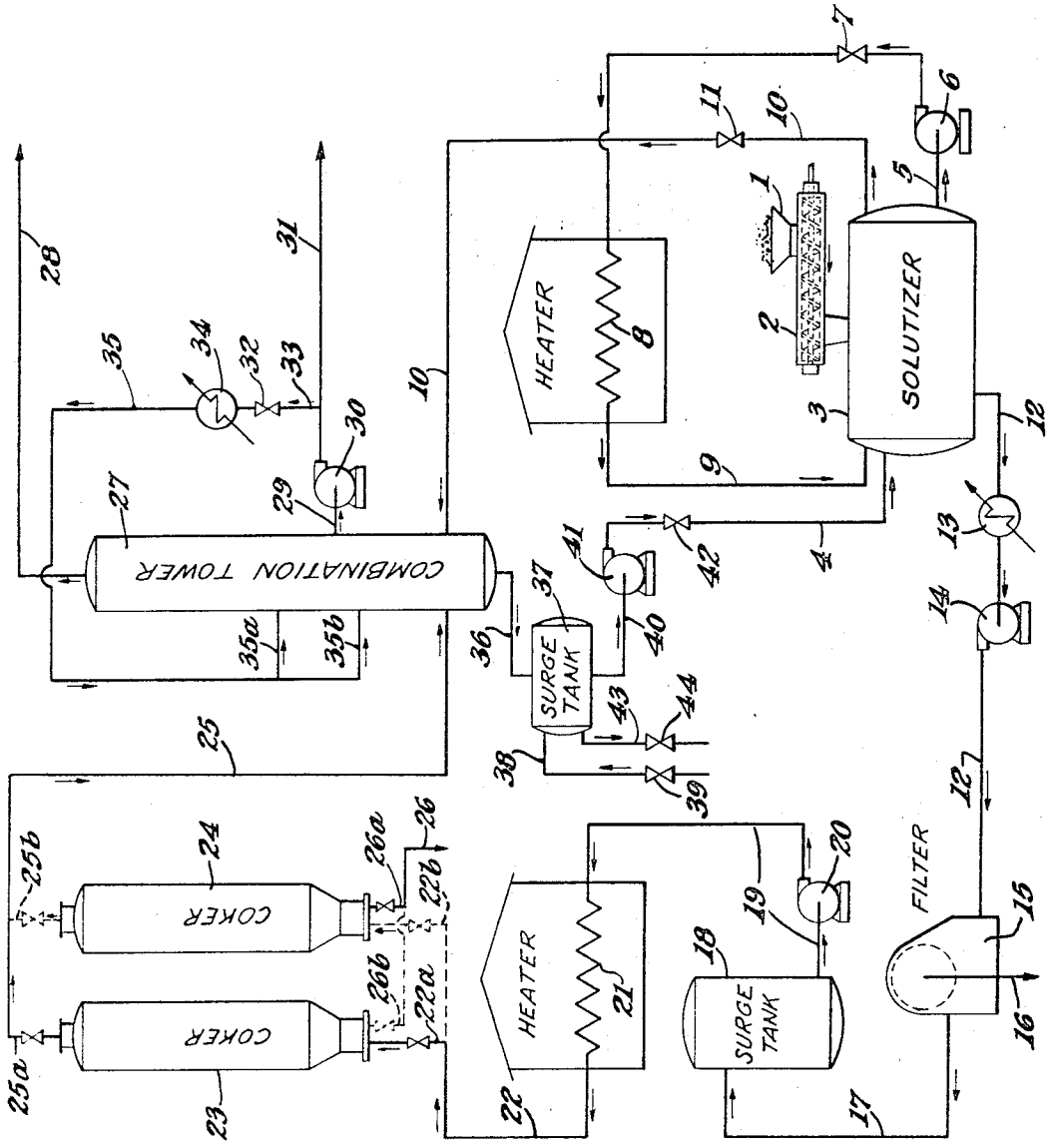
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COAL SOLVATION WITH NONHYDROGENATED SOLVENT IN THE ABSENCE OF ADDED HYDROGEN

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COAL SOLVATION WITH NONHYDROGENATED SOLVENT IN THE ABSENCE OF ADDED HYDROGEN

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Continuation-in-part of application Ser. No. 831,310, Aug. 3, 1959. This application Jan. 25, 1965, Ser. No. 431,758

6 Claims. (Cl. 208—8)

This application is a continuation-in-part replacement of U.S. application Ser. No. 831,310 filed Aug. 3, 1959, now abandoned.

