Feb. 12, 1974 E. GORIN ET AL 3,791,956

CONVERSION OF COAL TO CLEAN FUEL

Filed Feb. 16, 1973 3 Sheets-Sheet 1

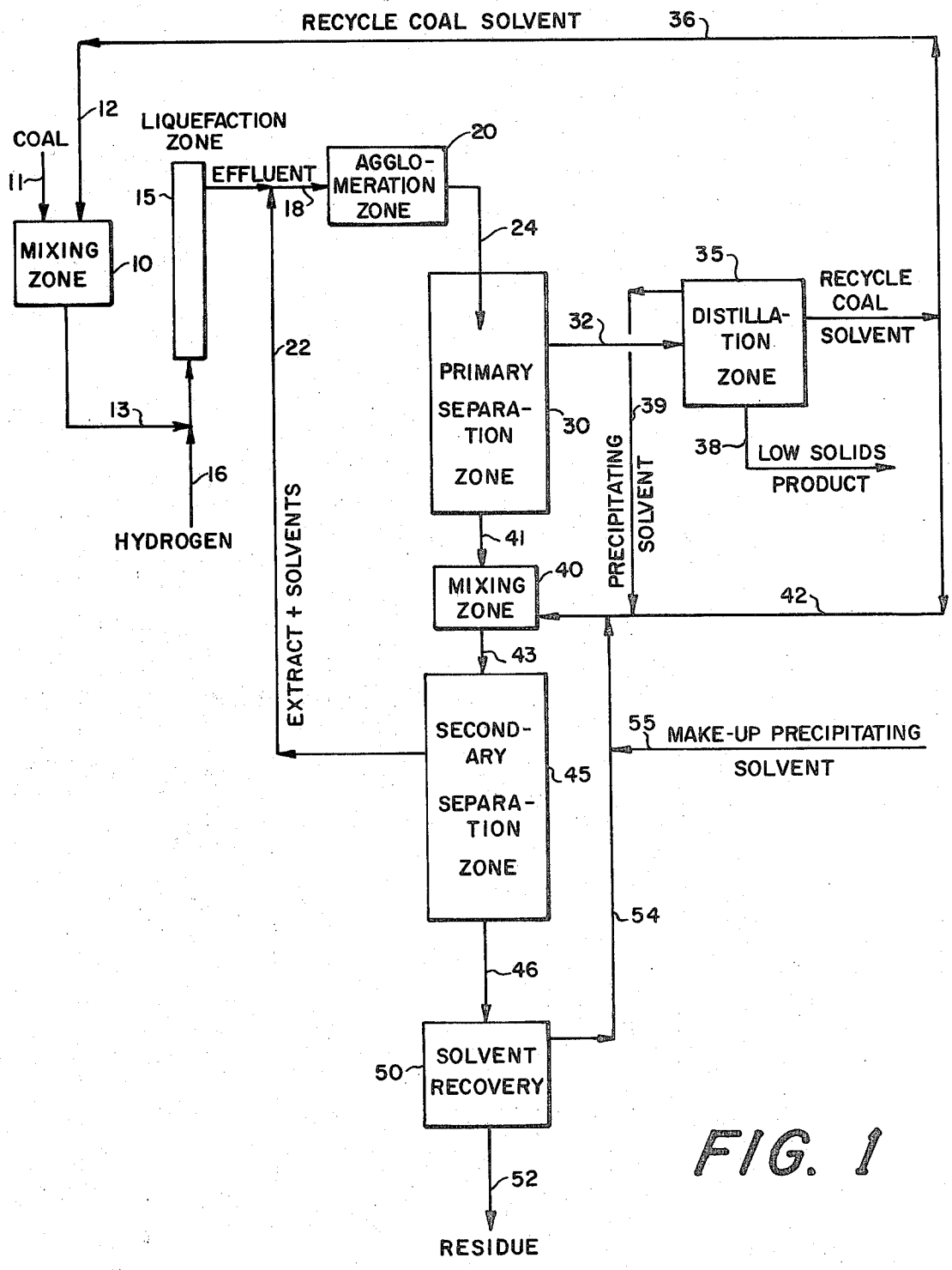


FIG. 1

CONVERSION OF COAL TO CLEAN FUEL

