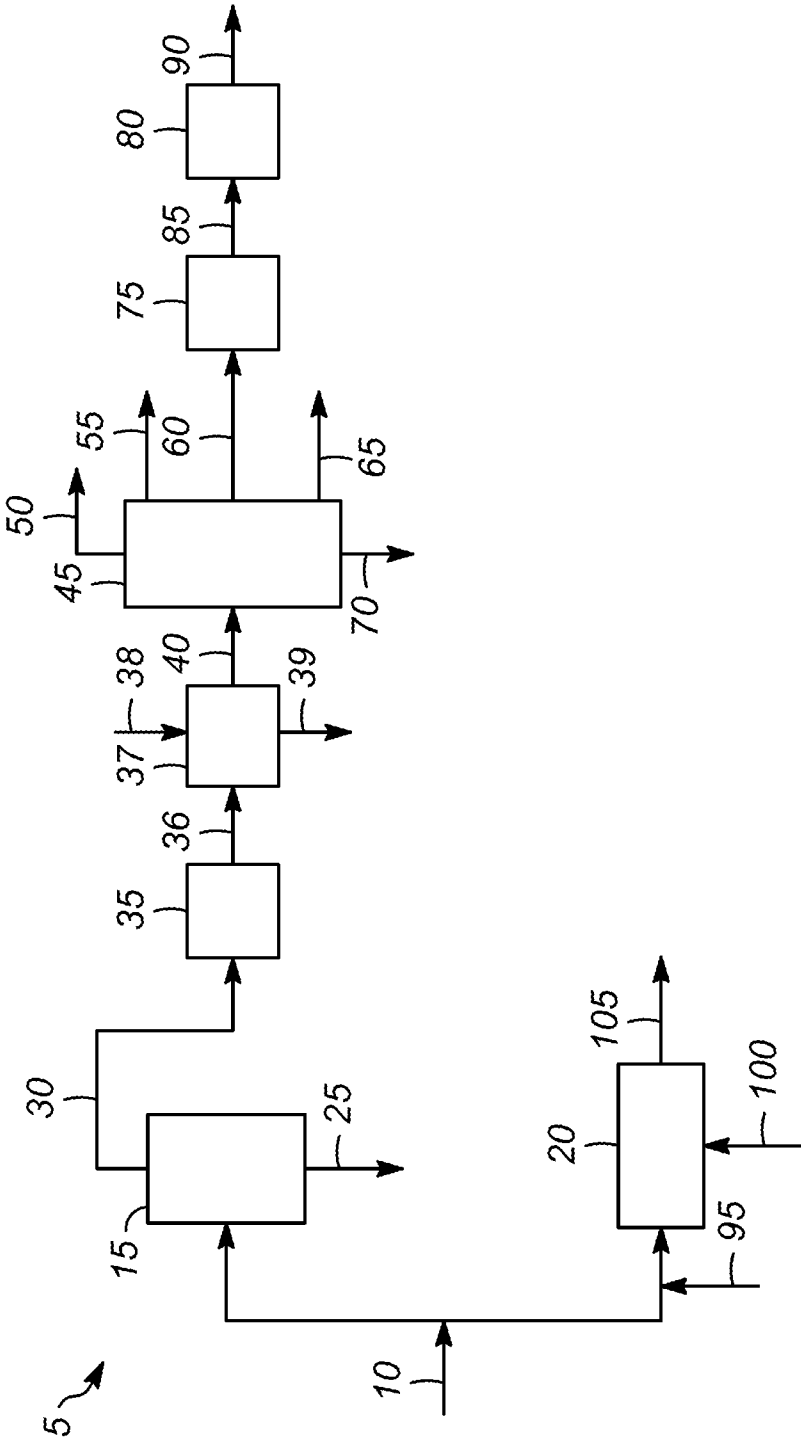
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(57) **ABSTRACT**

A process for removing ash and heavy hydrocarbon compounds from coal is described. The coal feed, the coal tar stream, or a coal tar fraction is contacted with a solvent to dissolve a soluble portion of the coal tar stream, the ash and heavy hydrocarbons being insoluble in the solvent, the solvent selected from the group consisting of dimethyl sulfoxide, sulfolane, dimethyl formamide, glyme, diglyme, ionic liquids, and combinations thereof, with the proviso that an anion of the ionic liquid is not a dialkylphosphate.





