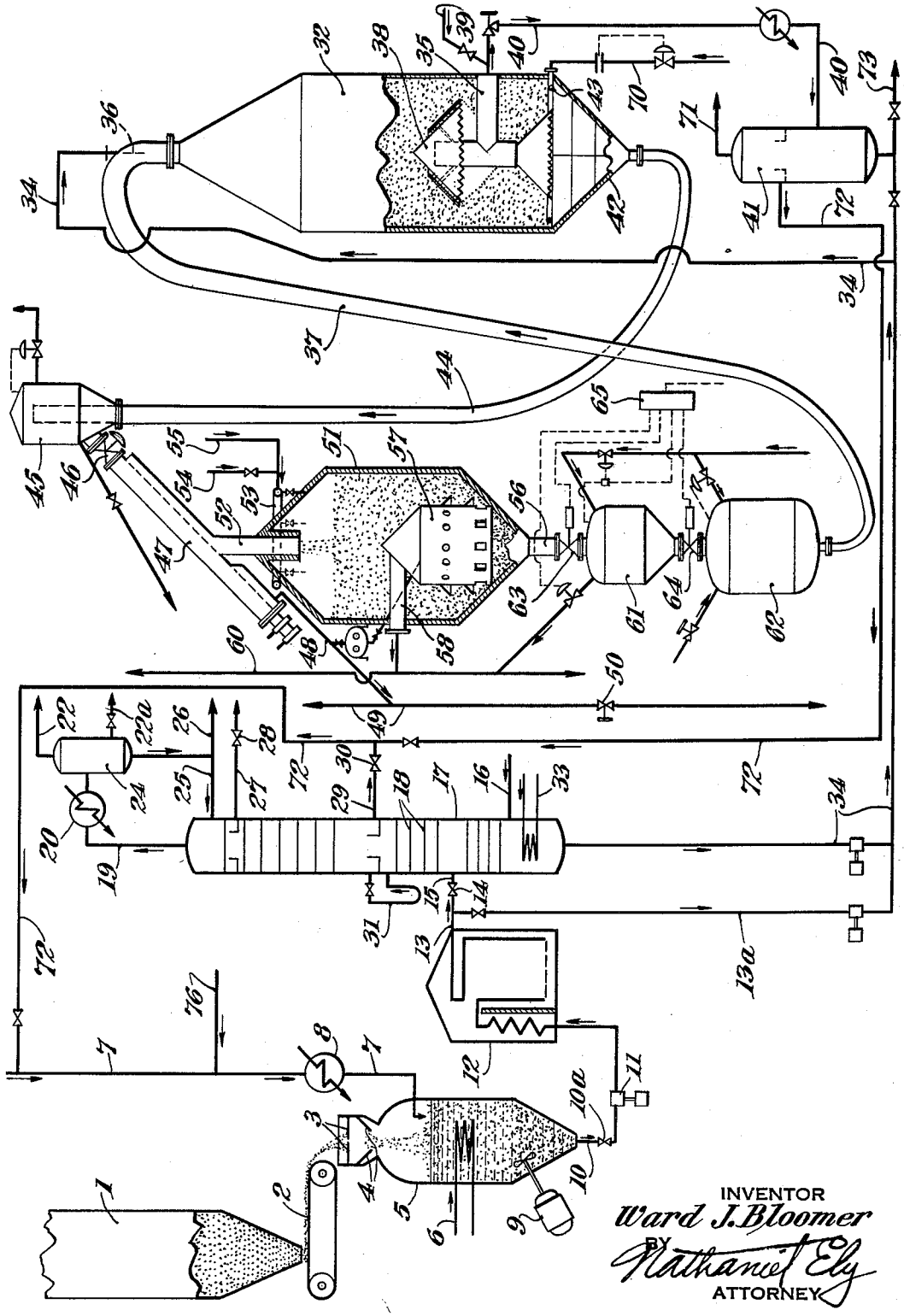
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GILSONITE CONVERSION

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INVENTOR
Ward J. Bloomer
BY
Nathaniel Ely
ATTORNEY

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GILSONITE CONVERSION

Ward J. Bloomer, Westfield, N. J., assignor to Lummus Company, New York, N. Y., a corporation of Delaware

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This invention relates to the recovery of available end-products from the class of material known as asphaltites of which gilsonite is a particular example.

It is known that the asphaltites and gilsonite in particular, is a solid bitumen which has a melting point in the order of 270° F. to about 400° F., normally contains less than 5% volatile matter and has a fixed carbon content in the range of 10–30%. The most particular characteristic of gilsonite, however, is the fact that when heated to a temperature above 500° F., there is formed a considerable quantity of gas and the release of substantial quantities of heat which tend to convert the liquid gilsonite directly into a solid coke material which has a relatively low value.