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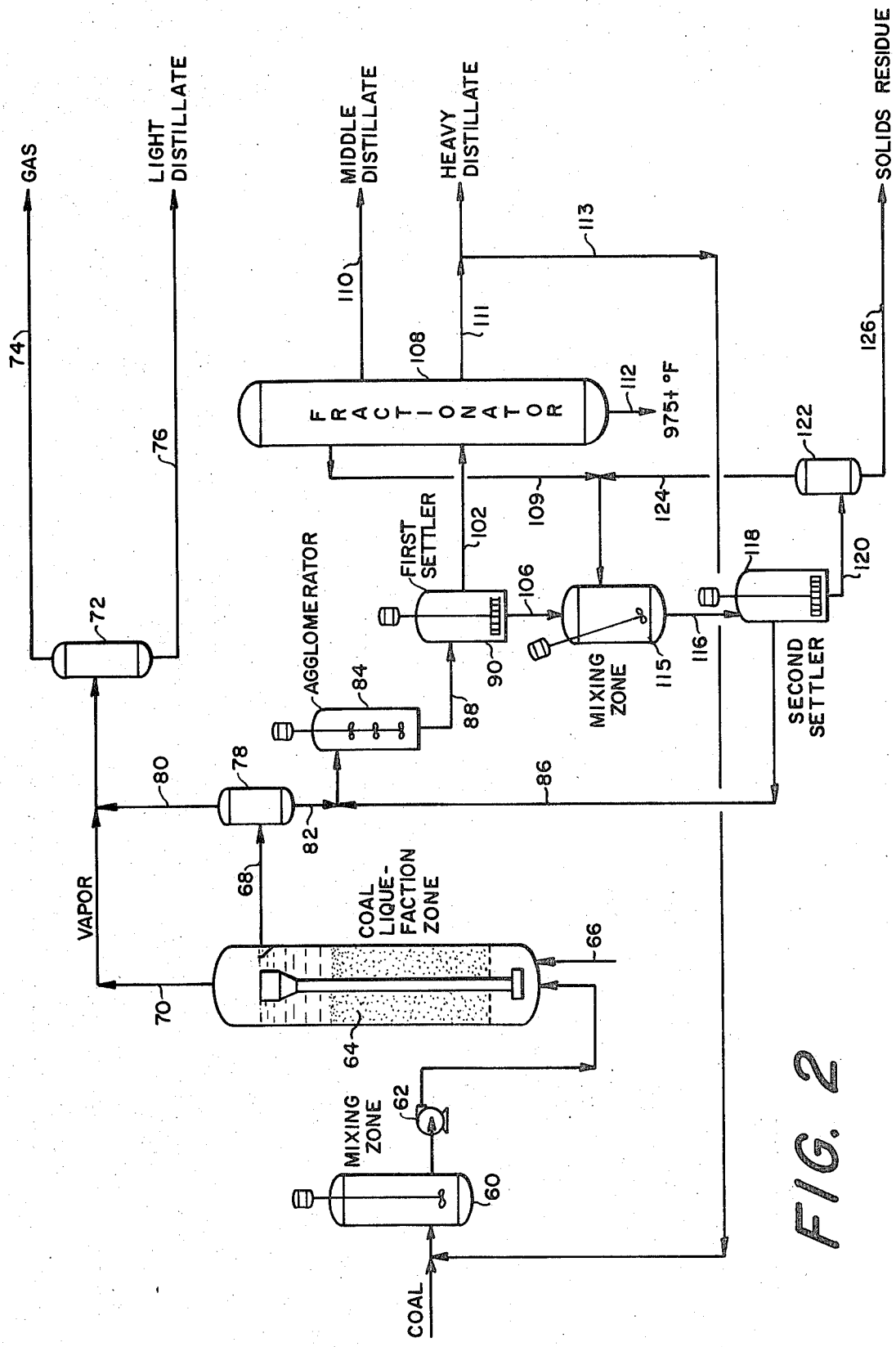


FIG. 2

CONVERSION OF COAL TO CLEAN FUEL

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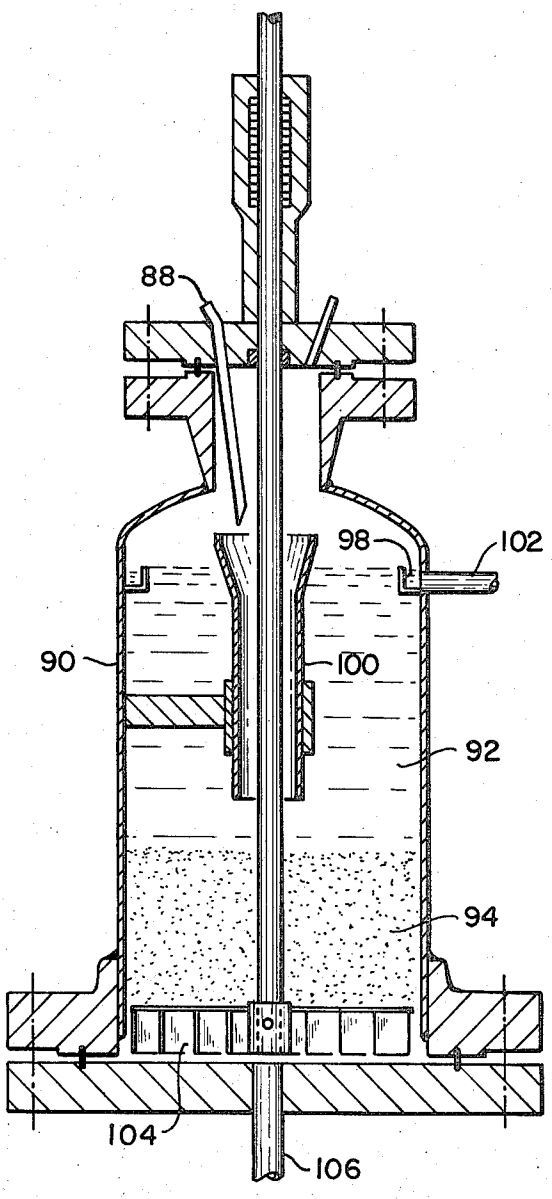


