May 6, 1952 W. W. ODELL ET AL 2,595,366

PROCESSING CARBONACEOUS SOLIDS

Filed June 14, 1947 2 SHEETS—SHEET 1

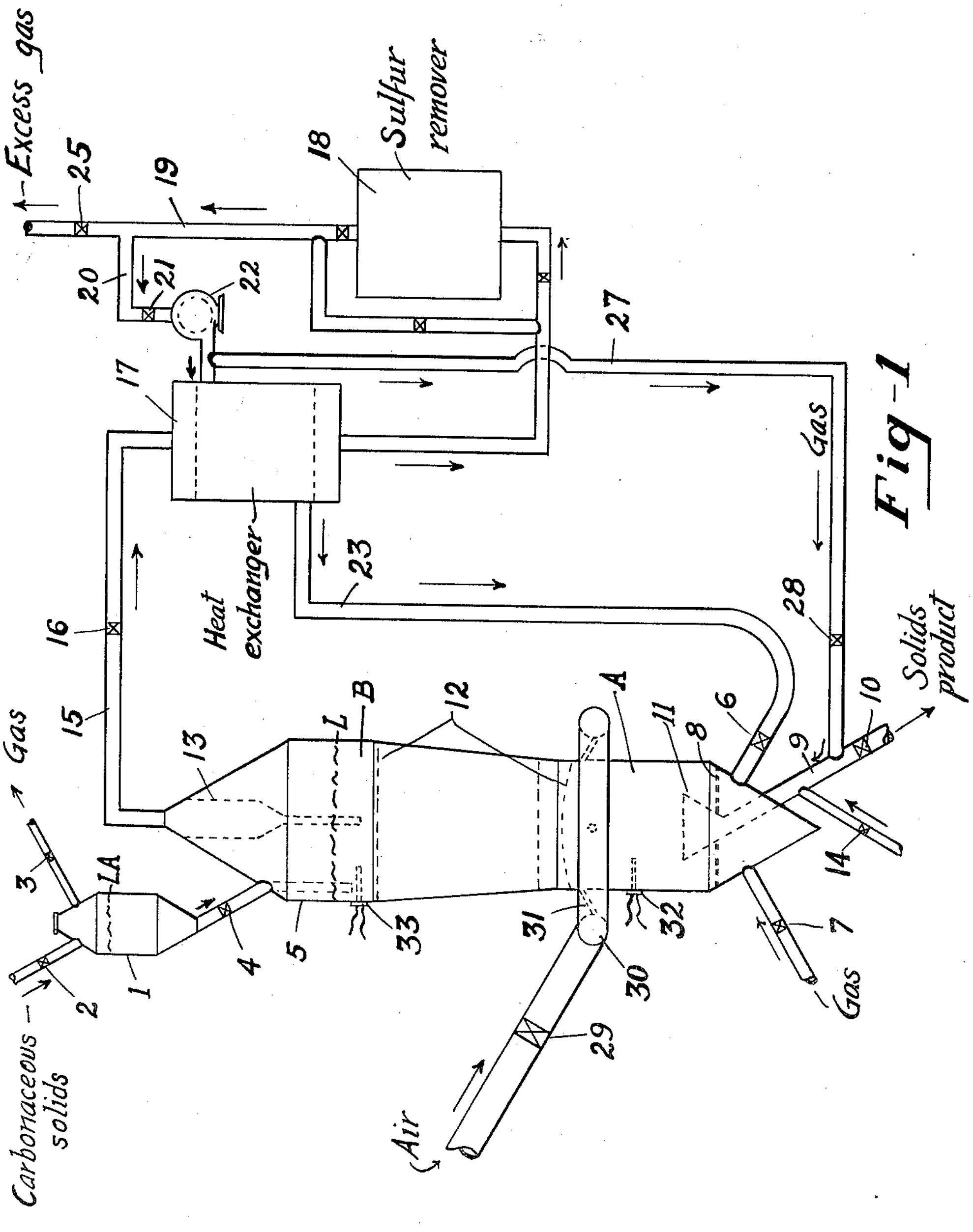

Fig-1

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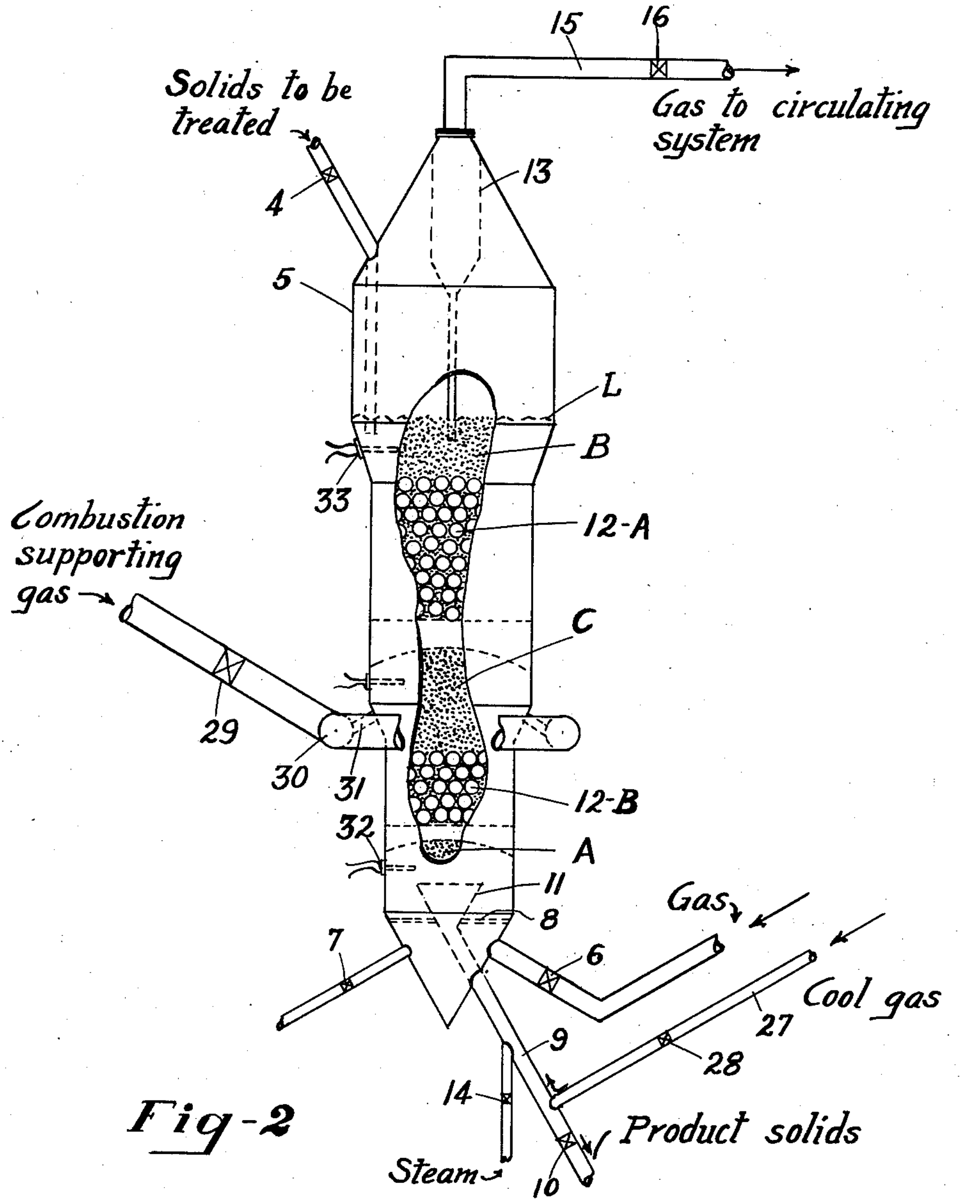

Fig-2

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PROCESSING CARBONACEOUS SOLIDS

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Application June 14, 1947, Serial No. 754,624

12 Claims. (Cl. 202—31)

This invention relates to a process of producing coke or char of low sulfur content, from coal, lignite and other carbonaceous solids of relatively high sulfur content. In particular it has to do with the treatment of coal initially in a finely divided state whereby the ash content is materially reduced, the carbonization of the coal at an elevated temperature, and elimination of the remaining sulfur compounds by subjecting the hot freshly carbonized coal in a fine state of subdivision to the reducing action of a reducing gas such as hydrogen.