PROCESS FOR REMOVING ASH AND HEAVY HYDROCARBONS FROM COAL TAR

[0001] This application claims the benefit of Provisional Application Ser. No. 61/905,904 filed Nov. 19, 2013, entitled Process for Removing Ash and Heavy Hydrocarbons from Coal Tar.

BACKGROUND OF THE INVENTION

[0002] Many different types of chemicals are produced from the processing of petroleum. However, petroleum is becoming more expensive because of increased demand in recent decades.

[0003] Therefore, attempts have been made to provide alternative sources for the starting materials for manufacturing chemicals. Attention is now being focused on producing liquid hydrocarbons from solid carbonaceous materials, such as coal, which is available in large quantities in countries such as the United States and China.

[0004] Pyrolysis of coal produces coke and coal tar. The coke-making or "coking" process consists of heating the material in closed vessels in the absence of oxygen to very high temperatures. Coke is a porous but hard residue that is mostly carbon and inorganic ash, which is used in making steel.

[0005] Coal tar is the volatile material that is driven off during heating, and it comprises a mixture of a number of hydrocarbon compounds. It can be separated to yield a variety of organic compounds, such as benzene, toluene, xylene, naphthalene, anthracene, and phenanthrene. These organic compounds can be used to make numerous products, for example, dyes, drugs, explosives, flavorings, perfumes, preservatives, synthetic resins, and paints and stains. The residual pitch left from the separation is used for paving, roofing, waterproofing, and insulation.

[0006] Coal and coal tar contain ash and heavy hydrocarbon compounds which must be removed in order to utilize the coal tar.

[0007] Thus, there is a need for a process for removing ash and heavy hydrocarbons from coal.

SUMMARY OF THE INVENTION

[0008] One aspect of the invention is a process for removing ash and heavy hydrocarbon compounds from coal. In one embodiment, the process includes pyrolyzing a coal feed into a coal tar stream and a coke stream; separating the coal tar stream into at least two fractions; contacting one or more of the coal feed, the coal tar stream, or one of the fractions with a solvent to dissolve a soluble portion of the coal tar stream, the ash and heavy hydrocarbons being insoluble in the solvent, the solvent selected from the group consisting of dimethyl sulfoxide, sulfolane, dimethyl formamide, glyme, diglyme, ionic liquids, and combinations thereof, with the proviso that an anion of the ionic liquid is not a dialkylphosphate.

BRIEF DESCRIPTION OF THE DRAWING

[0009] The FIGURE illustrates one embodiment of the process of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

[0010] The FIGURE shows one embodiment of an ash and heavy hydrocarbon removal process 5 of the present inven-

tion. The coal feed 10 can be sent to the pyrolysis zone 15, the gasification zone 20, or the coal feed 10 can be split into two parts and sent to both.

[0011] In the pyrolysis zone 15, the coal is heated at high temperature, e.g., up to about 2,000° C. (3600° F.), in the absence of oxygen to drive off the volatile components. Coking produces a coke stream 25 and a coal tar stream 30. The coke stream 25 can be used in other processes, such as the manufacture of steel.

[0012] The coal tar stream 30 which comprises the volatile components from the coking process can be sent to an optional contaminant removal zone 35, if desired.