FIG. 3

United States Patent Office 3,791,956 Patented Feb. 12, 1974

3,791,956

CONVERSION OF COAL TO CLEAN FUEL

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Filed Feb. 16, 1973, Ser. No. 333,097
Int. Cl. C10g 1/00
U.S. Cl. 208—8 2 Claims

ABSTRACT OF THE DISCLOSURE

Production of a low mineral content fuel by coal liquefaction processes which yield as their primary product a mixture of liquid and solids, a part of which is suspended in the liquid. The suspended solids are effectively removed with minimum loss of desired product by means of a precipitating solvent which serves three functions, (1) to effect agglomeration of the suspended fines, (2) to prevent redispersal of the fines in the washing operation, and (3) to recover desired product from the rejected solids.

BACKGROUND OF THE INVENTION

This invention relates to processes for the conversion of coal to a clean fuel, that is, a fuel which is substantially free of the mineral components normally found in coal.

In particular, the invention relates to coal liquefaction processes wherein a solvent (hereinafter sometimes called liquefaction solvent) is present during the liquefaction of the coal. Liquefaction may be achieved by hydrogenation, depolymerization, extraction, etc. The liquefaction solvent, which is generally coal-derived, may function as solvent for the coal or for the products, or both. It may also play a reactive role, for instance, in the depolymerization of the coal molecules. Examples of such coal liquefaction processes are described in U.S. Pats. Nos. 3,018,242; 3,117,921; 3,143,489; 3,158,561; 3,523,886; Re. 25,770; and 3,321,393.

The primary product of such coal liquefaction processes is a mixture of liquid and undissolved solids. Some gas is generally also produced. The liquid is a solution of coal liquefaction products dissolved in the liquefaction solvent. Most of the undissolved solids may be readily separated from the liquid by conventional solids-liquids separation processes such as filtration, centrifugation, sedimentation, hydroclones, etc. However, a part of the undissolved solids (the amount being a function of the particular coal and the particular liquefaction treatment) appears as extremely finely divided particles of the order of ten microns or less in size. These particles are rich in mineral matter normally found in all coals. Upon combustion of the fuel which contains them, they form ash.

Complete separation of such finely divided particles from the liquid in which they are suspended can not be accomplished by the usual mechanical separation techniques at ordinary temperatures (i.e. by filtration, centrifugation, settling, or hydroclones) because of the extremely fine state of subdivision of the solid particles and because of the high viscosity of the liquid. Separation is improved by operation at elevated temperatures due to a rapid decrease in liquid viscosity, as well as an increase in the density differential between liquid and solid. Even at these elevated temperatures and reduced viscosities, the conventional separation techniques may be only partially effective.

The prior art offers many solution to the problem of separation described above. Those which are pertinent to the present invention are those which use a precipitating solvent (sometimes called an anti-solvent) to effect agglomeration of the finely divided solids, thereby permitting use of conventional solids-liquid separation techniques. Illustrative of the prior art pertinent to such agglomeration are the following U.S. Pats.: 2,060,447; 2,631,982; 2,774,716; 2,871,181; 2,964,460; 2,989,458; 3,010,893; 3,018,241; 3,275,546; 3,519,553; 3,607,716; 3,607,717; 3,607,718; 3,607,719; 3,635,814; 3,642,608; and 3,687,837. The primary objective of the present invention is maximum recovery of a product which is substantially free of mineral matter with minimum load imposed upon the distillate recovery equipment which provides the required liquefaction solvent and precipitating solvent.

