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Sze et al.

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[54] **COAL LIQUEFACTION**

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[52] **U.S. Cl.** **208/8,** 208/10, 208/180

[51] **Int. Cl.** **G10g 1/04**

[58] **Field of Search** 208/8, 10, 179, 180

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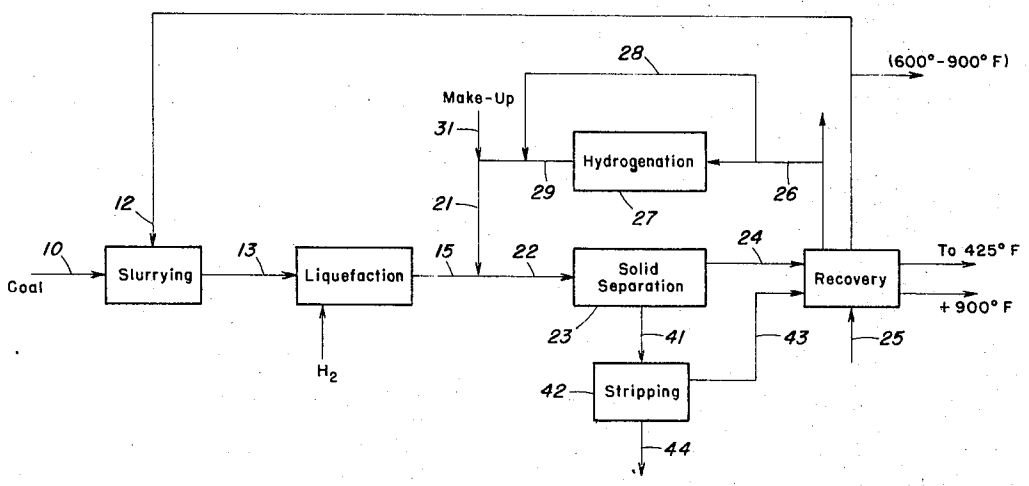
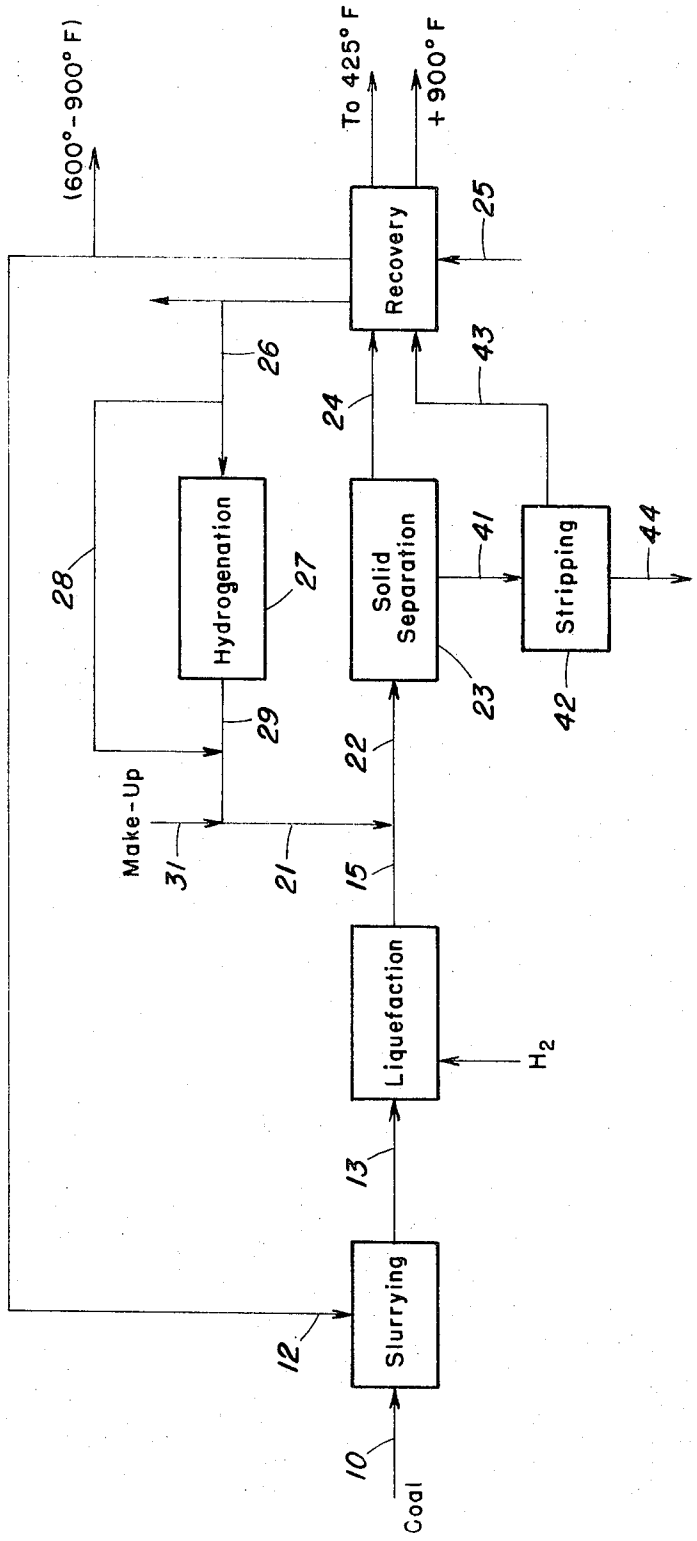
[57] **ABSTRACT**