This invention relates to a process for producing useful carbonaceous matter from coal and more particularly relates to the production of substantially ash-free coke from a coal selected from the group consisting of bituminous coal, subbituminous coal and lignite. This application relates to co-pending applications Ser. Nos. 831,311, filed Aug. 3, 1959, now abandoned, and 831,312 filed Aug. 3, 1959, now U.S. Patent 3,109,803.

Presently, most calcined anode carbon is produced from petroleum coke, with the remainder being produced from the coke obtained by carbonizing coal tar pitches. Considering the steadily increasing consumption of petroleum coke for manufacturing carbon anodes, coal represents a large potential source of coke suitable for such purpose.

Coal is primarily comprised of three-dimensional, condensed, cyclic structures of high molecular weight, such structures being predominantly six-membered rings. Bituminous coal has been considered to be an intimate mixture of bitumin and humin which are similar in that both are large, flat aromatic lamellar structures, but differ in molecular size, degree of aromaticity, oxygen content and extent of cross linking.

Mineral matter, fusain, volatile matter and moisture primarily constitute the remaining components present in coal. Mineral matter deposited in the sedimentary deposits by infiltration of ground waters during coalification and remaining after coal has been burned is called ash. Fusain, which is substantially consumed during the combustion of coal, may be considered a mineral charcoal.

The rank of coal (i.e. the degree of coalification) is determined by its carbon content which increases with the natural series of lignite, subbituminous coal and bituminous coal. In this series, the fixed carbon content generally increases whereas the moisture and oxygen content decreases.

The presence of fusain, mineral matter (analyzed as ash) and combined sulfur is the primary reason for the lack of utilization of coal for preparing coke suitable for making anodes to be used in the production of aluminum. Sulfur is usually present as organic sulfur, pyritic sulfur and/or inorganic sulfate. It is known that the ratio of inorganic sulfate and pyritic sulfur to organic sulfur increases with coals of increasing total sulfur content.

The following processes have been suggested for reducing fusain or mineral charcoal and mineral matter or ash present in various types of carbonaceous materials.

(1) Specific gravity separation or beneficiation—a method of separating the materials having a low specific gravity from the heavier materials which contain a substantial portion of the ash and fusain.

(2) Flotation and electrostatic separation—a process based on the differences between the surface properties of a coal particle and that of an ash particle.

(3) Chemical extraction of the ash—a process wherein the ash present in the raw coal is subjected to chemical attack, usually by an acid or alkali, to form soluble chemicals which are readily leached from the treated coal.

(4) Solvent extraction—a process wherein the coal substance is dissolved or extracted from the raw coal with a solvent to form a coal extract solution. The insoluble components, viz, fusain or mineral charcoal, mineral matter or ash and some of the other impurities remain substantially unchanged and are consequently suspended in the solution. These components are thereafter separated from the solution by filtration, decantation or the like.

One solvent extraction or solvation process for preparing a coal extract practiced in the past is referred to as the Pott-Broche process, named after Alfred Pott and Hans Broche. As described in their U.S. Patent No. 1,881,927, a coal extract was prepared by treating one part of a crushed carbonaceous material, such as pitcoal, lignite, peat and the like with two parts tetraline at a temperature of from 320 to 400° C. and at a pressure of about 100 atmospheres so as to form a solution of the coal substance. The minimum temperature of extraction was determined by the decomposition temperature of the initial coal. The insoluble and undissolved particles were separated from the solution by centrifugation and the tetraline was thereafter distilled from the solution to provide a coal extract which had a substantially smaller percentage of ash as compared to the weight percent of ash originally present in the raw carbonaceous material. The separation of the insoluble and undissolved particles from the solution was later found to be more effectively performed by passing the solution through a multiplicity of ceramic filter candles. Solvent losses occurring during the separation are one of the problems attending this technique of separation as well as other techniques for separating mineral charcoal or fusain, and mineral or ash and the like impurities from the solution. Another problem is the coking of the various process lines during extraction and separation.