SUMMARY OF THE INVENTION

The present invention is an improvement in the separation step of a coal liquefaction process which yields a primary product containing, as a first component in a liquid state, the solution of liquid product in the liquefaction solvent and, as the other and second component, the undissolved solids which are suspended in the first component and are not readily separable therefrom. It is with the separation of this second component that the present invention is primarily concerned, and not with the separation of the coarser, readily separable solids which reprevention is primarily concerned, and not with the separationof the coarser, readily separable solids which represent, in effect, a third component. The latter may be separated from the first component when ever it is convenient or desirable to do so, by conventional techniques.

The improvement embodying the present invention is the use of a precipitating solvent in such a way as to provide effective separation of the second component while at the same time permitting maximum recovery of the desired product from the coal liquefaction step.

The improvement in such separation comprises the following steps:

(1) adding a precipitating solvent to the mixture of the first and second components in an amount at least sufficient to cause precipitation of a deposit from the first component upon the second component, and agitating the resulting mixture while maintaining the temperature and pressure sufficiently high to keep the mixture fluid, to thereby effect agglomeration of the second component;

(2) separating the agglomerates of the second component from the mixture of the first component and the precipitating solvent in a primary separation zone;

(3) recovering the precipitating solvent from its admixture with the first component in a distillation zone;

(4) washing the agglomerates of the second component which are withdrawn from the primary separation zone in admixture with some of the first component with the precipitating solvent recovered in step (3) to recover said first component while preventing redispersion of the agglomerates;

(5) separating the washed agglomerates from precipitating solvent and recovered first component in a secondary separation zone; and

(6) recycling precipitating solvent and recovered first component to said agglomeration zone of step (1).

By virtue of the foregoing improvement, the precipitating solvent serves three purposes. Firstly, it assists directly in the agglomeration of the second component, thereby making the second component separable from the first component. Secondly, it serves to prevent redispersion of the agglomerates in the first component during the washing and subsequent final separation step. And thirdly, it serves to recover the first component which is withdrawn with the agglomerates from the primary separation step. Such triple use of the precipitating solvent minimizes the flow in the solvent recovery circuit and reduces the distillation equipment required.

DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic flowsheet of the present invention in its broadest aspects;

FIG. 2 is a schematic flowsheet of the preferred embodiment of the present invention; and

FIG. 3 is a cross-sectional view of the settler used in the preferred embodiment.

THE INVENTION (in its broadest aspects)

Referring to FIG. 1, finely divided coal and liquefaction solvent are introduced into a stirred Mixing Zone **10** through lines **11** and **12** respectively. The solvent-to-coal weight ratio is generally between 1 and 4.

Coal as used herein means any form of naturally occurring solid, ash-containing, hydrocarbonaceous substance, and includes, by way of example, bituminous and sub-bituminous coals, anthracite, and lignites.

A suitable liquefaction solvent is a mixture of polycycloc, aromatic hydrocarbons which is liquid under the conditions of temperature and pressure maintained during coal liquefaction. A suitable normal boiling range for such a solvent for example, is within the range 250 to 425° C. The solvent may be conveniently derived as a distillate fraction in the overall coal liquefaction process; in other words, from coal itself. Preferably, at least, a portion of the aromatic hydrocarbons is hydrogenated to provide a hydrogen transfer solvent.

The mixture of coal and liquefaction solvent in slurry form is transferred through a conduit **13** to a Coal Liquefaction Zone **15**. The liquefaction process used in the Liquefaction Zone may be any of the processes commonly used by those skilled in the art, exemplified by those described in the above-cited patents. The selected process may be continuous, batch, countercurrent or staged, and may use fixed beds, fluidized beds or ebullating beds, for example. The temperature at which the coal liquefaction is conducted generally lies between 300 and 500° C. The pressure may be selected over a wide range, e.g. between 1 and 6500 p.s.i.g. The residence time, too, varies widely, but is generally in the range of 1 to 120 minutes, being dictated by the other conditions as well as the desired extent of liquefaction. To attain depths of coal conversion between 50 and 80 percent or so of the moisture-and-ash-free coal, the addition of hydrogen is generally required, either by hydrogen transfer from the liquefaction solvent or extraneously, or both. As shown in FIG. 1, gaseous hydrogen may be introduced into the Liquefaction Zone through line **16**.

After liquefaction of the coal has been satisfactorily achieved, the effluent slurry product is removed from the Liquefaction Zone **15** through a conduit **18**. At this point, this product consists essentially of a solution of the coal liquefaction product in the liquefaction solvent, and the undissolved solids. The solids are made up of coarse and fine particulate solids, the latter being difficult to separate from their suspension in the liquid medium, even at high temperatures where the viscosity of the liquid is less. Generally, no attempt is made to separately remove the readily separable coarse solids since each such separation entails added cost and some loss of desired product, and the larger particles may actually aid in the removal of the smaller particles. The product from the Coal Liquefaction Zone is transferred by conduit **18** directly and without intentional cooling to an Agglomeration Zone **20**. Although not shown, gases and low boiling components are generally removed before introduction into the Agglomeration Zone.