Insoluble material is separated from a coal liquefaction product by use of a promoter liquid prepared from a fraction of the coal liquefaction product. The promoter liquid is prepared from a fraction having a 5 volume percent distillation temperature of at least 250°F. preferably at least 400°F. and a 95 volume percent distillation temperature of at least 350°F. and no greater than 750°F. by hydrogenating the fraction to raise the characterization factor thereof to at least 9.75.

14 Claims, 1 Drawing Figure




(600°-900° F)
To 425° F
+900° F
Recovery
25
26
43
24
Hydrogenation
27
Solid Separation
41
Stripping
44
28
23
42
29
22
Make-Up
31
21
15
Liquefaction
H_2
13
Slurrying
12
10
Coal

COAL LIQUEFACTION

This invention relates to the liquefaction of coal, and more particularly to the de-ashing of a coal liquefaction product.

Coal can be converted to valuable products by subjecting coal to solvent extraction, with or without hydrogen, to produce a mixture of coal extract and undissolved coal residue, including undissolved extractable carbonaceous matter, fusain and mineral matter or ash.

The finely divided mineral matter or ash and unreacted coal must be separated from the coal extract, and in general, this separation step has been the principal draw-back to the successful operation of a coal extraction process. The fine particle sizes encountered in coal solvation processes create numerous difficulties in attempting to use conventional separation techniques, such as filtration, centrifugation or settling. Attempts to use filtration techniques have not been particularly successful as a result of plugging of the filter pores with or without a precoat and the expense involved in providing the required filtration area.

Gravity settling techniques have also met with limited success as a result of low settling rates and inefficient ash removal. Centrifugation techniques have also been generally unsuccessful as a result of high cost and the difficulty in separating the lighter finely divided materials.

Accordingly, there is a need for an effective process for separating finely divided insoluble material from a coal liquefaction product.

An object of the present invention is to provide for improved coal liquefaction.

Another object of the present invention is to provide a new and improved process for separating finely divided insoluble material from a coal liquefaction product.

A further object of the present invention is to provide a process for separating insoluble material from a coal liquefaction product which does not require a filtration step.

Still another object of the present invention is to provide an improved gravity settler for coal deashing.

These and other objects of the present invention should be more readily apparent from reading the following detailed description thereof with reference to the accompanying drawing wherein:

The drawing is a simplified schematic flow diagram of a coal liquefaction process incorporating the teachings of the present invention.

The objects of the present invention are broadly accomplished, in one aspect, by use of a liquid which is indigenous to the coal liquefaction process, which promotes and enhances the separation of insoluble material from a coal liquefaction product. More particularly, the coal liquefaction product, comprised of a liquid coal extract of dissolved carbonaceous matter in a coal liquefaction solvent and insoluble material (ash and unreacted coal) is mixed with a liquid promoter having an aromaticity less than that of the liquefaction solvent to enhance and promote the separation of insoluble material and provide a liquid coal extract essentially free of insoluble material. The promoter liquid is produced by controlled hydrogenation of a fraction of the coal liquefaction product to provide the required aromaticity.