To date, the difficulties in heating gilsonite to a necessary extent have made it impossible to recover the valuable hydrocarbons on a continuous commercial basis. I have found that if the gilsonite is heated in the presence of a liquid hydrocarbon of selected boiling range in such ratios that the exothermic heat can be largely dissipated by the vaporization of the hydrocarbon, it is possible to heat the gilsonite to a sufficiently high degree so that it can be ultimately reduced to a dry coke by charging it to apparatus suited for that purpose without unwarranted formation of coke in the heater coil or vapor lines to such apparatus. During the heating, it is possible to recover valuable gaseous end-products of the lower boiling range such as nitrogenous and other base materials.

It will be apparent that it is necessary to have a liquid which will have a boiling range substantialy above the temperature of incipient coking of the gilsonite, which is around 500–550° F. In addition, there must be enough of the liquid having sufficiently high latent heat present so that enough of the exothermic heat is absorbed to prevent excessive gassing and temperature rise of the mixture. For this purpose, I prefer to use a gas oil of about 700–900° F. end point. Such a liquid has the advantage that it will tend to partially dissolve the gilsonite and being an adjacent fraction may be readily handled not only in a cracking heater, but in fractional distillation equipment and in a continuous coking operation. There are other liquids however, which could be used solely for the absorption of the exothermic heat which would permit the recovery of the low boiling end products following the preliminary cracking stage.

My invention further contemplates the reduction of the liquid residue from the first fractional distillation step to a dry coke which may be effectively used as a circulating medium in a continuous coking operation of the type described in the co-pending application of August Henry Schutte, Serial No. 252,306, filed October 20, 1951.

The attached drawing shows a schematic view of the complete equipment for the reduction of powdered gilsonite to its ultimate end products.

In accordance with a preferred form of embodiment of my invention, ground or pulverized gilsonite is collected in a hopper 1 from which it may be distributed at a desired rate by the conveying mechanism 2 into a dissolving

tank 5. Tank 5 is provided with lock gates 3 and 4 to maintain the tank in a closed manner.

The dissolving tank 5 is provided with a heating coil 6 which may be interconnected to a steam or other heat source to maintain the tank at about 500° F., the mixture being in a generally liquid mass resulting from the addition of suitable diluent from the line 7. A heater 8 is provided in line 7 to bring the diluent to the desired solution temperature. As heretofore described, this liquid is preferably a high boiling hydrocarbon of the general boiling range of a gas oil. Approximately ⅓ to ½ as much liquid is added to the tank as powdered gilsonite.

The gilsonite liquid mixture is maintained as a slurry or solution which is preferably agitated by the agitator 9 and is drawn off with substantial uniformity through the bottom drawoff 10 through the valve 10*a* and is pumped by the pump 11 to the heater 12.

Heater 12 is a suitable coil heater which may be provided with a convection as well as a main heat section. The oil is introduced at about 500° F. and is then heated to a temperature substantially above the temperature of incipient coking and preferably to about 770–775° F. during which partial cracking takes place.

During the heating period, substantial quantities of heat are released by the exothermic decomposition of the gilsonite. I have found that if the diluent has a sufficiently high latent heat and is present in sufficient quantities, enough of the exothermic heat is absorbed to prevent the gilsonite changing into coke. In fact, the appearance of the effluent from the heater 12 at 13 appears to be of a heavy gas oil nature. This material is introduced at the outlet temperature through valve 14 into the fractionating column 17. Introduction of the effluent into the column may result in some foaming. This can be effectively inhibited by the addition of a small amount of an anti-foam agent, at the point of introduction or at some point higher up in the column.

The column 17 is provided with suitable fractionating decks 18 and is preferably provided with a reboiler 33. Column 17 may also be provided with steam stripping at 16 for the purpose of driving the light ends overhead through the line 19.

The overhead vapors comprising condensible and non-condensible portions are withdrawn through line 19. These may be treated in a conventional manner to separate condensible hydrocarbon vapors from the products of gasification which include valuable amine compounds. The production and recovery of the amine compounds from the raw gilsonite is one of the important features of my invention.

The amines and other non-condensible gasification products separated from the condensed hydrocarbon vapors are withdrawn from separator 24 and passed by way of lines 22 and 22*a* to further treatment and separation in apparatus not shown. Condensed hydrocarbons are in part returned to the tower by line 25, the remainder being sent to the recovery system through line 26.

As described hereafter, the valuable amines may also be recovered in the coker distillate tower 41 when heater effluent is charged directly to the coking reactor.

In a lower part of the tower a heavy distillate is withdrawn through line 29 under the control of valve 30. The heavy distillate withdrawn is returned by lines 72 and 7 to dissolving tank 5. Some heavy distillate may appear in the asphalt bottoms in the lower part of the tower. While the presence of excess amounts of heavy distillate is not desirable in the asphalt bottoms since it will abstract heat from the coke bed in the coking reactor, the presence of some heavy distillate is desired to maintain the coker charge in partial dilution during transfer.