[0013] The contaminant removal zone 35 for removing one or more contaminants from the coal tar stream or another process stream may be located at various positions along the process depending on the impact of the particular contaminant on the product or process and the reason for the contaminant's removal, as described further below. For example, the contaminant removal zone can be positioned upstream of the separation zone 45. Some contaminants have been identified to interfere with a downstream processing step or hydrocarbon conversion process, in which case the contaminant removal zone 35 may be positioned upstream of the separation zone 45 or between the separation zone 45 and the particular downstream processing step at issue. Still other contaminants have been identified that should be removed to meet particular product specifications. Where it is desired to remove multiple contaminants from the hydrocarbon or process stream, various contaminant removal zones may be positioned at different locations along the process. In still other approaches, a contaminant removal zone may overlap or be integrated with another process within the system, in which case the contaminant may be removed during another portion of the process, including, but not limited to the separation zone or the downstream hydrocarbon conversion zone. This may be accomplished with or without modification to these particular zones, reactors or processes. While the contaminant removal zone is often positioned downstream of the hydrocarbon conversion reactor, it should be understood that the contaminant removal zone in accordance herewith may be positioned upstream of the separation zone, between the separation zone and the hydrocarbon conversion zone, or downstream of the hydrocarbon conversion zone or along other streams within the process stream, such as, for example, a carrier fluid stream, a fuel stream, an oxygen source stream, or any streams used in the systems and the processes described herein. The contaminant concentration is controlled by removing at least a portion of the contaminant from the coal tar stream 30. As used herein, the term removing may refer to actual removal, for example by adsorption, absorption, or membrane separation, or it may refer to conversion of the contaminant to a more tolerable compound, or both.

[0014] The contaminants can be removed by contacting the coal tar stream (or one of the fractions) with an adsorbent. The adsorbent can comprise a metal deposited on a support selected from the group consisting of molecular sieves, alumina, activated carbons, amorphous silica-alumina, clay, and silica gel; silver impregnated zeolite selected from the group consisting of faujasites (13X, CaX, NaY, CaY, and ZnX), chabazites, clinoptilolites and LTA (3A, 4A, 5A) zeolites; sulfur or a metal sulfide on an activated carbon support or an activated alumina support; or a metal sulfide, metal oxide, or metal carbonate on a support, the metal is selected from the group consisting of copper, silver, gold, antimony, lead, and

manganese, and the support selected from the group consisting of alumina, silica, clay, or carbon.

[0015] The decontaminated coal tar stream **36** from the contaminant removal zone **35** is sent to an extraction and separation zone **37**. A solvent stream **38** is introduced into the extraction and separation zone **37** and contacts the decontaminated coal tar stream **36**. The soluble portion of the coal tar stream dissolves in the solvent. The ash and heavy hydrocarbons are not soluble in the solvent. By heavy hydrocarbons we mean a dark brown to black material comprised of polycyclic aromatic hydrocarbons (PAHs) which may also contain heterocyclic oxygen, sulfur, and nitrogen compounds.

[0016] The solvent can include, but is not limited to, dimethyl sulfoxide, sulfolane, dimethyl formamide, glyme, diglyme, ionic liquids, and combinations thereof

[0017] Ionic liquids are non-aqueous, organic salts composed of ions where the positive ion is charge balanced with a negative ion. These materials have low melting points, often below 100° C., undetectable vapor pressure, and good chemical and thermal stability. The cationic charge of the salt is localized over hetero atoms, such as nitrogen, phosphorous, sulfur, arsenic, boron, antimony, and aluminum, and the anions may be any inorganic, organic, or organometallic species. Suitable ionic liquids include, but are not limited to, imidazolium-based ionic liquids, pyridinium-based ionic liquids, pyrrolidinium-based ionic liquids, ammonium-based ionic liquids, sulphonium-based ionic liquids, phosphonium-based ionic liquids, caprolactam-based ionic liquids, or combinations thereof Suitable anions for the ionic liquids include, but are not limited to, halides, sulfates, acetates, tosylates, phosphates, antimonates, borates, aluminates, imides, thiocyanates, dicyanimides, or combinations thereof

[0018] The ash and heavy hydrocarbons **39** are separated from the solvent/soluble portion of the coal tar stream. The separation can be effected by any suitable separation means, including, but not limited to, settling, decantation, filtration, centrifugation, using a filter column, and combinations thereof.