The liquid which is employed to enhance and promote the separation of insoluble material from the coal liquefaction product is generally a hydrocarbon liquid having a characterization factor (K) of at least about 9.75 wherein:

$$K = \sqrt[3]{T_B}/G$$

wherein T_B is the molal average boiling point of the liquid (°R); and G is specific gravity of the liquid (60°F./60°F.).

The characterization factor is an index of the aromaticity/parafinicity of hydrocarbons and petroleum fractions as disclosed by Watson & Nelson *Ind. Eng. Chem.* 25 880 (1933), with more parafinic materials having higher values for the characterization factor (K). The promoter liquid which is employed is one which has a characterization factor (K) in excess of 9.75 and which is also less aromatic than the liquefaction solvent; i.e., the characterization factor K of the promoter liquid has a value which is generally at least 0.25 unit higher than the characterization factor of the liquefaction solvent.

The following Table provides representative characterization Factors (K) for various materials:

Table

Anthracene	8.3
Naphthalene	8.4
425–500°F. Coal Tar Distillate	8.8
550–900°F. Coal Tar Distillate	9.1
600–900°F. Coal Tar Distillate	9.0
400–450°F. Coal Tar Distillate	9.4
Benzene	9.8
Tetrahydronaphthalene	9.8
o-xylene	10.3
Decahydronaphthalene	10.6
Cyclohexane	11.0
425–500°F. Boiling Range Kerosene	11.9
n-Dodecyclbenzene	12.0
Propylene Oligomers (pentamer)	12.2
Cetene	12.8
Tridecane	12.9
n-Hexane	12.9
Hexadecane or cetane	13.0

The liquid which is used to enhance and promote the separation of insoluble material is further characterized by a 5 volume percent distillation temperature of at least about 250°F. and a 95 volume percent distillation temperature of at least about 350°F. and no greater than about 750°F. The promoter liquid preferably has a 5 volume percent distillation temperature of at least about 310°F. and most preferably of at least about 400°F. The 95 volume percent distillation temperature is preferably no greater than about 600°F. The most preferred promoter liquid has a 5 volume percent distillation temperature of at least about 425°F. and a 95 volume percent distillation temperature of no greater than about 500°F. The 5 volume and 95 volume percent distillation temperature may be conveniently determined by ASTM Test No. D 86–67 or No. D 1160 with the former being preferred for those liquids having a 95 volume percent distillation temperature below 600°F. and the latter for those above 600°F. The methods for determining such temperatures are well known in the art and further details in this respect are not required for a fully understanding of the invention. It is also to be understood that the reported temperatures are corrected to atmospheric pressure.

The amount of liquid promoter used for enhancing and promoting the separation of insoluble matter from

the coal liquefaction product will vary with the particular liquid employed, the coal liquefaction solvent, the coal used as starting material and the manner in which the liquefaction is effected. As should be apparent to those skilled in the art, the amount of liquid promoter used should be minimized in order to reduce the overall costs of the process. It has been found that by using the liquid of controlled aromaticity, in accordance with the teachings of the present invention, the desired separation of insoluble material may be effected with modest amounts of liquid promoter. In general, the weight ratio of liquid promoter to coal solution may range from about 0.2:1 to about 4.0:1, with the particular amount which is employed being dependent upon the characterization factor (K) of the promoter liquid, with promoter liquids of the present invention having lower characterization factors generally requiring greater amounts of promoter liquid. In accordance with the present invention, in general, the hydrogenation of the fraction recovered from the coal extract to produce the promoter liquid is not effected to a degree in which the characterization factor (K) is above about 11.0; i.e., in general the characterization factor is from about 9.75 to about 11.0. At such characterization factors, the liquid promoter is generally used in an amount from about 1:1 to about 4:1 and preferably **850**°about 1.5:1 to about 3.0:1.