When coal is carbonized in a fine state of subdivision, such as occurs when powdered coal is continuously introduced into a fluidized mass of incandescent carbonaceous solids which solids may be previously carbonized coal, the resulting product can be treated, as defined hereinafter, by treatment with a reducing gaseous fluid at a temperature of the order of 400° to 600° C., and the sulfur content of the carbonized product decreased by virtue of contact of the reducing gases with the freshly carbonized finely divided product. It has long been known that reducing gases when passed over hot coke will tend to reduce the sulfur content of the coke but no practical application of this has been possible, so far as we are aware, for a number of reasons. First coking processes have not been developed whereby coking coal could be carbonized in a fine state of division without forming lump coke. Again lump cokes does not readily lend itself to the desulfurizing process by treatment with hydrogen.

Another difficulty in the practical application of known procedures, so far as we are aware, is that of eliminating sulfur present initially in the coal as gypsum. The gypsum at high temperatures of the order of 1300° to 1500° C. is dissociated but at the temperatures common to carbonation dissociation does not occur but rather a partial reduction occurs whereby a calcium sulphide, chiefly the monosulphide, forms and the elimination of this sulfur is a lengthy procedure unless the char or coke under process is very finely divided.

The present invention deals with improvements and combinations whereby the sulfur initially present in various forms in coal can be very largely eliminated in a relatively short period of time yielding a carbonized product in a finely divided state with a very low sulfur content.

One of the objects of this invention is to produce a solid highly carbonaceous finely divided fuel which has so low a sulfur content that it is a preferred fuel for use in metallurgical and gas-

making processes. Another object is to produce a carbonized solid fuel of low sulfur content which can be utilized in the manufacture of briquettes for domestic or other uses. Still another object is to produce an active carbon of low sulfur content which may be used with or combined with catalysts usable in promoting chemical reactions. Other objects will appear from the disclosures made hereinafter.

We have described in Serial No. 726,530 filed in the U. S. Patent Office February 5, 1947, a process by which coal can be largely de-ashed and appreciable portions of the gypsum and pyrite when they are present with the coal can be eliminated therefrom by fluidizing the coal in a finely divided state under definitely prescribed conditions. We have also described in Serial No. 754,623 filed in the U. S. Patent Office June 14, 1947, and now abandoned a process in which the treated (partly de-ashed) coal is carbonized rapidly at high temperature by introducing it in a fine state of division into a hot fluidized mass of finely divided coke to produce a char which has a very large surface area per unit of weight.

According to our present invention coal, thus treated (de-ashed) and quickly carbonized, is subsequently treated with a stream of hot reducing gas such as one comprising hydrogen or hydrogen in admixture with carbon monoxide at a temperature of the order of 400° to 600° C., whereby the sulfur content of the quickly carbonized product is not only materially reduced but it is reduced so rapidly that the process is commercially feasible. Although we do not limit ourselves to the treatment of a freshly carbonized product nor to one that has been quickly carbonized, nevertheless such a fuel is preferable in the rapid production of a product low in sulfur. Whereas our previous inventions, starting with coal, include the carbonization of coal in a fine state of subdivision, preferably by quickly heating it in a continuous process in a hot fluidized bed of coke at a temperature preferably above 900° F. and below about 2000° F., and preferably conducting carbonization in the presence of some steam and/or subsequently steaming the carbonized product for a brief period, the basic or specific feature with which this invention is particularly concerned is the treatment of the carbonized product thus prepared while still hot and while at a temperature of the order of 400° to 600° C. with a reducing gas such as or including hydrogen for a period sufficient to materially reduce the sulfur content of the carbonized product. Best results are obtained when the carbonized product treated with a reducing gas has been freshly prepared as described.