The primary function of the Agglomeration Zone **20** is to effect agglomeration of the very finely divided solids which are suspended in the liquid. The desired agglomeration is accomplished by mixing a precipitating solvent which is miscible with the liquid coal liquefaction product (sometimes called herein "first component"). A suitable precipitating solvent is an aliphatic or naphthenic hydrocarbon or a mixture of aliphatic or naphthenic hydrocarbons. Generally, a precipitating solvent is selected which normally boils within the temperature range of 75 to 200° C., although other solvents may work as well. The selection of precipitation solvent depends also on economic factors such as cost of separation from liquefaction solvent and type of solids separator. The precipitating solvent is miscible with the liquefaction solvent but does not readily dissolve the benzene-insoluble components of the coal liquefaction product. Hence, when added in sufficient amount under proper conditions to the coal liquefaction product, a precipitate is formed. The required precipitating solvent is added by a conduit **22** which is connected to the transfer conduit **18**.

Agglomeration of the finely divided solids (sometimes herein called "second component") is effected in the Agglomeration Zone **20** under the following conditions. The temperature is maintained between 250 and 370° C. by any suitable means. The pressure is maintained sufficiently high to be above the initial boiling point of the liquid to prevent loss of the precipitating solvent and is generally between 5 and 200 p.s.i.g.

The weight ratio of precipitating solvent to the first component generally lies between 0.05 and 0.5. By thus regulating the conditions in the Agglomeration Zone, a controlled amount of high molecular weight hydrocarbonaceous material, largely benzene-insolubles, is precipitated. The mixture is stirred very vigorously so that the precipitated material is uniformly dispersed and distributed over the finely divided solids to serve, apparently, as a binder in the formation of agglomerates. Care must be taken to allow sufficient time for the desired agglomeration to occur. Generally, a minimum time of about 5 minutes is required. However, the minimum time required is a function of the other variables as well as the particular composition of the coal liquefaction product.

The effluent mixture of liquid and solids, including the agglomerates from the Agglomeration Zone, together with precipitating solvent, is conducted by a conduit **24**, without intentional cooling, to a Primary Separation Zone **30**. In the broadest aspects of this invention, any suitable solids-liquid equipment may be used for effecting separation in this zone. Such equipment includes filters, centrifuges, hydroclones, and settlers, etc. Since it is desired to obtain from this zone an effluent liquid which is as free of solids as possible, the equipment, whatever it may be, should be operated to obtain an essentially solids-free liquid at the expense of some liquid being carried along or entrained with the solids. The amount of such liquid varies with the type of equipment and its operation. However, the amount of the desired coal liquefaction product accompanying the separated solids is generally at least 5 weight percent of the total coal liquefaction product and may be as much as 30 percent or more, depending upon coal liquefaction process employed.

The now-clarified liquid, i.e. the first component together with the precipitating solvent, is conducted through a conduit **32** to a Distillation Zone **35** which consists of any suitable fractionator for fractionally distilling relatively high boiling liquids. The primary function of the fractionator is to recover precipitating solvent from the clarified liquid. However, if desired, liquefaction solvent, which is generally higher boiling than the precipitating solvent, may also be recovered. It depends upon the subsequent treatment, if any, of the nondistillable portion of the clarified liquid, whether the liquefaction solvent is recovered at this point or in some subsequent treatment step which is not a part of the present invention. If such liquefaction solvent is recovered in the Distillation Zone **35**, it is recycled through conduit **36** to the Mixing Zone **10**. The desired low mineral content product is withdrawn from the Distillation Zone **35** through a conduit **38** to a point of use, or further treatment. As is, the product is useful as a fuel for power plants. With further treatment

such as hydrocracking and hydrofining, it may be converted to low boiling distillate fuels.

The distillate fraction from the Distillation Zone which constitutes the precipitating solvent is conducted by a conduit **39** to a Mixing Zone **40** where it is mixed with the second component and accompanying first component which is withdrawn from the Primary Separation Zone through a conduit **41**. The amount of precipitating solvent added to the Mixing Zone **40** is generally such as to serve three functions, to wit: (1) to prevent redispersal of agglomerates, (2) to wash the solids free of coal liquefaction product and liquefaction solvent, and (3) to effect precipitation of the required amount of benzene-insolubles in the Agglomeration Zone. The amount required in the Agglomeration Zone may include more than that required for effecting agglomeration since we have found that an excess will cause deposition of so-called "soluble ash," i.e. metallo-organo compounds which are in solution. Generally, it is the latter function that determines the amount of precipitating solvent introduced ino the Mixing Zone since, as will be seen, the washing may be effected by its own recirculating system of wash solvent. The amount required to prevent redispersal of agglomerates is relatively small, albeit most important to the successful operation of the present invention. It is usually desirable that some liquefaction solvent also be added to the Mixing Zone **40** through a conduit **42** to improve the recovery of coal liquefaction product.