It is to be understood, however, that greater amounts of liquid promoter may be employed, but the use of such greater amounts is uneconomical. In addition, the use of an excess of liquid promoter may result in a precipitation or separation of an excessive amount of desired coal derived products from the coal extract. More particularly, as the amount of liquid promoter employed is increased, a greater amount of ash containing under flow is separated from the coal solution, but such an increased separation is accompanied by an increased separation of desired coal derived products from the coal solution. By using the liquid promoters of the present invention, not only may modest amounts of solvent be employed, but, in addition, ash may be effectively separated from the coal solution; e.g., in amounts greater than 99 percent, without an excessive loss of desired coal derived products.

More particularly, coal, such as bituminous coal, on a moisture ash free basis (MAF) may contain from about 5 to about 10 percent of insoluble material, such as fusain, and accordingly, at a minimum, from about 5 to about 10 percent, of the MAF coal, is lost in the process. In the recovery of coal derived products by a solvation process, the potential product loss is measured by the amount of 850°F+ product which is not recovered from the coal in that it is this fraction, which includes insoluble coal material, such as fusain, which can not be recovered from the residual solid product of the coal deashing. In accordance with the present invention, on a MAF coal feed basis, product loss of 850°F+ components (on an ash free basis) can be maintained at a value of no greater than about 40 percent, by weight, and preferably no greater than about 25 percent, by weight. In general, the loss of 850°F+ products, on a MAF coal basis, is from about 10 to about 25 percent, by weight. In addition, the net coal product (the extracted carbonaceous matter, excluding promoter liquid, liquefaction solvent and gas make), hereinafter sometimes referred to as "coal product," contains less than about 1 percent insoluble material, generally less than 0.1 percent insoluble material and most preferably less than 0.05 percent insoluble material, all by weight. The specific amount of insoluble material which is permitted to be present in the coal product is dependent upon product standards, and the deashing is controlled in order to provide the required specifications. Based on an Illinois type coal, the production of a coal product having less than 0.05 percent, by weight, insoluble material, corresponds to 99+ percent ash removal, but as should be apparent to those skilled in the art, the percent ash removal to provide a coal product having the required minimum amount of insoluble material is dependent upon the initial ash content of the coal. Thus, in accordance with the present invention, the liquid promoter is added to the coal solution in an amount, as hereinabove described, to provide a coal product in which insoluble material is present in an amount of less than about 1 percent, by weight, and most preferably of less than 0.05 percent, by weight, with the loss of 850°F+ product being from about 10 to about 40 percent, by weight, preferably from about 10 to about 25 percent, by weight, on a MAF coal feed basis; i.e., from about 60 to about 90 percent, by weight, of the MAF coal feed is recovered as either gas make or liquid fuel product.

The promoter liquid which is used to promote the separation of insoluble material, as hereinabove described, is indigenous to the process and, accordingly, such promoter liquid is produced from the essentially solid free coal extract. More particularly, a fraction having a 5 volume percent distillation temperature and a 95 volume percent distillation temperature which is required for the promoter liquid is recovered from the coal extract product as known in the art; e.g., by fractional distillation. In general, such a fraction is in excess of that required for producing the promoter liquid (product having 5 volume percent and 95 volume percent distillation temperatures identical to that of the promoter liquid is continuously produced from the liquefaction of coal) and, accordingly, only a portion of this fraction is used for producing the promoter liquid, with the remaining portion constituting a portion of the net liquefaction product.

The indigenous fraction which is to be ultimately employed as the promoter liquid must then be subjected to a controlled hydrogenation in order to produce a promoter liquid having a controlled aromaticity; i.e., a characterization factor K in excess of 9.75. In accordance with the present invention, all or a portion of the fraction is hydrogenated, as known in the art, in order to produce a promoter liquid having the required characterization factor K. In general, such a hydrogenation is effected at a temperature from about 450°F. to about 8850°F. and a pressure of from about 400 psig to about 2,000 psig in the presence of a suitable hydrogenation catalyst, such as nickel tungsten sulfide, nickel molybdate, nickel molybdenium sulfide and the like. It is to be understood, however, that the present invention is not to be limited to such conditions in that the conditions of hydrogenation form no part of the present invention, except insofar as they are used to provide a promoter liquid having the required characterization factor K. The hydrogenation of liquids is well known in the art and, accordingly, those skilled in the art can readily select those conditions required to produce the required characterization factor K.

The promoter liquid produced by hydrogenation of all or a portion of the fraction of the coal liquefaction product recovered for production of the promoter liquid may then be employed to promote and enhance the separation of solid material from the coal liquefaction product.