The operation will be better understood with reference to the accompanying drawing, in which;

Figure 1 shows diagrammatically in elevation but largely as a flow diagram one type of apparatus in which the process of this invention may be practiced, adapted for the treatment of previously carbonized finely divided solid fuel at the aforementioned temperatures with a reducing gas; and

Figure 2 shows in a similar diagrammatic manner another embodiment of the invention particularly suited for operation at temperatures above about 1650° F., depicting especially a reactor with a double packed portion.

Referring to Figure 1, reservoir **1** is adapted to receive hot, freshly carbonized, highly carbonaceous finely divided solids from a carbonizer through valve **2**. It functions as a stripper for gases associated with the solids, the gases passing out through valve **3**. The hot carbonized solids pass down under control through valve **4** into reactor **5** wherein they are fluidized by means of gas which may pass chiefly through valves **6** and **7** into the reactor through grid **8**. Gasiform fluid also may pass into **5** through valve **28** and conduit **9**, and also through valve **14**. The reactor has a packed zone **12** located in a middle zone thereof. This packed zone contains large size bodies as shown in similar packed zones in Figure 2. The bed of fluidized solids extends above and below **12** into zones B and A respectively, the top level of the bed being at L. The gas stream from the reactor passes out hot through offtake **15**, valve **16**, heat exchanger **17**, sulfur removal apparatus **18** and offtake **19** from which a portion is bled through valve **25** and a portion passes on through **20**, valve **21**, pump **22**, exchanger **17** and conduit **23** back to the reactor. For cooling purposes gas may be returned to the reactor through bypass conduit **27** and valve **28**. The sulfur removal may be effected in any known manner as by contacting the sulfur containing gas with a sulf-reactive metal or the like.

Fresh reducing gas may be supplied to the system through valve **7**. Combustion supporting gas may be introduced into the reactor through valve **29**, and bustle pipe **30**. Thermocouples are shown at **32** and **33**.

In Figure 2 the same system of numbering is employed except that the packed portions of the reactor **5** are numbered **12A** and **12B**, and the free zone wherein finely divided solids may be in free fluidized motion are lettered A, B, and C.

Irrespective of the packed zones, **12** of Figure 1 and **12**–A and **12**–B of Figure 2, it will be understood that the fluidized bed of solids is continuous from top level L to the bottom zone A, the bed density in the packed zone being less than in the non-packed zones. The packing is supported on supporting grids which are pervious to the flow of gas therethrough and in which the finely divided solids are substantially in a fluidized state.

Example 1

Referring to Figure 1 and considering the treatment of a carbonized de-ashed coal made by quickly heat treating finely divided coal in a fluidized bed of finely divided coke at a temperature above 900° F. and below 2200° F. preferably at 1050° to 1650° F. the operation may be conducted as follows: Pass the hot, freshly carbonized coal, which may be called char, from a carbonizer down through valve **2** into stripping reservoir **1** at a controlled rate which is sufficient to maintain a level therein as at LA with valve **4** open and allow the hot char to pass downwardly into reactor **5** at a rate adapted to maintain the bed level therein at L, meanwhile allowing stripped gas from **1** to pass out through valve **3**. Fluidizing gas is introduced through valve **6** and as needed through valve **7** also, the combined volume of fluidizing gas being sufficient, at least, to maintain the char in **5** in a fluidized state. The gas from **6** and **7** passes up as a stream through porous member **8** into the bed of char. The gas introduced through **6** is a reducing gas preferably substantially free of H_2S and at a temperature above 100° F. and preferably above 600° F. but below that temperature at which damage to equipment might occur. The treated char is removed from the reactor through conduit **9** and valve **10**, preferably continuously at a rate adjusted to suit the desired reduction in the sulfur content of the char; the rate being synchronized with the feed rates so that the levels L and LA are maintained.

The gas stream passing up through grid **8** and flaring member **11** passes serially through a free zone A, the packed portion **12** and free zone B and then through dust separator **13**, offtake **15**, valve **16**, heat exchanger **17** wherein its temperature is reduced, sulfur removal vessel **18**, conduits **19** and **20**, valve **21**, pump **22** and back through exchanger **17** wherein its temperature is raised, through conduit **23** and valve **6** and is again circulated through reactor **5**. Excess gas is bled from the system through valve **25**; additional fresh reducing gas as may be required is introduced through valve **7**. The by-pass conduit **27** with valve **28** is used to reduce the temperature of the outgoing treated char by allowing cooled gas to pass up through conduit **9** and through the char in flaring member **11**.