In U.S. Patent No. 2,166,321, Pott described a process for utilizing the coal extract obtained by the aforementioned process to produce a semi-coke or full coke. The semi-coke or full coke was produced by blending the coal extract with a semi-coke (previously produced by the process) and charging the resultant mixture to a coke oven. The mixture was heated in the coke oven to a temperature of from 450 to 1100° C. to produce the semi-coke or full coke depending on whether a low or high temperature, respectively, was maintained in the coking oven. The coke product withdrawn from the coke oven had a low ash content and was suitable for use as a metallurgical coke for manufacturing electrodes.

A variation of the Pott-Broche method is found in Franke, U.S. Patent No. 2,686,152. A Pott-Broche type of solvent extraction is initially carried out, either with a hydrogenated polynuclear solvent or in the presence of added hydrogen. The solution is then distilled to dryness to drive off solvent, the coke is recovered and distillate is fractionated. The patentee notes that, if the extract solution is filtered prior to coking, only a powdery char with no coke structure results.

It is a principal object of our invention to provide a novel method for producing substantially ash-free coke from a coal selected from the group consisting of bituminous coal, subbituminous coal and lignite.

A further object of our invention is to provide a novel method for preparing not only substantially ash-free coke but also valuable products, such as gas, aromatic solvents and oils, and ammonia from a coal selected from the group consisting of bituminous coal, subbituminous coal and lignite.

A still further object of our invention is to provide a novel closed-cycle process for preparing substantially ash-free coke from a de-ashed solution of carbonaceous mat-

ter by coking such solution whereby the vaporous products recovered from the coking unit provide at least a portion of the solvent required to prepare the solution of carbonaceous matter.

Further objects and a fuller understanding of our invention may be had by referring to the following description taken in conjunction with the accompanying drawing, in which the figure is a schematic flow-diagram illustrating a preferred embodiment of the invention for producing substantially ash-free coke.

Prior to treatment in our novel process, the raw coal to be treated is crushed and ground in conventional crushing and grinding equipment. The particle size distribution may range up to 10 mm., however, we prefer a particle size distribution whereby a major portion of the coal particles pass through a #100 mesh screen.

In accordance with our invention we propose to digest and/or dissolve the extractable carbonaceous matter (which excludes fusain) present in the pulverized raw coal under conditions of elevated temperatures and pressures utilizing a high boiling liquid solvent of high aromaticity thereby forming a solution of such extractable carbonaceous matter (hereinafter referred to as a coal solution). Fusain and the mineral matter or ash are substantially unaffected by the solvent and are suspended in the coal solution.

The coal solution while in a free flowing state is filtered to separate the suspended solids (including undissolved extractable carbonaceous matter) and is thereafter heated to a temperature above incipient coking and passed to a coking unit. Product coke is withdrawn from the coking unit and passed to subsequent processing units including coke handling and calcining facilities. The vaporous products are withdrawn from the coking unit and are introduced into a fractionating unit wherein they are separated into various fractions, such as, for example: a light gas and gasoline fraction; a medium and heavy middle oil distillate fraction; and a heavy bottoms distillate fraction. A portion or all of the high boiling liquid solvent of high aromaticity utilized for dissolving or digesting the extractable carbonaceous matter present in the raw coal is derived from a distillate fraction recovered from the fractionating unit.

In preparing the coal solution, we prefer to use a ratio of high boiling liquid solvent to raw coal of from about 2:1 to about 3:1, however, a ratio range of from ½:1 to 6:1 may be used. A lower ratio within the range of from ½:1 to about 1½:1 does not as efficiently dissolve or digest the extractable carbonaceous matter whereas a higher ratio within the range of from about 3:1 to 6:1 places a heavier load on the filtration unit and makes filtration uneconomical. Further, high solvent ratios place an additional heat load on subsequent heating apparatus as a result of additional vaporization requirements.