It is generally desirable to reduce the gilsonite to its

ultimate end products of gases, coker distillate and solid coke. This may be accomplished by pumping the asphalt bottoms in line **34** to the continuous coker indicated at **32** through which passes a continuous stream of relatively small, hard coke pebbles as disclosed in August H. Schutte's U. S. Patent 2,600,078.

The continuous coker **32** is maintained full of such carbon particles and the liquid asphalt solution in line **34** is injected onto the coke pebbles at **36**. This results in a continuous vaporization of the lighter ends of the charge by the preheated coke. The vapors are removed through the vapor outlet **35** and the coke particles continue to move downward through the outlet zone **42**. A steam purging ring is provided at **43** below the vapor take-off **35** and in such position as to insure dryness of the coke particles as they pass downward and out through the outlet conduit **44**.

Pressure is maintained in the reactor **32** sufficient to discharge the coke particles through the line **44** to the vapor release chamber **45** from which the particles flow through control valve **46** and through the separator **47** with the desired size of particles passing into the reheater **51** through the inlet **52**. Oversize particles may be removed from the bottom of the separator **47**. Undersize particles may also be removed from the separator **47** and returned to the system through the line **48**.

Reheater **51** is of a radiant type being provided with a series of burners **53** in the upper part of the reheater, such burners being supplied by gas and air from the lines **54—55** in a well known manner. Particles of coke are heated to the desired extent and continuously moved downward past the flue gas collector **57** with the flue gases removed at line **58** and passed to the stack at **60**.

Outlet from the reheater is through a series of lock and forwarding tanks **61** and **62** which are provided with suitable valves **63** and **64** that are in turn operated in cycle manner by the controller **65**. The tanks are provided with steam inlets which are also controlled by the controller to permit the movement of the coke particles to the reactor **32** by line **37**.

The vaporous material removed from the reactor **32** which is recovered through line **40** is quenched by oil entering line **39**, partially condensed and passed into the fractionator **41** from which a heavy gas oil is removed at **72** and is used to supplement, if necessary, the hydrocarbon used as the gilsonite diluent. A lighter overhead material may be removed at **71** and a heavier product may be removed at **73**. The material in line **73** may be recycled to the coker **32** if desired.

Example I

In accordance with my invention a mixture of 75 weight per cent of gilsonite, and 25 weight per cent of a 100 neutral distillate blend were heated in a lead bath type heater and introduced into an atmospheric flash chamber with the following results:

Run No.	Heater Bath Temp., ° F.	Heater Outlet Temp., ° F.	Overhead Yield, Wt. Percent
3A	600	580	0
3D	652	648	Trace
4B	700	690	0.5
5B	723	720	1.6
5D	752	739	12.7
7A	777	772	22.2

Example II

In an experiment carried out at 100 mm. Hg abs. in which a waxy gas oil blend was used as solvent and 100 parts per million of an effective anti-foam agent was added, the following results were obtained.

Run No.	Flash Chamber Pres., mm. Hg Abs.	Heater Bath Temp., ° F.	Heater Outlet Temp., ° F.	Overhead Yield, Wt. Percent
13A	100	749	732	33.1
16	100	750	737	31.0

Example III

In a further experiment carried out at 100 mm. abs. in which a topped coker distillate recycled from batch coking of gilsonite was used as solvent, with 100 parts per million of an anti-foam agent added, the following results were obtained:

Run No.	Flash Chamber Pres., mm. Hg Abs.	Heater Bath Temp., ° F.	Heater Outlet Temp., ° F.	Overhead Yield, Wt. Percent
16	100	750	737	31.0

In all the foregoing runs, it will be noted that the heater temperature was maintained considerably above the normal coking temperature of gilsonite with no coking of gilsonite occurring.

While I have found it desirable to direct the flow of heater effluent into a flash or distillation tower as previously described, I also contemplate charging the heater effluent directly to the coking reactor by line **13***a*, by-passing tower **17**. The presence of considerable quantities of diluent along with other gasification products will abstract some heat from the coking reactor bed and the end product distribution obtained in the coking reactor will vary somewhat from that obtained in the heater-tower system because of temperature and processing differences. The desired amine compounds can be recovered along with the diluent in the coking distillate tower **41**. Condensed reaction product with desired diluent properties may be returned to the dissolving tank along with the original diluent.

It is apparent that my invention provides a simple method of treating gilsonite to obtain valuable products therefrom by effectively avoiding exothermic heat and the accompanying formation of coke.

It will be evident to those skilled in the art that various modifications of this invention can be made in view of the foregoing description without departing from the spirit or scope of the disclosure or from the scope of the appended claims.