Sulfur exists in lignite, coal, coke, char and certain other carbonaceous solids in a number of different forms which should be considered in treating them at elevated temperatures. For example the sulfur present as free sulfur, held in solid solution, upon reacting with hydrogen generates a small amount of heat; that is, the reaction is exothermic substantially as follows:

(1) $H_2+S=H_2S+3.9$ calories

This reaction is not favored by extremely high temperatures; a temperature approximately or somewhat above the boiling point of sulfur is preferred. Similarly ferrous sulfide is reduced by reaction with hydrogen with the evolution of heat as in Equation 2.

(2) $FeS+H_2=H_2S+Fe+14.3$ calories

At higher temperatures the endothermic reaction expressed by Equation 3 occurs

(3) $FeS+H_2O=FeO+H_2S-17.6$ calories

Although water vapor is not usually considered a reducing gas, it may advantageously be employed with reducing gas as a fluid for treating hot carbonaceous solids in a fine state of subdivision at high temperatures, namely above about 1500° F. Under these conditions the reaction for the removal of sulfur is more complex than indicated by Equation 3. For example carbon reacts with steam at high temperatures forming carbon monoxide and hydrogen, also by endothermic reactions; the carbon monoxide, hydrogen and carbon are reducing agents tending to reduce iron sulfide and oxides to iron simultaneously forming volatile sulfur compounds.

The heat recovered in the heat exchanger **17** of Figure 1 is usually sufficient to carry on the operation, when the char supplied through valve **2** is at a high temperature. However, when considerable sulfur is to be removed or when the temperature of the char thus fed to the reactor **5** is not at a sufficiently high temperature to maintain the desired temperature in the bed of char in process the extra heat may be supplied by introducing a relatively small amount of air or other combustion supporting gas as through valve **29**, bustle-pipe **30** and inlets **31**.

Summarily the operation comprises passing a stream initially comprising essentially a reducing gas at an elevated temperature upwardly through a bed of finely divided solid char, preferably fluidized, while said char is at an elevated temperature suitable for the chemical reaction of sulfur contained therein, continuously feeding fresh hot char to an upper portion of the bed and continuously withdrawing the treated char from a zone of said bed adjacent the bottom thereof, meanwhile introducing a relatively small amount of steam and/or air into a zone of said bed adjacent the bottom thereof, as required; the total amounts of air and steam used preferably should be kept to such low limits that the gas stream as discharged through offtake **15** of Figure 1 is still a reducing gas.

Under some conditions the gas discharged from stripper **1** through **3** of Figure 1 is a reducing gas and this, under these conditions, may be circulated as a reducing gas using valve **7** as a control. However, it will be noted that in applying the foregoing disclosure the reducing gas circulated through the system may be generated in amounts required by the use of the small amounts of steam and combustion supporting gas described when the temperature in the fluidized bed of char in reactor **5** is maintained at a temperature above about 1600° F. and preferably in the range 1650° to 1750° F. Higher temperatures may be employed but commonly employed alloy steels are not so satisfactory as materials of construction of certain exposed equipment at these temperatures.

Referring to Figure 2 the operation is substantially as described but additional economies are obtainable. By introducing a combustion supporting fluid in zone C separated from zones A and B by packed zones **12A** and **12B** respectively it is possible to maintain a high temperature in zone C and a lower temperature in zones A and B. Zone B is cooler because of the endothermic water gas reaction in the bed and also because fresh feed is or may be supplied at a lower temperature through feed valve **4**. Zone A may be appreciably cooler by virtue of cooling fluid introduced therein through valve **28**, and/or valve **14**. Similarly a cooling gasiform medium may be introduced through valve **7**. In this manner the treated char not only contacts the reducing gas after passing through the hottest zone or combustion zone C but some of the sensible heat initially in the char as it leaves zone C is returned thereto in the upwardly moving stream of reducing gases from zone A. This not only permits a heat economy to be made but it minimizes the amount of combustion supporting fluid which may be required in zone C.