It is also to be understood that the indigenous promoter liquid of controlled aromaticity may be employed in combination with a promoter liquid from an external source, such as a kerosene fraction, provided that the resulting blend has a characterization factor as hereinabove described.

The separation of the insoluble material from the coal extract is generally effected at a temperature from about 300°F. to about 600°F., preferably from about 350°F. to about 500°F., and at a pressure from about 0 psig to about 500 psig, preferably at a pressure from about 0 psig to about 300 psig. It is to be understood that higher pressures could be employed, but as should be apparent to those skilled in the art, lower pressures are preferred. The insoluble material is preferably separated by gravity settling with the essentially insoluble material free coal extract being recovered as an overflow and the insoluble material as underflow. In such gravity settling, the amount of underflow should be minimized in order to minimize the loss of heavier products in the underflow. The underflow withdrawal rate to obtain desired results is deemed to be within the scope of those skilled in the art. In general, such a rate is from about 20 to about 25 wt. percent of the total feed (liquefaction product and promoter liquid). The residence time for such settling is generally in the order of from about 0.5 to about 6 hours, and preferably from about 0.5 to 3.0 hours.

The invention will be further described with respect to an embodiment thereof illustrated in the accompanying drawing. It is to be understood, however, that the scope of the invention is not to be limited thereby.

Referring to the drawing, ground or pulverized coal, generally bituminous, sub-bituminous or lignite, preferably bituminous coal, in line **10** is introduced into a coal solvation and slurrying zone **11** along with a coal liquefaction solvent in line **12.** The coal liquefaction solvent may be any one of the wide variety of coal liquefaction solvents used in the art, including both hydrogen donor solvents, non-hydrogen donor solvents and mixtures thereof. These solvents are well known in the art and, accordingly, no detailed description thereof is deemed necessary for a full understanding of the invention. As particularly described, the coal liquefaction solvent is a 600°F.–900°F. solvent which is recovered from the coal liquefaction product and which has not been subjected to hydrogenation subsequent to the recovery thereof. The solvent is added to the coal in an amount sufficient to effect the desired liquefaction, and in general, is added in an amount to provide a solvent to coal weight ration from about 1:1 to about 20:1, and preferably from about 1.5:1 to about 5:1.

A coal paste is withdrawn from zone **11** through line **13** and introduced into a coal liquefaction zone **14** wherein, as known in the art, the coal is converted to liquid products. The liquefaction zone **14** is operated as known in the art and may be catalytic or non-catalytic and may be effected in the presence or absence of added hydrogen. The hydrogenation may be effected in a fixed catalyst bed, fluidized catalyst bed or in an expanded or ebullating bed. The details of the coal liquefaction step form no part of the present invention and, accordingly, no details thereof are required for a full understanding of the invention. As particularly described, the coal liquefaction is effected in the presence of added hydrogen. The hydrogenation, as known in the art, increases the recovery of coal products and also reduces the sulfur and nitrogen content of the recovered liquid coal product. The liquefaction is preferably effected in an upflow ebullated bed, as known in the art; e.g., as described in U.S. Pat. No. 2,987,465 to Johanson. The coal liquefaction zone, as known in the art, includes means for recovering the various gaseous products.

A coal liquefaction product, comprised of a liquid coal extract of dissolved carbonaceous matter in the coal liquefaction solvent and insoluble material (ash and undissolved coal) is withdrawn from the liquefaction zone **14** through line **15** and mixed with promoter liquid in line **21** of controlled aromaticity, i.e., the characterization factor of the promoter liquid has a value which is generally at least 0.25 unit greater than the characterization factor of the coal liquefaction solvent. As particularly described, the promoter liquid is a fraction which has 5 volume percent and 95 volume percent distillation temperatures which fall within the range from about 425°–500°F.

The combined stream of coal liquefaction product and promoter liquid in line **22** is introduced into a gravity separation zone **23,** comprised of a gravity settler which may be any one of those known in the art, wherein an essentially solids free overflow is separated from a solid containing underflow. Although the gravity settler may be any one of those generally known in the art, the settler is preferably of a special type developed for the present invention as described in copending application Ser. No. 304,319 filed concurrently herewith.