The effluent slurry from the Mixing Zone **40** is conducted by a conduit **43** to a Secondary Separation Zone **45**. The latter serves to complete the washing of the solids to free them of coal liquefaction product and liquefaction solvent and to complete the separation of solids, including agglomerates. Again, any suitable solids-liquid separation equiment may be used. The equipment may consist of a series of filters, centrifuges, hydroclones or settlers to ensure that all, or nearly all, of the coal liquefaction product is recovered. The clarified liquid consisting essentially of precipitating solvent, coal liquefaction product and liquefaction solvent is recycled to the Agglomeration Zone **20** by the conduit **22**. The washed and separated solids, together with entrained precipitating solvent, are withdrawn through a conduit **46** to a Solvent Recovery Zone **50**. The residue from distillation, consisting essentially of unconverted or partially converted coal and mineral matter, now essentially free of coal liquefaction product, is discharged through a conduit **52**. The solvent is recycled through a conduit **54** to the Mixing Zone **40**. Make-up precipitating solvent may be added as required through a conduit **55**.

The net result of the practice of the above-described invention is a low solids product issuing from conduit **38** and a residue issuing through conduit **52** which contains little or none of the desired coal liquefaction product issuing from the Coal Liquefaction Zone **15**.

PREFERRED EMBODIMENT

FIG. 2 illustrates a preferred embodiment of this invention in conjunction with a particular coal liquefaction step. The latter was selected as an example of a suitable coal liquefaction step with which the present invention may be used. Coal is crushed to a size wherein most of the coal is less than 8 mesh U.S. Sieve Series, and is largely in the size range between 40 and 200 mesh. The crushed coal is usually dried first, although the drier is not shown.

The coal, which may or may not be preheated in the presence or absence of hydrogen, is introduced into a Mixing Zone **60** where it is mixed with liquefaction solvent. In this embodiment, the liquefaction solvent serves two primary purposes, one, as slurry agent for the coal, and the other, as solvent for the products.

The liquefaction solvent has a normal boiling point in excess of 400° F. and is a hydrogen donor. It is a hydrocarbon oil which consits essentially of partially hydrogenated polycyclic hydrocarbons, including naphthenic hydrocarbons, which are liquid under the temperature and pressure of hydroconversion. Mixtures of these hydrocarbons are employed, and are derived from intermediate or final steps of the process of this invention. Those hydrocarbons or mixtures thereof normally boiling between about 260° C. and 425° C. are particularly preferred.

The ratio of liquefaction solvent to coal is between 0.1 and 10. From the Mixing Zone, the coal slurry is pressurized by a pump **62** and pumped into a Coal Liquefaction Zone **64**. The combination of coal solids with the liquefaction solvent to form a slurry provides a practical method of handling the coal and charging it into the high pressure reactor. The viscosity of the slurry depends primarily upon temperature, solids concentration and composition of the liquefaction solvent. Sometimes, therefore, it is preferred to preheat the coal solids or the liquefaction solvent, or both, or to add a less viscous liquid to the slurry. Because of the difficulties involved in pumping high solids content slurries, the preferred slurry contains less than 60 percent coal solids by weight of slurry or mixture.

In the Coal Liquefaction Zone, the coal solids undergo liquefaction. This liquefaction is accomplished in the presence of molecular hydrogen and a suitable hydrogenation or hydrocracking catalyst. A hydrogen-containing gas is introduced into the Coal Liquefaction Zone by way of a hydrogen inlet line **66** which may consist of several inlets appropriately spaced. The Coal Liquefaction Zone is operated under conditions at which a major portion of the coal solids in the slurry rapidly undergoes conversion to liquid and gaseous products. The zone is maintained at a temperature of between 500° F. and 1000° F. with a total pressure ranging between 500 and 5,000 p.s.i.g. The preferred temperature range is between 725° F. and 875° F., depending upon the activity of the catalyst and the residence time of the reactants. The partial pressure of molecular hydrogen in the reactor is between 400 and 4,000 p.s.i.a. Hydrogen is injected into the zone at a rate sufficient to maintain this hydrogen partial pressure and the desired amount of hydrogen in solution in the liquids in the zone. Generally, the injection rate is in the range between 6,000 and 75,000 standard cubic feet (s.c.f.) per U.S. ton of reactants charged to the zone. The preferred hydrogen injection rate is between 25,000 and 75,000 s.c.f. per ton of reactants. The rate of coal charge per effective cubic foot of reactor volume is between 15 and 250 pounds per hour. A preferred type of reactor is the ebullated bed (described in U.S. Re. Pat. No. 25,770) and the preferred coal charge rate per cubic foot is in the range between 15 and 200 pounds per hour. The ratio of the coal feed rate to catalyst in the Coal Liquefaction Zone on a volume basis is in the range between 0.5 and 5.0. The catalyst size is in the size range between 200 mesh and 3 mesh.