Solvation or digestion of the extractable carbonaceous matter is accomplished in a solutizer or solvation vessel at through-put times varying from about 5 to 120 minutes. A temperature of from about 750 to 825° F. and above the final decomposition temperature (where there is substantial heat decomposition of the extractable carbonaceous matter) of the initial coal is preferably maintained in the solutizer. It is believed that within this temperature range, the cyclic, three dimensional components of the coal are thermally depolymerized with the resulting constituents being soluble in the solvent. The coal solution which is formed has physical properties similar to those of low temperature carbonization coal tar pitches. Operating within this temperature range, we have dissolved more than 95% of the extractable carbonanceous matter based on the available ash-free, fusain-free and sulfur-free coal. Digestion or solvation may be performed at temperatures of from 600 to 850° F., however, operating at the lower temperatures within this range has the disadvantage of dissolving a substantially smaller portion of the extractable carbonaceous matter whereas polymerization of the high boiling solvent and the coal solution has been observed at temperatures above about 825° F. For effective and efficient solvation, polymerization of both the coal solution and the highly aromatic solvent must be minimized. A pressure range of about 1 to 7 or 8 atmospheres is preferably maintained on the solutizer, however, the solutizer may be operated at pressures up to 100 atmospheres. The preferred pressure range is substantially lower than pressure ranges previously required, since the solvent has a substantially higher boiling range as compared to the solvents, such as coal tar, water gas tar, tetraline, phenanthrene and anthracene oil which have been heretofore suggested for the solvation of the extractable carbonaceous matter.

It is to be noted that in accordance with the present invention, solvent extraction is carried out with non-hydrogenated solvents and in the absence of added hydrogen, a significant departure from the prior art teachings in view of the results obtained.

Filtration (including conventional dewatering, washing and drying of the filter cake) of the coal solution is performed at a temperature of from about 400 to 700° F. utilizing a metallic filter medium precoated with a conventional filter aid. Precoating the filter substantially improves the separation of undissolved and insoluble particles from the coal solution. With a precoated filter, we have prepared filtered coal solutions which when analyzed for ash only varied between 0.08 and 0.02 weight percent, irrespective of the ash content of the raw coal which varied from 1 to 20 percent. This indicated that the efficiency of de-ashing by filtration was a function of the size of the ash particle rather than the ash content. Using conventional diatomaceous earth filter aids, the combustible carbonaceous matter of the filter cake is sufficient to permit its further use as fuel for process steam, and the like. Carbonaceous filter aids permit still further heat recovery from the filter cake.

The sulfur content of low sulfur coals (i.e. about 1.0 to 2.0 weight percent sulfur) has been reduced to values of from 0.4 to 0.6%, which represented an average reduction of about 70 percent. Since the organic sulfur present in the raw coal is readily soluble in the highly aromatic solvent, the quantity of sulfur separated from the raw coal is a function of the pyritic and sulfate sulfur content of the coal with the final sulfur content of the filtered coal solution primarily being a function of the organic sulfur content of the coal. With high sulfur coals, the ratio of inorganic sulfate and pyritic sulfur to organic sulfur is normally greater than for low sulfur coals. Consequently, with high sulfur coals, we have observed a greater percentage-wise reduction of the sulfur content, reducing the sulfur content of the filtered coal solution to as low as 0.6 weight percent, which correspond to an over-all reduction of the sulfur content of from 70 to 90%.

The filtered coal solution is readily coked in any conventional coking unit used for coking petroleum feedstocks by heating the solution to a temperature above its incipient coking temperature and thereafter coking the solution to produce substantially ash-free coke having a wide range of volatile matter, i.e., of about 4 to 26%.

We prefer to coke the coal solution in a delayed coking unit basically comprised of coke drums. Utilizing such a unit the filtered coal solution is heated to a temperature of from about 850 to 1050° F. and is introduced into one of the coke drums. The lower limit above the incipient coking temperature to which the coal solution is heated is dictated by the upper limit of the volatile matter desired in the coke product, normally 8 to 16%. The upper limit above the incipient coking temperature to which the coal solution is heated is dictated by the lower limit of the volatile matter in the coke product, normally about 6 to 8%, since coke having less than 6% volatile matter is extremely hard and is consequently difficult to remove from the coke drum.