[0019] The solvent is separated from the soluble portion of the coal tar stream and recycled. The ionic liquids, and solvents can be regenerated by stripping the liquids with steam or nitrogen at a temperature of about 50° C.-150° C. or above, extracting with a solvent, or other known methods. The solvents can be further purified using any suitable process, if needed. Dimethyl sulfoxide and sulfolane can be purified by distillation or vacuum distillation, for example. Dimethyl formamide can be regenerated by distillation

[0020] The treated coal tar feed **40** with the ash and heavy hydrocarbons **39** removed is sent to a separation zone **45** where it is separated into two or more fractions. Coal tar comprises a complex mixture of heterocyclic aromatic compounds and their derivatives with a wide range of boiling points. The number of fractions and the components in the various fractions can be varied as is well known in the art. A typical separation process involves separating the coal tar into four to six streams. For example, there can be a fraction comprising NH₃, CO, and light hydrocarbons, a light oil fraction with boiling points between 0° C. and 180° C., a middle oil fraction with boiling points between 180° C. to 230° C., a heavy oil fraction with boiling points between 230 to 270° C., an anthracene oil fraction with boiling points between 270° C. to 350° C., and pitch. The process will be designed to minimize the pitch by removal of as much as possible with the ash.

[0021] The light oil fraction contains compounds such as benzenes, toluenes, xylenes, naphtha, coumarone-indene, dicyclopentadiene, pyridine, and picolines. The middle oil fraction contains compounds such as phenols, cresols and cresylic acids, xylenols, naphthalene, high boiling tar acids, and high boiling tar bases. The heavy oil fraction contains benzene absorbing oil and creosotes. The anthracene oil fraction contains anthracene. Pitch is the residue of the coal tar distillation containing primarily aromatic hydrocarbons and heterocyclic compounds.

[0022] As illustrated, the coal tar feed **40** is separated into gas fraction **50** containing gases such as NH₃ and CO as well as light hydrocarbons, such as ethane, hydrocarbon fractions **55**, **60**, and **65** having different boiling point ranges, and pitch fraction **70**.

[0023] Suitable separation processes include, but are not limited to fractionation.

[0024] One or more of the fractions **50**, **55**, **60**, **65**, **70** can be further processed, as desired. As illustrated, fraction **60** can be sent to one or more hydrocarbon conversion zones **75**, **80**. For example, where hydrocarbon conversion zone **80** includes a catalyst which is sensitive to sulfur, fraction **60** can be sent to hydrocarbon conversion zone **75** for hydrotreating to remove sulfur and nitrogen. Effluent **85** is then sent to hydrocarbon conversion zone **80** for hydrocracking, for example, to produce product **90**. Suitable hydrocarbon conversion zones include, but are not limited to, hydrotreating zones, hydrocracking zones, fluid catalytic cracking zones, alkylation zones, and transalkylation zones.

[0025] Hydrotreating is a process in which hydrogen gas is contacted with a hydrocarbon stream in the presence of suitable catalysts which are primarily active for the removal of heteroatoms, such as sulfur, nitrogen, oxygen, and metals from the hydrocarbon feedstock. In hydrotreating, hydrocarbons with double and triple bonds may be saturated. Aromatics may also be saturated. Typical hydrotreating reaction conditions include a temperature of about 290° C. (550° F.) to about 455° C. (850° F.), a pressure of about 3.4 MPa (500 psig) to about 6.2 MPa (900 psig), a liquid hourly space velocity of about 0.5 hr⁻¹ to about 4 hr⁻¹, and a hydrogen rate of about 168 to about 1,011 Nm³/m³ oil (1,000-6,000 scf/bbl). Typical hydrotreating catalysts include at least one Group VIII metal, preferably iron, cobalt and nickel, and at least one Group VI metal, preferably molybdenum and tungsten, on a high surface area support material, preferably alumina Other typical hydrotreating catalysts include zeolitic catalysts, as well as noble metal catalysts where the noble metal is selected from palladium and platinum.

[0026] Hydrocracking is a process in which hydrocarbons crack in the presence of hydrogen to lower molecular weight hydrocarbons. Typical hydrocracking conditions may include a temperature of about 290° C. (550° F.) to about 468° C. (875° F.), a pressure of about 3.5 MPa (500 psig) to about 20.7 MPa (3000 psig), a liquid hourly space velocity (LHSV) of about 1.0 to less than about 2.5 hr⁻¹, and a hydrogen rate of about 421 to about 2,527 Nm³/m³ oil (2,500-15,000 scf/bbl). Typical hydrocracking catalysts include amorphous silica-alumina bases or low-level zeolite bases combined with one or more Group VIII or Group VIB metal hydrogenating components, or a crystalline zeolite cracking base upon which is deposited a Group VIII metal hydrogenating component. Additional hydrogenating components may be selected from Group VIB for incorporation with the zeolite base.