In carrying out the desulfurization operation as described there is a relationship between the initial size of particles of char treated, the duration of the treating period, the amount of sulfur reduction and the velocity of the gas stream passing up through the char in process. The finer the size of the char the lower the stream velocity should be. For any particular char the optimum superficial velocity can be determined by experiment but it has been found that with mean particle size of 200 mesh and of 6 mesh the approximate velocities of the gas stream leaving the bed, for satisfactory conditions, should be about 0.35 and 2.5 feet per second respectively; velocity calculated as for a gas stream in an empty reactor at standard conditions of temperature and pressure. The finer the size of char particles treated the more readily or more quickly is the sulfur content reduced. Whereas the size of char used depends upon the ultimate use to which the treated product is put, it is preferable, other factors remaining the same, to treat char having a mean particle size less than one-eighth (⅛) inch. The sulfur content of the treated product, without using excessive or uneconomical amounts of reducing gas, may be as low as 0.1 percent or even lower according to size of char treated, the initial sulfur content of the feed char, and the duration of the treating period.

In the foregoing consideration has been given primarily to the removal of sulfur and its compounds from carbonized carbonaceous solids (char). It is not always desirable to precarbonize before treating. In other words finely divided carbonaceous solids such as oxidized coal, animal bones, and certain other materials may be treated as described by feeding them into the reactor without precarbonization. Bone is treated to make a carbonaceous absorbent rather than to desulfurize.

The residence time of the carbonaceous solids within the reactor in counter contact with hot gases is usually more than one minute and the total depth of the fluidized bed is preferably more than 10 feet and may advantageously be more than 20 feet particularly as the diameter increases from about 5 feet. With the fluidized solids descending through the reactor at a mean velocity of 5 feet per minute, the duration of residence time of the carbonaceous solids in the reactor, when the bed is 20 feet deep, is 4 minutes. Employing the latter residence time and using a gas velocity of say 1 foot per second the approximate volume of gas passed per square foot of mean sectional area of the reactor during the 4 minutes is 240 cubic feet. The approximate quantity of carbonaceous solids treated with this 240 cubic feet of gas in 4 minutes is say 300 pounds. In order to increase the volume of gas used per unit weight of carbonaceous solids treated one can decrease the rate of discharging the treated char which increases residence time in the reactor, or within certain limits, increase the gas velocity, or both. It will be noted that the bed density in the reactor as shown in the Figures at 5 is less in the packed portion (or portions) than in the remainder of the bed.

Organic sulfur reacts with steam yielding hydrogen sulfide and carbon dioxide commonly by reactions which are endothermic and these reactions are favored by high temperatures; the overall reactions wherein the carbon dioxide formed reacts with incandescent carbon to form carbon monoxide absorb a greater amount of heat than the straight sulfur reactions. Sulfur combines with carbon monoxide to form carbonyl sulfide and this reaction is exothermic but occurs at an elevated temperature. The total amount of heat energy due to the oxidation or removal of sulfur from the char is very small, but elevated temperatures are required for promoting the desulfurizing reactions. It will become evident that, with good heat exchange in exchanger **17** of the figures, only a small amount of heat energy need be supplied to the system and this can be accomplished by introducing the combustion supporting fluid as through **29** and **30** as described. By removing the treated char from the reaction chamber at a lower temperature than that at which it is fed into the reactor makes possible the further economy of heat.

Before defining my claims attention is called to the fact that the operation described can be conducted at reduced pressure, normal, or at superatmospheric pressure. It is usually economical to operate at pressures not appreciably greater than atmospheric. The duration of time the solids in process are resident in the reactor is determined by the result sought. Samples of the treated material as discharged through **9** and **10** of the figures are analyzed and if they signify undertreatment the residence time is increased by decreasing the rate of discharge of solid through **9** and **10** and, when desired, the rate of circulation of reducing gas is increased. The circulation rate can only be varied within limits determined by the quantity of blown over solids and by the desirability to maintain the solids in a fluidized state in the reactor.