The coal solution may also be coked in a contact cok-

ing unit (comprised of a reactor having a downwardly flowing bed of a particulate material on which the feed is spread and coked) and at higher temperatures so as to obtain product coke having less than 6% volatile matter. Higher temperatures are permissible using a contact coking unit since there is not the practical limitation of decoking a drum filled with coke of less than 6% volatile matter. Further, contact coking procedures do not require preheating of the coker feed to a temperature above its incipient coking temperature, since the feed may be heated in the coker to a temperature above incipient coking by the sensible heat of the contact particles.

The fractionating unit is controlled under conditions of temperature and pressure to provide all or a portion of the highly aromatic liquid solvent for the solvation of the coal. The initial boiling temperature (converted to one atmosphere) of such solvent is from 650 to 850° F. and has physical properties similar to that of a low temperature tar distillate. Of course, the boiling point will rise as products are distilled off during solvation, if this is allowed. It has been observed when processing bituminous coal that sufficient solvent (for a 2:1 to 3:1 ratio of solvent to coal) is usually obtained from the fractionating unit by operating the unit to provide a distillate having an initial boiling temperature (converted to one atmosphere) of about 750° F. The fractionating unit is preferably operated at pressures of from 1 to 7–8 atmospheres; however, it is generally contemplated that pressures up to 100 atmospheres may be maintained on the unit. The solutizer may be considered to be an extension of the fractionating unit, but it is not necessary to operate both the fractionating unit and solutizer at the same pressure. Generally the overall level of pressure in the solutizer will be at some higher pressure than the fractionating unit because of the normal increase of solutizer tempertaure over tower bottom temperature and because of the presence of some lower boiling coal decomposition products formed in the solutizer. Solvent requirements may necessitate a higher pressure on both units to provide more solvent from the fractionating unit by the inclusion of light ends normally lost to side streams and/or overhead when operating the fractionating unit at lower pressures.

Referring to the drawing, ground or pulverized coal selected from the group consisting of bituminous coal, subbituminous coal and lignite, and/or mixtures thereof are collected in a hopper **1** from which it is continuously distributed at a desired rate by a conveying mechanism **2** into a solutizer tank **3** maintained at a pressure of from 1 to 7–8 atmospheres. As illustrated, conveying mechanism **2** is a screw type feeder which introduces the coal into solutizer **3** without loss of pressure therein. Any conventional means of mechanical transfer may suffice, providing the means allows for positive transfer of the coal into solutizer **3** without a substantial loss of pressure therein.

The high boiling liquid solvent of high aromaticity is introduced into solutizer **3** through line **4** at a rate so as to provide a ratio of solvent to coal of from ½:1 to 6:1. Normally, we prefer a solvent to coal ratio of from about 2:1 to about 3:1, since effective extraction rates are obtained within this ratio range while minimizing filtration costs. Solutizer **3** is maintained at a temperature of from about 600 to 850° F., preferably of from about 750 to 825° F. and above the final decomposition temperature of the initial coal whereby a substantial portion of the extractable carbonaceous matter in the raw coal is thermally depolymerized. The products of depolymerization are soluble in the highly aromatic solvent and thereby form, with the solvent, the coal solution. Undissolved and insoluble solids including undissolved extractable carbonaceous matter, and insoluble mineral matter or ash and mineral charcoal or fusain are suspended in the coal solution. An agitator (not shown) may be provided to agitate the coal-solvent mixture during solvation.

Solvation temperatures are maintained in solutizer **3** by withdrawing and circulating a portion of the coal solution and coal-solvent mixture through an external heating system. The withdrawn portion is passed through line **5** by pump **6** under the control of valve **7** to heater **8** and thereafter reintroduced through line **9** into solutizer **3**. In this manner, solvation temperatures are maintained within solutizer **3** without the necessity of a high temperature and pressure heating system, which would be the case, if the solvent was preheated to a temperature sufficient to maintain solvation temperatures within the solutizer. A through-put time of the raw coal of from about 5 to 120 minutes is normally sufficient to dissolve or digest effectively and efficiently the extractable carbonaceous matter. Since the solvent may have an initial boiling temperature (converted to one atmosphere) as low as 650° F., whereas solvation temperatures may be as high as 850° F., solutizer **3** is provided with vent line **10** under the control of valve **11** to permit the withdrawal of the lower boiling components of the solvent and any volatile matter vaporized from the raw coal. In this respect, it has been observed that the quantity of volatile matter is practically negligible. A substantially uniform coal solution, wherein undissolved and insoluble solids are suspended, with include mineral charcoal or fusain and mineral matter or ash, is withdrawn through the bottom draw-off **12** and is drawn through cooler **13** by pump **14** and passed to a continuous rotary filter **15**.