The overflow, essentially free of insoluble material, is withdrawn from separation zone **23** through line **24** and introduced into a recovery zone **25** for recovering various fractions of the coal extract. The recovery zone **25** may be comprised of one or more fractionators to distill various fractions from the product. As particularly described, the recovery zone is operated to recover a first fraction boiling up to about 425°F.; a second fraction having 5 and 95 percent volume distillation temperatures of from about 425° to about 500°F.; a third fraction (500°–600°F.) which may be employed as a distillation fuel blendstock; a fourth fraction (600°–900°F.) a portion of which may be used as the coal liquefaction solvent in line **12** and a further portion of which may be recovered as product, and a residual product (+900°F.) of low ash and reduced sulfur content which may be used as fuel or subjected to further treatment. It is to be understood, however, that the present invention is not limited to the above representative separation in that the different fractions may be recovered as products or as the fraction to be used as a promoter liquid. The promoter liquid, however, is preferably derived from a fraction having a 5 volume distillation temperature of no lower than about 425°F. and a 95 volume percent distillation temperature of no greater than about 500°F.

The portion of the fraction (425°–500°F.) required to meet the promoter liquid requirements for the process is passed through line **26** and at least a portion thereof is introduced into a hydrogenation zone **27,** with the

remaining portion, if any, being bypassed through line **28.** The hydrogenation zone **27,** as hereinabove described, is operated to provide a promoter liquid with the required characterization factor; i.e., a blend of the hydrogenated portion and the by-passed protion has the required characterization factor. A hydrogenated product is withdrawn from hydrogenation zone **27** through line **29,** combined with any liquid in line **28** and the combined stream in line **21** employed as the promoter liquid. Start-up liquid or make-up blending liquid may be added through line **31.**

The underflow containing dispersed insoluble material withdrawn from separation zone **23** through line **41** is introduced into a stripping zone **42** wherein material boiling below about 900°F. is stripped therefrom and introduced into the recovery zone **25** through line **43.** The ash rich stripper bottoms in line **44** may then be subjected to calcination or coking. Alternatively, part of the stripper bottoms may be used as feedstock to a partial oxidation process for producing hydrogen. As a further alternative a portion of the stripper bottoms may be used for plant fuel. These uses and other should be apparent to those skilled in the art from the teachings herein. In accordance with the present invention, the stripper bottoms in line **44** contains from about 10 to about 40 percent, by weight, of the MAF coal.

The invention will be further described with respect to the following example which further illustrates the present invention. Unless otherwise indicated all parts and percents are by weight.

EXAMPLE

An aromatic distillate fraction is obtained from a coal liquefaction product. It exhibits a characterization factor of 8.8 and 5 volume percent and 95 volume percent distillation temperature range of 425°–500°F. It is pumped through a preheater admixed with preheated hydrogen and fed to a fixed bed catalytic reactor packed with a commercial sulfided nickel-tungsten catalyst. The hydrogen stream purity is 75% H_2. The following reaction parameters are used: LHSV 1.0hr^{-1}, an operating pressure of 1,400 psig, and an inlet temperature of 650°F. Hydrogen is fed to the reactor at a rate equivalent to 400 SCF of hydrogen per gallon of liquid feedstock.

Reactor effluent is quickly cooled to 200° ± 10°F, and routed to a high pressure gas/liquid separator which vents gas continuously under automatic pressure control. At the conclusion of the run the high pressure is vented down to atmospheric pressure and a liquid product with a characterization factor of 9.9 is withdrawn.

This hydrogenated liquid is then used to remove ash from a coal liquefaction product comprised of insoluble material and carbonaceous matter dissolved in a coal liquefaction solvent (600°–900°F coal tar distillate). 1,200 grams of this promoter solution and 300 grams of coal liquefaction product are added to a 2 liter rocking bomb made of stainless steel and electrically heated. The contents in the bomb is heated with rocking to 500°F over about 30 minute period. Bomb contents are then allowed to settle vertically for about 4 hours at 500°F without any rocking. At the end of the settling period, 250 grams of an ash-rich underflow stream are withdrawn through the bottom valve. The remainder of the bomb's contents is withdrawn through the bottom valve and this ash-lean solution solution contains 0.01 wt. percent solids, which corresponds to an ash removal of 98+ percent.