[0027] Fluid catalytic cracking (FCC) is a catalytic hydrocarbon conversion process accomplished by contacting heavier hydrocarbons in a fluidized reaction zone with a catalytic particulate material. The reaction in catalytic cracking is carried out in the absence of substantial added hydrogen or the consumption of hydrogen. The process typically employs a powdered catalyst having the particles suspended in a rising flow of feed hydrocarbons to form a fluidized bed. In representative processes, cracking takes place in a riser, which is a vertical or upward sloped pipe. Typically, a pre-heated feed is sprayed into the base of the riser via feed nozzles where it contacts hot fluidized catalyst and is vaporized on contact with the catalyst, and the cracking occurs converting the high molecular weight oil into lighter components including liquefied petroleum gas (LPG), gasoline, and a distillate. The catalyst-feed mixture flows upward through the riser for a short period (a few seconds), and then the mixture is separated in cyclones. The hydrocarbons are directed to a fractionator for separation into LPG, gasoline, diesel, kerosene, jet fuel, and other possible fractions. While going through the riser, the cracking catalyst is deactivated because the process is accompanied by formation of coke which deposits on the catalyst particles. Contaminated catalyst is separated from the cracked hydrocarbon vapors and is further treated with steam to remove hydrocarbon remaining in the pores of the catalyst. The catalyst is then directed into a regenerator where the coke is burned off the surface of the catalyst particles, thus restoring the catalyst's activity and providing the necessary heat for the next reaction cycle. The process of cracking is endothermic. The regenerated catalyst is then used in the new cycle. Typical FCC conditions include a temperature of about 400° C. to about 800° C., a pressure of about 0 to about 688 kPa g (about 0 to 100 psig), and contact times of about 0.1 seconds to about 1 hour. The conditions are determined based on the hydrocarbon feedstock being cracked, and the cracked products desired. Zeolite-based catalysts are commonly used in FCC reactors, as are composite catalysts which contain zeolites, silica-aluminas, alumina, and other binders.

[0028] Transalkylation is a chemical reaction resulting in transfer of an alkyl group from one organic compound to another. Catalysts, particularly zeolite catalysts, are often used to effect the reaction. If desired, the transalkylation catalyst may be metal stabilized using a noble metal or base metal, and may contain suitable binder or matrix material such as inorganic oxides and other suitable materials. In a transalkylation process, a polyalkylaromatic hydrocarbon feed and an aromatic hydrocarbon feed are provided to a transalkylation reaction zone. The feed is usually heated to reaction temperature and then passed through a reaction zone, which may comprise one or more individual reactors. Passage of the combined feed through the reaction zone produces an effluent stream comprising unconverted feed and product monoalkylated hydrocarbons. This effluent is normally cooled and passed to a stripping column in which substantially all C5 and lighter hydrocarbons present in the effluent are concentrated into an overhead stream and removed from the process. An aromatics-rich stream is recovered as net stripper bottoms, which is referred to as the transalkylation effluent.

[0029] The transalkylation reaction can be effected in contact with a catalytic composite in any conventional or otherwise convenient manner and may comprise a batch or continuous type of operation, with a continuous operation being

preferred. The transalkylation catalyst is usefully disposed as a fixed bed in a reaction zone of a vertical tubular reactor, with the alkylaromatic feed stock charged through the bed in an upflow or downflow manner. The transalkylation zone normally operates at conditions including a temperature in the range of about 130° C. to about 540° C. The transalkylation zone is typically operated at moderately elevated pressures broadly ranging from about 100 kPa to about 10 MPa absolute. The transalkylation reaction can be effected over a wide range of space velocities. That is, volume of charge per volume of catalyst per hour; weight hourly space velocity (WHSV) generally is in the range of from about 0.1 to about 30 hr⁻¹. The catalyst is typically selected to have relatively high stability at a high activity level.