The coal solution is cooled to a temperature of from about 400 to 700° F. during its pasasge through cooler **13**. Preferably, the rotary filter **15** is precoated with conventional filter aids and is normally operated at a pressure of about 40 to 60 p.s.i.g. to effect efficient removal of substantially all of the suspended solids including undissolved carbonaceous matter. The filter cake is washed and dried to recover absorbed solvent and is withdrawn from filter **15** through line **16**. The substantially de-ashed coal solution is passed through line **17** to surge drum **18** from which it is passed through line **19** and pump **20** to heater **21** (a suitable coil heater). The coal solution is heated to a temperature of from about 850 to 1050° F. in heater **21** and is passed therefrom through line **22** to a coking unit.

The coking unit, as illustrated, is a delayed coker and is comprised of coke drums **23** and **24**. The heated coal solution in line **22** is introduced through line **22*a*** into coker **23** wherein the charge is decomposed into coke and a vaporous effluent. The coker overhead in line **25*a*** is passed through line **25** to a fractionating unit. While coker **23** is being filled with coke, coker **24** is being decoked with product coke withdrawn through lines **26*a*** and **26** for subsequent processing. In normal operation, cooling and decoking of coker **24** is completed prior to the filling of the coker **23**. With decoking completed on coker **24** and having filled coker **23** to a predetermined level, the coker charge is diverted to coker **24** through line **22*b***, with the vaporous effluent in line **25*b*** being passed to the fractionating unit through line **25**. After cooling, coker **23** is decoked, with product coke being passed through lines **26*b*** and **26** for subsequent processing.

The coker overhead in line **25** is introduced into a fractionating or combination tower **27** which is provided with suitable fractionating decks (not shown). Introduction of the effluent into the tower may result in some foaming. This may be effectively inhibited by the addition of a small amount of an antifoam agent, at the point of introduction or at some elevated point in the tower. The hereinbefore mentioned distillate and volatile matter in line **10**, which are evolved during solvation, are introduced into the lower portion of the tower **27**.

The combination tower overhead products in line **28** comprising condensible and non-condensible components are passed to conventional processing units to separate the condensible components from the non-condensible

components. A medium and heavy middle oil is withdrawn from an intermediate point on the tower **27** through line **29** by pump **30** and is passed through line **31** to refining units (not shown). A portion of the middle oil in line **29**, under the control of valve **32**, is passed through line **33**, waste heat boiler **34**, and line **35** and is thereafter split into at two portions (lines **35***a* and **35***b*) for introduction as reflux into tower **27**.

Tower bottoms in line **36** are passed to surge tank **37**. By properly controlling the temperature level within tower **27**, the distillate fraction in line **36** has an initial boiling temperature (converted to one atmosphere) of from 650 to 850° F. and represents all or a portion of the solvent to be used for dissolving or digesting the extractable carbonaceous matter in the pulverized raw coal feed. As hereinbefore mentioned, when processing bituminous coal, solvent requirements may usually be satisfied by operating the fractionating unit so as to obtain a distillate having an initial boiling temperature (converted to one atmosphere) of about 750° F. If additional solvent is required (over the 750° F.+distillate) the fractionating unit may be operated to provide a distillate having an initial boiling temperature (converted to one atmosphere) as low as 650° F. or if desired, the fractionating unit may be operated to provide the latter distillate, withdrawing excess solvent over solutizer requirements for utilization in other processes.

Start-up or make-up solvent is introduced into surge tank **37** through line **38** under the control of valve **39**. The quantity of solvent necessary to provide a solvent to coal ratio of from ½:1 to 6:1 is withdrawn from tank **37** through line **40** and passed by pump **41** under the control of valve **42** into line **4**. Aftr start-up, should the quantity of captive solvent exceed solvation requirements, excess solvent may be withdrawn from tank **37** through line **43** under the control of valve **44**. It is generally contemplated that solvent requirements for the solvation of bituminous coal may be fulfilled by controlling the operating pressure and temperature of the combination tower so as to permit the direct introduction of the distillate fraction in line **36** to solutizer **3**, in which case, surge tank **37** is superfluous.