In the Coal Liquefaction Zone, coal solids, liquefaction solvent, hydrogen and catalyst are in intimate association. At reactor conditions, when the solid coal liquefies, hydrogen aids fixing and stabilization of the molecules. This liquefaction of a major portion of the coal is aided by the presence of the liquefaction solvent. The hydrogen consumption to simply fix the molecules at the time of liquefaction depends on the convertible carbon content of the coal and has been estimated to be less than 1.5 pounds of hydrogen per 100 pounds of coal solids. Additional hydrogen is consumed in partially hydrogenating reactor liquids and in hydrocracking. In a hydroconversion process such as the present one, the hydrogen consumption will range between 0.5 pound and 15 pounds per hundred pounds of reactant. The hydrogen consumption rate depends on reactor hydrogen partial pressure, reactor temperature, reactant residence time, catalyst activity, and composition of the reactants.

Solids-laden reactor liquids are withdrawn overhead from the Coal Liquefaction Zone by the way of effluent line **68**. The vapor components of the reaction are removed by way of gas effluent line **70**. The reactor vapors contain molecular hydrogen and other light or low-boiling materials. The reactor vapors may be treated in any manner in a separation zone **72** to separate the non-condensable gases to gas line **74** from the condensable ones passing to light distillate line **76**. The particular vapor handling system will depend on the type of process and reaction system. A gas stream composed predominantly of hydrogen is usually recycled to the Coal Liquefaction Zone for further reaction with coal and reactor liquids.

The reactor liquid which is conducted through liquid effluent line **68** consists essentially of two components. The first component is a combination of low boiling and middle boiling hydrocarbon liquids and a residuum material, i.e. materials boiling above 975° F. The second component in this preferred embodiment consists of essentially all the undissolved solids, not just the finely divided suspended solids. The reason for including all solids is the fact that the presence of the coarse seems to aid in the separation of the suspended fines. The next four steps of the overall process represent the preferred embodiment of our invention whose objective is the effective separation and recovery of the first component substantially free of the second component.

Vaporous and gaseous products are removed from the Coal Liquefaction Zone through conduit **70**. The reactor liquid is introduced into a liquid-gas separator **78** which is operated at reactor conditions and is principally used to disengage gas bubbles and vapors trapped in the reactor liquid. Lower boiling fractions of the liquid which might interfere with subsequent operations are also removed either in the separator **78** or other suitable equipment. Conduit **80** serves to remove such products.

The topped effluent mixture of liquid and solids is passed through a reducing valve in line **82** (not shown) to a mixer-agglomerator **84**. The function of the mixer-agglomerator is to convert the more finely divided components of the second component to agglomerates which may be readily separated from the liquid. The desired agglomeration is accomplished by mixing a precipitating solvent with the effluent mixture from the flash still in the proper proportions and under the proper conditions of temperature, pressure, agitation, and holding time. Both the temperature and pressure in the mixer-agglomerator **84** are usually significantly lower than in the Liquefaction Zone **64**.

The preferred precipitating solvent is an aliphatic or naphthenic hydrocarbon or mixture of hydrocarbons of this type having a normal boiling point or boiling range within the temperature range of 150 to 225° C. For example, suitable precipitating solvents are cyclohexane, n-decane, Decalin, etc. Such precipitating solvents do not ordinarily occur in the effluent liquid leaving the Coal Liquefaction Zone **64**, at least in sufficient quantity to effect the agglomeration, but may be recovered from subsequent hydrocracking steps if such are incorporated in the process. The precipitating solvent is introduced into the Agglomerator **84** through a valved line **86** and conduit **82** in such amount that the ratio of precipitating solvent to the liquefaction solvent entering the Agglomerator **84** from line **82** lies between .1 and .5. The temperature within the Agglomerator is maintained between 250 and 370° C. by any suitable means. The pressure is maintained sufficiently high to assure that the precipitating solvent remains liquid. By thus regulating the conditions in the Agglomerator, a controlled amount of high molecular weight hydrocarbonaceous material, most if not all of which is benzene-insoluble, is precipitated. The mixture is stirred very vigorously so that the precipitated material is uniformly dispersed and distributed over the finely divided solids to act apparently as a binder in the formation of the agglomerates. Care must be taken to allow sufficient time for the desired agglomeration to occur. Generally, a minimum time of about five minutes is required. However, the minimum time required is a function of the other variables as well as the particular composition of the reactor liquid. If desired, more than the required amount of precipitating solvent for effective agglomeration may be added, for we have found that certain of the soluble metallo-organo compounds are removed from solution thereby.