[0030] Alkylation is typically used to combine light olefins, for example, mixtures of alkenes such as propylene and butylene, with isobutane to produce a relatively high-octane branched-chain paraffinic hydrocarbon fuel, including isohexane and isooctane. Similarly, an alkylation reaction can be performed using an aromatic compound such as benzene in place of the isobutane. When using benzene, the product resulting from the alkylation reaction is an alkylbenzene (e.g. toluene, xylenes, ethylbenzene, etc.). For isobutane alkylation, typically, the reactants are mixed in the presence of a strong acid catalyst, such as sulfuric acid or hydrofluoric acid. The alkylation reaction is carried out at mild temperatures, and is typically a two-phase reaction. Because the reaction is exothermic, cooling is needed. Depending on the catalyst used, normal refinery cooling water provides sufficient cooling. Alternatively, a chilled cooling medium can be provided to cool the reaction. The catalyst protonates the alkenes to produce reactive carbocations which alkylate the isobutane reactant, thus forming branched chain paraffins from isobutane. Aromatic alkylation is generally now conducted with solid acid catalysts including zeolites or amorphous silica-aluminas

[0031] The alkylation reaction zone is maintained at a pressure sufficient to maintain the reactants in liquid phase. For a hydrofluoric acid catalyst, a general range of operating pressures is from about 200 to about 7100 kPa absolute. The temperature range covered by this set of conditions is from about -20° C. to about 200° C. For at least alkylation of aromatic compounds, the temperature range is about from 100-200C at the pressure range of about 200 to about 7100 kPa.

[0032] In some processes, all or a portion of the coal feed **10** is mixed with oxygen **95** and steam **100** and reacted under heat and pressure in the gasification zone **20** to form syngas **105**, which is a mixture of carbon monoxide and hydrogen. The syngas **105** can be further processed using the Fischer-Tropsch reaction to produce gasoline or using the water-gas shift reaction to produce more hydrogen.

[0033] While at least one exemplary embodiment has been presented in the foregoing detailed description of the invention, it should be appreciated that a vast number of variations exist. It should also be appreciated that the exemplary embodiment or exemplary embodiments are only examples, and are not intended to limit the scope, applicability, or configuration of the invention in any way. Rather, the foregoing detailed description will provide those skilled in the art with a convenient road map for implementing an exemplary embodiment of the invention. It being understood that various changes may be made in the function and arrangement of

elements described in an exemplary embodiment without departing from the scope of the invention as set forth in the appended claims.

What is claimed is:

1. A process for removing ash and heavy hydrocarbon compounds from coal comprising:

pyrolyzing a coal feed into a coal tar stream and a coke stream;

separating the coal tar stream into at least two fractions;

contacting one or more of the coal tar stream, or one of the fractions with a solvent to dissolve a soluble portion of the coal tar stream, or one of the fractions, the ash and heavy hydrocarbons being insoluble in the solvent, the solvent selected from the group consisting of dimethyl sulfoxide, sulfolane, dimethyl formamide, glyme, diglyme, ionic liquids, and combinations thereof, with the proviso that an anion of the ionic liquid is not a dialkylphosphate.

2. The process of claim 1 further comprising separating the ash and heavy hydrocarbons from the solvent.

3. The process of claim 2 wherein the ash and heavy hydrocarbons are separated from the solvent by one or more of settling, decantation, filtration, centrifugation, and using a filter column.

4. The process of claim 1 wherein the solvent is the ionic liquid and wherein the ionic liquid comprises imidazolium-based ionic liquids, pyridinium-based ionic liquids, pyrrolidinium-based ionic liquids, ammonium-based ionic liquids, sulphonium-based ionic liquids, phosphonium-based ionic liquids, caprolactam-based ionic liquids, or combinations thereof.

5. The process of claim 1 wherein the solvent is the ionic liquid and wherein the anion of the ionic liquid comprises halides, sulfates, acetates, tosylates, phosphates, antimonates, borates, aluminates, imides, thiocyanates, dicyanides, or combinations thereof.