The following examples will further illustrate the nature of this invention, it being understood that the invention is not limited to the operating conditions or quantities therein:

EXAMPLE I

In accordance with our invention a bituminous coal having the following proximate analysis:

Analysis

	Percent by wt.
Moisture	1.4
Volatile matter	36.6
Fixed carbon	56.6
Ash	5.4

was crushed and ground to a particle size distribution whereby 37.3% was retained on a #100 mesh screen. For comparison purposes, combined sulfur was analyzed as being 1.46 weight percent. The crushed coal and a tar distillate having an initial boiling temperature of about 750° F. (converted to one atmosphere) were introduced into the solutizer zone to provide a 3 to 1 ratio of solvent coal. The mixture was agitated while maintaining a temperature of 800° F. and a pressure of 5 atmospheres. The resulting coal solution was cooled to a temperature of 450° F. and passed through a filter precoated with a standard filter aid. The filtered coal solution represented an 86.3% recovery of extractable carbonaceous matter based on the crushed coal and had the following properties:

Properties

Sp. gravity (100°/100° F.)	1.2407
Softening point, ° F. (B. & R.)	152
Sulfur (wt. percent)	0.46
Carbon residue (wt. percent):	
Ramsbottom	30.9
Conradson	31.3
CS_2 solubility (wt. percent):	
Bitumen	76.66
Ash	0.02
Difference	23.22

The filtered coal solution was heated to 910° F. and coked to provide a coke which was recovered from the coker having the following properties:

Properties

	Wt. percent
Volatile matter	10.9
Sulfur	0.27
Ash	0.13
Iron	0.036
Silicon	0.009
R_2O_3	0.112
Nickel	0.0073
Titanium	0.013
Vanadium	0.00024
Boron, p.p.m.	------

EXAMPLE II

Following the procedures of Example I, a bituminous coal having identical properties as the coil in Example I and a distillate having an initial boiling temperature (converted to one atmosphere) of about 750° F. were introduced into the solutizer zone to provide a 2 to 1 ratio of solvent to coal. The mixture was agitated while maintaining a pressure of 5 atmospheres and a temperature of 800° F. The resulting coal solution was cooled to a temperature of about 450° F. and passed through a filter precoated with a standard filter aid. The filtered coal solution represented a 67.5% recovery of extractable carbonaceous matter based on whole coal and had the following properties:

Properties

Specific gravity 100°/100° F.	1.2523
Softening point, ° F. (B. & R.)	212
Sulfur (wt. percent)	0.44
Carbon residue (wt. percent): Conradson	35.9
CS_2 solubility (wt. percent):	
Bitumen	68.35
Ash	0.07
Difference	31.58

The filtered coal solution was heated to 910° F. and coked to provide a coke which was removed from the coker having the following properties:

Properties

	Wt. percent
Volatile matter	6.5
Sulfur	0.28
Ash	0.12
Iron	0.025
Silicon	0.003
R_2O_3	0.096
Nickel	0.0015
Titanium	0.014
Vanadium	0.00024
Boron, p.p.m.	10

The quality of the coke prepared according to the above examples match that of coke prepared from the best petroleum residue.

The overhead from the coker was condensed and had the following properties:

Properties

Specific gravity at—	
100°/60° F.	1.148
25°/25° C.	1.160

Properties—Continued

Viscosity at 210° F., centipoises	12.0
Conradson carbon (wt. percent)	0.18
Sulfonation residue	None
Sulfur (wt. percent)	0.38

Of the coker effluent, about 50% had an initial boiling temperature above 750° F.