Oxidation, conducted at a temperature below 600° C. or about 500° to 550° C. with oxygen is conducive to the removal of sulfur from such products as high temperature coke and this is particularly true of cokes containing only small amounts of sulfate sulfur and a larger amount of other forms of sulfur. This preferential oxidation of sulfur instead of the carbon of the coke is possible because the ignition temperature of the coke is above 550° C. We find that many carbonaceous solid fuels that contain sulfur compounds, such as low temperature coke, ignite at temperatures below 550° C. and hence preferential oxidation of sulfur is not possible at the latter temperature. Frequently its oxidation with combustion of some carbon is not economical.

It is economical to promote some oxidation reactions, as described above, wherein the heat generated is utilized in maintaining the system at the chosen temperature, wherein the overall result of reaction is the production of a reducing gas, and wherein the carbonaceous solids thus treated are finally subjected to a strongly reducing atmosphere before they are finally discharged from the reactor in which they are treated. We find that oxidation of sulfur in solid fuels by blasting with air occurs with most fuels, simultaneous with oxidation of carbon and the difficulty heretofore experienced has been that the accompanying oxidation of carbon generates so much heat that difficulties are experienced. It is necessary, to obtain optimum results, to maintain a quite uniform temperature throughout the mass of solids being treated, in the oxidation zone, or rather in the zone in which combustion supporting gas is introduced. This cannot be accomplished in a fixed bed but is readily accomplished in the manner described above.

The foregoing description and examples of operations have been presented to show specific applications of this invention and some of the results attainable by its use. Other modifications

will be obvious to those skilled in the art hence only such limitations should be imposed on the invention as are indicated in the following claims.

We claim:

1. The process of beneficiating carbonaceous solids in the form of finely divided de-ashed and carbonized coal comprising passing a mass of said solids substantially continuously downwardly in a fluidized state through a stationary porous mass of substantially uniformly sized, relatively large-size heat-resistant solids confined in a reaction chamber, while passing a stream of hot, initially strongly reducing gas upwardly therethrough in counter current contact with the said carbonaceous solids, introducing into the gas stream during its contact with said carbonaceous solids in said reactor a relatively small amount of a combustion supporting gas and promoting combustion reactions in said stream in contact with the latter solids, controlling the supply of said combustion-supporting gas so as to generate the heat in the reactor while maintaining the resulting reacting gas reducing, discharging the resulting reducing gas stream overhead, substantially continuously discharging the thus treated carbonaceous solids from beneath the mass of said large-size solids while continuously feeding fresh de-ashed and carbonized coal to be treated to the reactor above the latter mass, synchronizing the feed and discharge of said carbonaceous solids so that a continuous deep bed of the latter solids is maintained in said reactor, said bed extending over the entire cross-sectional area of said chamber from an upper point substantially above said bed through said bed to a lower point substantially below said bed, and maintaining the latter bed in a fluidized state over its entire height by controlling the velocity of the gaseous stream passing therethrough.

2. The process of beneficiating carbonaceous solids in the form of finely divided de-ashed and carbonized coal comprising passing a mass of said solids substantially continuously downwardly in a fluidized state through a stationary porous mass of substantially uniformly sized, relatively large-size heat-resistant solids confined in a reaction chamber, while passing a stream of hot initially strongly reducing gas upwardly therethrough in counter current contact with the said carbonaceous solids, maintaining the latter solids while in said chamber as a deep continuous fluidized bed, introducing into the gas stream at a zone between the top and bottom of said bed a combustion supporting gas promoting combustion therein sufficient to maintain the solids while in said zone at a temperature above about 500° C. but below about 1100° C., and insufficient to destroy the reducing character of the resulting gas stream, discharging the resulting gaseous stream overhead and withdrawing the thus treated carbonaceous solids adjacent the bottom of said bed, meanwhile feeding fresh de-ashed and carbonized coal to be treated substantially continuously to a top portion of said bed.