6. The process of claim 1 further comprising regenerating the used solvent and recycling the regenerated solvent.

7. The process of claim 6 wherein the solvent is dimethyl sulfoxide, sulfolane, dimethyl formamide, or combinations thereof, and further comprising purifying the regenerated solvent before recycling the regenerated solvent.

8. The process of claim 6 wherein the solvent is the ionic liquid and wherein regenerating the used solvent comprises stripping the used solvent with steam or nitrogen at a temperature of about 50° C. or above, or extracting with a solvent.

9. The process of claim 1 further comprising removing at least one contaminant from the coal tar stream or one of the fractions by solvent extraction, adsorption with an adsorbent, or oxidation to form a treated stream having a reduced level of the at least one contaminant, the at least one contaminant comprising nitrogen containing compounds, sulfur containing compounds, and metals.

10. The process of claim 9 wherein the contaminant is the sulfur containing compounds and wherein removing at least one contaminant from the coal tar stream or one of the fractions comprises oxidizing the sulfur containing compounds and extracting the oxidized sulfur containing compounds by solvent extraction.

11. The process of claim 9 wherein removing the at least one contaminant comprises contacting the coal tar stream or the one of the fractions with an adsorbent, and wherein the adsorbent comprises a metal deposited on a support selected

from the group consisting of molecular sieves, alumina, activated carbons, amorphous silica-alumina, clay, and silica gel; silver impregnated zeolite selected from the group consisting of faujasites (13X, CaX, NaY, CaY, and ZnX), chabazites, clinoptilolites and LTA (3A, 4A, 5A) zeolites; sulfur or a metal sulfide on an activated carbon support or an activated alumina support; or a metal sulfide, metal oxide, or metal carbonate on a support, the metal is selected from the group consisting of copper, silver, gold, antimony, lead, and manganese, and the support selected from the group consisting of alumina, silica, clay, or carbon.

12. The process of claim 1 further comprising processing at least one of the fractions to produce at least one product.

13. The process of claim 12 wherein the at least one fraction is processed by at least one of hydrotreating, hydrocracking, fluid catalytic cracking, alkylation, and transalkylation.

14. A process for removing ash and heavy hydrocarbon compounds from coal comprising:

pyrolyzing a coal feed into a coal tar stream and a coke stream;

separating the coal tar stream into at least two fractions;

contacting one or more of the coal tar stream, or one of the fractions with a solvent to dissolve a soluble portion of the coal tar stream or one of the fractions, the ash and heavy hydrocarbons being insoluble in the solvent, the solvent selected from the group consisting of dimethyl sulfoxide, sulfolane, dimethyl formamide, glyme, diglyme, ionic liquids, and combinations thereof, with the proviso that an anion of the ionic liquid is not a dialkylphosphate;

separating the ash and heavy hydrocarbons from the solvent; and

processing at least one of the fractions to produce at least one product.

15. The process of claim 14 wherein the ash and heavy hydrocarbons are separated from the solvent by one or more of settling, decantation, filtration, centrifugation, and using a filter column.

16. The process of claim 14 wherein the solvent is the ionic liquid and wherein the ionic liquid comprises imidazolium-based ionic liquids, pyridinium-based ionic liquids, pyrrolidinium-based ionic liquids, ammonium-based ionic liquids, sulphonium-based ionic liquids, phosphonium-based ionic liquids, caprolactam-based ionic liquids, or combinations thereof.

17. The process of claim 14 wherein the solvent is the ionic liquid and wherein the anion of the ionic liquid comprises halides, sulfates, acetates, tosylates, phosphates, antimonates, borates, abominates, imides, thiocyanates, dicyanides, or combinations thereof.

18. The process of claim 14 further comprising regenerating the used solvent and recycling the regenerated solvent.

19. The process of claim 14 further comprising removing at least one contaminant from the coal tar stream or one of the fractions by solvent extraction, adsorption with an adsorbent, or oxidation to form a treated stream having a reduced level of the at least one contaminant, the at least one contaminant comprising nitrogen containing compounds, sulfur containing compounds, and metals.

20. The process of claim 14 wherein the at least one fraction is processed by at least one of hydrotreating, hydrocracking, fluid catalytic cracking, alkylation, and transalkylation.