EXAMPLE III

The following example illustrates that a lower grade coal having a high ash and sulfur content may be effectively de-ashed to provide a coal solution having an ash content substantially equal to those derived from the high grade coals illustrated in Examples I and II. A bituminous coal having the following proximate analysis:

Analysis

	Percent by weight
Moisture	2.69
Volatile matter	37.7
Fixed carbon	38.9
Ash	19.66

was crushed and ground to a particle size distribution whereby substantially all of the crushed coal passed through a #35 mesh screen. For comparison purposes, the combined sulfur was analyzed as being 3.72 weight percent. The crushed coal and a tar distillate having an initial boiling temperature (converted to one atmosphere) of 750° F. were introduced into the solutizer zone to provide a 3 to 1 ratio of solvent to coal. The mixture was agitated while maintaining a temperature of 800° F. and a pressure of 5 atmospheres. The resulting coal solution was cooled to a temperature of 550° F. and passed through a filter precoated with a standard filter aid. The filtered coal solution represented a 67.3% recovery of extractable carbonaceous matter based on the crushed coal and had the following properties:

Properties

Sp. gravity (100°/100° F.)	1.2516
Softening point	147
Sulfur (wt. percent)	0.63
Carbon residue (wt. percent): Ramsbottom	32.7
CS_2 solubility (wt. percent):	
Bitumen	76.81
Ash	0.06
Difference	23.13

Approximately 89 weight percent of the original sulfur content of the raw coal was separated from the coal solution and was carried in the filter residue as ash in the form of inorganic pyritic and sulfate sulfur. The percent increase in sulfur reduction would appear to be a result of the increased ratio of inorganic pyritic and sulfate sulfur to organic sulfur in low grade coals.

EXAMPLE IV

The following example illustrates that lignite may be effectively and efficiently de-ashed according to our process regardless of the substantially smaller percentage of coal substance available in the raw lignite.

A dried lignite having the following proximate analysis:

Analysis

Moisture	1.9
Volatile matter	45.2
Fixed carbon	38.0
Ash	15.1

was crushed and ground to a particle size distribution whereby 20.9% was retained on a #35 mesh screen. The crushed lignite and a distillate having an initial boiling temperature (converted to one atmosphere) of about 750° F. were introduced into a solutizer zone to provide a 3 to 1 ratio of solvent to lignite. The mixture was agitated while maintaining a temperature of 800° F. and a pressure of 5 atmospheres on the solutizer zone. The resulting coal solution was cooled to a temperature of 600° F. and passed through a filter precoated with a standard filter aid. The filtered coal solution represented a 21.0% recovery of extractable carbonaceous matter based on the crushed lignite and had the following properties:

Properties

Specific gravity (100°/100° F.)	1.2355
Softening point	121.5
Sulfur (wt. percent)	0.57
Carbon residue (wt. percent): Ramsbottom	28.0
CS_2 solubility (wt. percent):	
Bitumen	85.13
Ash	0.02
Difference	14.85

Note that in all of the above examples, the sulfur and ash content of the filtered coal solutions were substantially the same. This illustrates that the effectiveness of de-ashing and desulfurizing a coal solution is not restricted to any particular grade or rank of coal selected from the group consisting of bituminous coal, subbituminous coal and lignite.

While we have shown and described a preferred form of our invention, we are aware that variations may be made thereto and we, therefore, desire a broad interpretation of our invention within the scope of the disclosure herein and the following claims.

What is claimed is:

1. A process for producing a substantially ash-free solution of carbonaceous matter from a coal selected from the group consisting of bituminous coal, subbituminous coal and lignite which are comprised of extractable carbonaceous matter, fusain and mineral matter, which comprises:

mixing the coal in crushed form with a non-hydrogenated, high boiling liquid solvent of high aromaticity having an initial boiling temperature, converted to one atmosphere, of from about 650° F. to about 850° F.;

heating said mixture in the absence of added hydrogen to a temperature of from about 600° F. to about 850° F. to form a solution of said extractable carbonaceous matter wherein solids including fusain and mineral matter are suspended; and

separating said suspended solids from said solutions.

2. A process as defined by claim 1, wherein said mixture is heated to a temperature of from 750° F. to 825° F. and above the final decomposition temperature of the initial coal.

3. A process as defined in claim 1, wherein heating of said mixture is performed under a pressure of from 1 to about 8 atmospheres.

4. A process as defined in claim 1, wherein said coal is mixed with from one-half to six parts of said solvent.

5. A process as defined in claim 1, wherein said solution is cooled to a temperature of from about 400° F. to about 700° F. prior to separating said suspended solids from said solution.

6. A process as defined in claim 1, wherein ratio of solvent to coal is from about 2:1 to about 3:1.

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