I claim:

1. The method of recovering valuable hydrocarbon constituents of a normally solid asphaltite, which comprises forming a solution of ground asphaltite together with a high boiling hydrocarbon liquid having an end point of between 700 to 900° F. and directly heating such mixture to a cracking temperature above the incipient coking temperature of the asphaltite in the absence of coke formation, the quantity of liquid and its latent heat being such that the exothermic heat given off by the asphaltite during heating and cracking is largely dissipated in the vaporization of the high boiling liquid.

2. The method of recovering valuable hydrocarbon constituents of a normally solid asphaltite which comprises forming a solution of ground asphaltite with a high boiling liquid hydrocarbon, heating the mixture directly to a cracking temperature above normal coking temperature of the asphaltite in the absence of coking, the quantity of liquid and its latent heat being such that exothermic heat given off by the asphaltite is dissipated in the vaporization of the high boiling hydrocarbon and coking of the asphalite during heating is prevented, and introducing the effluent from the heating step into a frac-

tionator to recover valuable overhead and bottoms products.

3. The method of recovering valuable constituents of a normally solid asphaltite as claimed in claim 2 in which the bottoms from the fractionator are converted to dry coke and vaporous overhead and said overhead is condensed and used in part as the liquid solvent for the asphaltite.

4. The method of recovering valuable constituents of a normally solid asphaltite which comprises forming a solution of ground asphaltite with a high boiling liquid hydrocarbon diluent, heating the mixture directly to a temperature above which the asphaltite normally cokes and at which some cracking of the asphaltite occurs, absorbing exothermic heat from the asphaltite during cracking by direct vaporization of the high boiling liquid hydrocarbonate diluent and preventing the formation of coke during said heating, introducing the vaporized hydrocarbon diluent and cracking products of the asphaltite into a distillation tower, recovering an overhead product including amines and condensible hydrocarbons, withdrawing a side cut of a high boiling hydrocarbon from said tower suitable as the asphaltite diluent, withdrawing asphalt bottoms from said tower and converting said bottoms to a valuable coke product.

5. The method of recovering valuable constituents of a normally solid asphaltite as claimed in claim 4 wherein the withdrawn asphalt bottoms are in the presence of at least a portion of the hydrocarbon diluent.

6. The method of recovering valuable constituents of a normally solid asphaltite as claimed in claim 4 wherein at least a portion of the condensible hydrocarbons are condensed and returned to the tower as a reflux.

7. The method of recovering valuable hydrocarbon constituents and amines from normally solid asphaltites which comprises mixing ground asphaltite with a high boiling liquid diluent, heating the mixture to a temperature above the incipient coking temperature of the asphaltite at which cracking of the asphaltite will occur, removing exothermic heat released by the asphaltite during heating by direct vaporization of the high boiling liquid diluent so that no coking of the asphaltite occurs during cracking, passing heated effluent to a vacuum distillation tower, removing tower overhead comprising amines and hydrocarbon vapors, separating the amines and condensed hydrocarbon vapors and returning at least a portion of the condensed hydrocarbons as tower reflux, withdrawing an intermediate cut from said tower having a high boiling point and suitable as asphaltite solvent, returning said side cut to be mixed with ground asphaltite, withdrawing asphalt bottoms from the vacuum tower and converting said bottoms to a coke product, withdrawing a vapor product from said bottoms conversion, condensing said vapor products and returning a part of the condensed vapor product as asphaltite solvent.

8. The method of recovering valuable hydrocarbon constituents from normally solid gilsonite which comprises mixing ground gilsonite with a high boiling liquid hydrocarbon diluent, heating the mixture to a temperature above the incipient coking temperature of the gilsonite in the absence of coke formation with partial cracking of the gilsonite, removing exothermic heat released by the asphalt during heating by direct vaporization of the high boiling liquid hydrocarbon diluent, passing the vaporous products of heating which include the vaporized diluent to a coking reactor, coking the effluent therein, passing vapors from the coking reaction to a coking distillate recovery tower, withdrawing an overhead product from said recovery tower comprising amines and condensible hydrocarbons, withdrawing a side cut suitable as a gilsonite solvent and withdrawing an asphalt bottoms as product.

9. The method of recovering valuable hydrocarbon constituents of normally solid gilsonite as claimed in claim 8 wherein the amines and hydrocarbons present in the overhead from the distillate recovery tower are separated and at least a portion of the condensed hydrocarbons is returned to the distillate recovery tower.

10. The method of recovering valuable hydrocarbon constituents of normally solid gilsonite as claimed in claim 8 wherein the withdrawn side cut is returned as ground gilsonite diluent and the asphalt bottoms from the distillate recovery tower are returned to the coking reactor.

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