The effluent mixture of liquid and agglomerated solids, together with precipitating solvent, is conducted by a line **88** without intentional cooling to a First Settler **90**. The preferred embodiment of the Settler **90** is shown in vertical cross-section in FIG. 3. Referring now to the latter figure, line **88** carries the effluent slurry into the top of the settler vessel near the center thereof. The settler is designed to confine two zones, a supernatant quiescent zone **92** and a lower mildly agitated slurry zone **94**. The effluent mixture falls from the line **88** through a centrally located baffle **100**. At the lower exit of the baffle, the solids continue to fall while the liquid reverses direction to overflow a circular weir **98** from which it is withdrawn through a valved conduit **102**. The solids and some entrained liquid fall into the lower slurry zone **94** which is mildly agitated by a motor driven paddle **104**, to facilitate drainage of the underflow through a valved draw-off line **106**. The underflow, comprising unconverted coal and mineral matter, together with some liquid, is withdrawn through valved conduit **106**.

The now-clarified liquid is conducted through the line **102** to a fractionator **108**. The liquid is separated into several fractions. The lowest boiling fraction is that used as the precipitating solvent and is removed through line **109**. A middle distillate in the 400 to 600° F. range is removed through a line **110**. Such products are normally refined or used for heater oil or jet fuel production. Heavy distillates in the 650 to 975° F. range are removed by line **111**. Such products may be refined in a hydrocracker or catalytic cracker, or may be used in heavy fuel blending. The 975+° F. residual fraction is removed by line **112** and may be used for fuel purposes or for coking or extreme hydroconversion. A portion of the heavy distillate is recycled by conduit **113** to the Mixing Zone **60** to be used as the liquefaction solvent.

The precipitating solvent is conducted by conduit **109** to a Mixing Zone **115** where it is mixed with the underflow from the First Settler. Here the two streams are thoroughly mixed and then transferred by line **116** to a Second Settler **118**. This settler is the same in construction and operation as the First Settler **90**. It serves to separate gravitationally the solids, including agglomerates of the second component, from the mixture of precipitating solvent and recovered liquid products of coal liquefaction. The latter mixture is recycled by conduit **86** to the Agglomerator **84**. The washed and separated solids are withdrawn through a conduit **120** to a heated solvent recovery zone **122** where any entrained liquid is distilled off to be recycled by a conduit **124** to the Mixing Zone. The solvent recovery zone may be a fluidized bed carbonization unit or a rotary kiln. The solid residue is discharged from the solvent recovery zone through a conduit **126**.

According to the provisions of the patent statutes, we have explained the principle, preferred construction and mode of operation of our invention and have illustrated and described what we now consider to represent its best embodiment. However, we desire to have it understood that, within the scope of the appended claims, the invention may be practiced otherwise than as specifically illustrated and described.

We claim:

1. In a coal liquefaction process which employs a liquefaction solvent wherein a product is recovered which contains a solution of coal liquefaction products in said liquefaction solvent as a first component, and finely divided

undissolved solids as a second component; and wherein a separation step is included for the separation of the second component from the first component, which uses a precipitating solvent to effect agglomeration of the second component, the improvement which comprises:

(a) adding to said product in an agglomeration zone said precipitating solvent in an amount sufficient to cause the precipitation of a deposit upon the second component, agitating the mixture and maintaining the temperature and pressure sufficiently high to keep the mixture fluid, to effect agglomeration of the second component;

(b) thereafter feeding said product to a primary separation zone wherein the major portion of the first component of a low solids content is separated from the second component;

(c) conducting said major portion of the first component to a distillation zone wherein the precipitating solvent is recovered as a distillate fraction thereof;

(d) conducting the second component admixed with some of the first component from the primary separation zone to a mixing zone;

(e) conducting said precipitating solvent from step (c) to said mixing zone to wash the agglomerates free of said first component and to prevent redispersion of the agglomerated second component;

(f) conducting the agglomerated second component from the mixing zone to a secondary separation zone wherein the agglomerated second component is separated from said precipitating solvent; and

(g) conducting said precipitating solvent, together with the recovered first component, to said agglomeration zone.

2. A process according to claim 1 wherein both of said separation zones are settlers wherein the second component is separated by settling.

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U.S. Cl. X.R.

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