The present invention is particularly advantageous in that insoluble materials can be separated from a coal liquefaction product without requiring filtration. In addition, by proceeding in accordance with the present invention ash and insoluble material separation can be maximized with minimum loss of desired coal derived products and with modest amounts of promoter liquid. The effectiveness of promoter liquid as used in the present invention is further illustrated in copending application Ser. No. 304,319, which is hereby incorporated by reference. Moreover, such separation is effected with materials which are indigenous to the process which facilitates the overall operation.

Although the prior art, in particular U.S. Pat. No. 3,607,716, discloses a process for separating insoluble material from a coal liquefaction product, without filtration, by use of a fractionating solvent, such as hexane, as hereinabove described, the ash removal with such light fractionating solvents is accompanied by a loss of desired coal derived products. In addition, such a process requires operating pressures higher than those required in the present invention which increases processing costs.

Numerous modifications and variations of the present invention are possible in light of the above teachings and, therefore, within the scope of the appended claims the invention may be practised other than as particularly described.

What is claimed is:

1. A process for separating insoluble material from a coal liquefaction product produced from a coal feed and comprised of insoluble material and a coal solution of carbonaceous matter dissolved in a coal liquefaction solvent, comprising:

a. introducing said coal liquefaction product and a liquid promoter into a gravity settling zone, said liquid promoter having a 5 volume percent distillation temperature of at least about 250°F., and a 95 volume percent distillation temperature of at least about 350°F. and no greater than about 750°F., said liquid promoter having a characterization factor (K) of at least 9.75, said liquid promoter having a characterization factor greater than said coal liquefaction solvent and being added in an amount sufficient to produce a coal extract essentially free of insoluble material;

b. recovering by gravity settling as overflow a coal extract essentially free of insoluble material;

c. separating from said coal extract a fraction having a 5 volume distillation temperature of at least about 250°F. and a 95 volume distillation temperature of at least about 350°F. and no greater than about 750°F.;

d. hydrogenating at least a portion of said fraction to produce a liquid promoter having a characterization factor greater than the characterization factor of said coal liquefaction solvent, said characterization factor being at least 9.75; and

e. employing liquid promoter from step (d) in step (a).

2. The process of claim **1** wherein the weight ratio of liquid promoter to coal solution is from about 0.2:1 to about 4.0:1.

3. The process of claim 2 wherein said promoter liquid has a 5 volume percent distillation temperature of at least about 400°F.

4. The process of claim 1 wherein the characterization factor *K* of the promoter liquid is no greater than about 11.0.

5. The process of claim 4 wherein the weight ratio of liquid promoter to coal solution is from about 1:1 to about 4:1.

6. The process of claim 5 wherein said promoter liquid has a 5 volume percent distillation temperature of at least about 400°F.

7. The process of claim 1 wherein the ratio of liquid promoter to coal solution is from about 0.2:1 to about 4.0:1 and in an amount to provide a coal product from said coal feed containing less than about 0.05 percent, by weight, of insoluble material and a coal residue contain no greater than about 40 percent, by weight, of the moisture ash free coal as an ash free +850°F. fraction.

8. The process of claim 1 wherein the gravity settling is effected at a temperature from about 300°F. to about 600°F and a pressure from about 0 psig to about 500 psig.

9. The process of claim 1 wherein said fraction and promoter liquid have a 5 volume percent distillation temperature of no less than about 425°F. and a 95 volume distillation temperature of no greater than about 500°F.

10. The process of claim 1 wherein the 95 volume percent distillation temperature of said fraction and promoter liquid is no greater than about 600°F.

11. The process of claim 7 wherein the gravity settling is effected at a temperature from about 300°F. to about 600°F. and a pressure from about 0 psig to about 500 psig.

12. The process of claim 11 wherein the characterization factor of said liquid promoter is no greater than about 11.0.

13. The process of claim 12 wherein the weight ratio of liquid promoter to coal solution is from about 1:1 to about 4:1.

14. The process of claim 13 wherein said fraction and promoter liquid have a 5 volume percent distillation temperature of no less than about 425°F and a 95 volume percent distillation temperature of no greater than about 500°F.

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