3. The process of desulfurizing a solid fuel in the form of finely divided de-ashed and carbonized coal containing sulfur comprising passing a mass of said finely divided hot solid fuel substantially continuously downwardly in a fluidized state through a stationary, porous mass of substantially uniformly sized relatively large-size heat-resistant solids confined in a reaction chamber while passing a stream of hot initially strongly-reducing gas upwardly therethrough in counter current contact with said fuel, discharging the resulting gaseous stream overhead, maintaining said fuel

while in said chamber in a deep continuous fluidized bed extending over the entire cross-sectional area of said chamber from an upper point substantially above said mass to a lower point substantially below said mass, and discharging the thus treated fuel from adjacent the bottom of said bed at such a controlled rate that the continuity of said fluidized bed between said points is maintained and the residence time of said fuel in said chamber in contact with said reducing gas is sufficient to lower the sulfur content of said fuel.

4. The process defined in claim 3 in which the hot fuel is supplied to the reaction chamber at a temperature above 200° C. but below about 1100° C.

5. The process defined in claim 3 in which the hot reducing gas is introduced into the reaction chamber at a temperature of the order of 100° to 500° C.

6. The process defined in claim 3 in which the hot fuel is fed to the reaction chamber at a temperature of the order of 400° to 1000° C. and the hot gas is supplied to the said chamber at a temperature of the order of 100° to 300° C.

7. The process defined in claim 3 in which some of the gas discharged overhead is returned to the reaction chamber as at least a part of the stream of strongly reducing gas.

8. The process defined in claim 3 in which the coal treated has been previously subjected to dry treatment for the removal therefrom of pyritic sulfur and gypsum.

9. The process of beneficiating finely divided de-ashed and carbonized coal, comprising passing a mass of said carbonized coal substantially continously downwardly in a fluidized state through a stationary porous bed of heat-resisting large-size solids confined in a reactor and occupying a portion only of the height of said reactor while passing a stream of hot reducing gas upwardly therethrough in countercurrent contact with said carbonized coal, maintaining the latter solids in said fluidized state in said reactor at temperatures above 400° C. but below about 1100° C., discharging the gas stream overhead, maintaining said mass in said reactor as a deep continuous fluidized mass extending over the entire cross-sectional area of the reactor from an upper point substantially above said bed, through said bed to a lower point substantially below said bed, the apparent density of said fluidized mass being lower within said bed than in reactor portions above and below said bed, substantially continuously withdrawing the thus treated carbonized coal from adjacent the bottom of said fluidized mass, substantially continuously feeding fresh carbonized and de-ashed coal to be treated to a portion of the latter mass adjacent the top thereof at a rate adapted to maintain the depth of said fluidized mass in said reactor substantially uniform, and controlling the rates of solids charge and withdrawal so as to maintain the continuity of said fluidized mass between said upper and lower points.

10. The process of beneficiating carbonaceous solids in the form of finely divided de-ashed and carbonized coal, comprising passing a mass of said carbonized coal substantially continuously downwardly in a fluidized state through two stationary porous beds of heat-resistant large-size solids confined in superimposed spaced relationship within a reactor, passing a stream of hot reducing gas upwardly through said reactor in countercurrent contact with said carbonaceous solids, maintaining the latter solids in said fluidized state in said reactor at temperatures above 400° C. but below about 1100° C., discharging the gas stream overhead, maintaining said mass in said reactor as a deep continuous fluidized mass extending over the entire cross-sectional area of said reactor from an upper point substantially above the upper one of said beds, through said beds and the space between said beds to a lower point substantially below the lower one of said beds, the apparent density of said fluidized mass being lower within said beds than in reactor portions above said upper bed, between said beds and below said lower bed, substantially continuously withdrawing the thus treated carbonaceous solids from the bottom of said fluidized mass, substantially continuously feeding fresh carbonized and de-ashed coal to be treated to a top portion of said mass at a rate to maintain the depth of said mass substantially uniform, and controlling the rates of solids charge and withdrawal so as to maintain the continuity of said fluidized mass between said upper and lower points.

11. The process of claim 10 in which a combustion-supporting gas is supplied to said space between said beds at a rate adequate to generate heat by combustion but insufficient to destroy the reducing character of said gas.

12. The process of claim 11 in which the temperature of said fluidized mass within said space is higher than the temperature of said fluidized mass above said upper bed and below said lower bed.

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