

PATENT SPECIFICATION

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(54) CARBON BLACK PROCESS AND APPARATUS

(71) We, PHILLIPS PETROLEUM COMPANY, a corporation organised and existing under the laws of the State of Delaware, United States of America, of Bartlesville, Oklahoma, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for producing carbon black and apparatus for such process.

In one of its more specific aspects, this invention relates to the injection of hydrocarbon feed into carbon black furnaces.

There has recently been invented a process for the production of carbon black by the pyrolytic decomposition of a hydrocarbon feed in which feed-stock is brought into contact with hot combustion gases as a highly concentrated mass of finely divided particles. The feedstock is brought to a temperature within the lower range of carbon black forming temperatures and passed as a hot concentrated mass through the reaction zone at low velocities to produce an exceptionally large particle, low structure black.

This process is carried out in a vertical reactor by injecting the hydrocarbon feed-stream downwardly into the reactor into the hot combustion gases, the hot combustion gases being produced by the oxidation of a fuel with an oxidant, preferably air, the air being supplied in an amount within the range of 240 to 450 SCF per US gallon of hydrocarbon feed. The reaction mass formed of the feed and combustion gases is conducted through the reaction zone at a velocity within the range of 3.5 to 11 feet per second at a temperature within the range of from 2400°F to 2500°F. The reaction is carried out at reactor velocities in the range 3.5—11 feet per second and at temperatures

within the lower 15 percent of the range of carbon black formation temperatures. The preferred reactor operating pressures are from 15 psia to 30 psia.

Much of the success of the process depends upon the speed with which the preheated hydrocarbon feed is brought within the prescribed operating conditions, that is, how rapidly the hydrocarbon feed can be formed into a concentrated mass within the combustion gases at the desired velocity and temperature.

It is sought to achieve these objects by introducing downwardly into the reactor the hydrocarbon feed, at least in part at supersonic velocities. This invention seeks to provide a method and apparatus for doing so.

According to this invention there is provided a process for the production of carbon black by pyrolytic decomposition of a hydrocarbon feedstock, which comprises introducing the feedstock as a mass of finely-divided particles downwardly into a pyrolysis zone containing a mass of hot combustion gas produced by the combustion of a fuel in the presence of a free oxygen-containing gas supplied to the pyrolysis zone in an amount equivalent to from 240 to 450 standard cubic feet of air per U.S. gallon of feedstock, conducting the reaction mass so formed downwardly through the pyrolysis zone at a velocity in the range 3.5 to 11 feet per second and at a temperature in the range 2400°F to 2500°F, and recovering the carbon black product from the effluent from said zone, wherein at least part of the feedstock is introduced into said zone at supersonic velocity.

Also provided according to the invention is a carbon black reactor comprising a substantially cylindrical pyrolysis chamber disposed about a vertical axis and having inlets in the upper end for hydrocarbon feed, fuel, and free oxygen-containing gas, and an outlet in the lower end for the

11 recovery of product carbon black, and comprising a hydrocarbon feed introduction nozzle fitted in the upper end of the said chamber and connected to the said hydrocarbon feed inlet, the said nozzle comprising at least one port adapted to inject the feed at supersonic velocity.

5 The nozzle forming part of the reactor of the invention facilitates the method of the invention partly by establishing shock waves at the exit from the port or ports so as to disperse the hydrocarbon feed and to enable it to be brought rapidly to the optimum conditions under which it is desired to conduct the pyrolytic decomposition.

10 The method and apparatus of this invention are illustrated by reference to the attached drawings in which various embodiments of the nozzle forming part of a reactor of the invention are depicted.

20 FIGURES 1 and 2 depict a nozzle comprised of a single port, the discharge through the port being supersonic.

25 FIGURES 3 and 4 depict a nozzle comprised of a plurality of ports, the discharge through all ports being supersonic.

30 FIGURES 5 and 6 depict a nozzle comprised of a port centrally positioned to a plurality of encircling ports, the velocity through the centrally positioned port being supersonic and the velocity through the encircling ports being subsonic.

35 Referring now to FIGURES 1 and 2, there is shown nozzle 1 adapted at its inlet end 7 with suitable means, for example, threads 2, for attachment to the feed introducing conduit and having an outlet end 3. The passageway through the nozzle is comprised of two sections 4 and 5, section 4 converging and section 5 diverging from throat 6. Both the convergence of section 4 and the divergence of section 5 can be at any angle while producing supersonic flow although a convergence of from about 38 to 40° and a divergence of about 30° have been found satisfactory. Any diametric relationship can be used for sections 4 and 5 which provide a supersonic velocity of the hydrocarbon feed from outlet end 3.

50 Referring now to FIGURES 3 and 4, there is shown nozzle 11 adapted at its inlet end 17 with suitable means, for example, threads 12, for attachment to the feed introductory conduit, and having an outlet end 13. A plurality of passageways 18 are formed of sections 14 and 15, these being of converging and diverging configuration, respectively. Throats 16 are located between sections 14 and 15. In all aspects, except in that a plurality of passageways exist through the nozzle, it is comparable to the nozzle of FIGURE 1.

60 Any number of passageways can be employed, these passageways being disposed in any configuration but preferably being comprised of at least one centrally-disposed passageway encircled by a ring of passageways. Similarly, all passageways can be of equal dimensions or the passageways can be of different dimensions to impart different velocities to the fluid passing through the passageways, with all velocities being supersonic.

65 Referring now to FIGURES 5 and 6, there is shown nozzle 21 adapted at its inlet end 27 with suitable means, for example, threads 22, for attachment to the feed introductory conduit and having an outlet end 23. In this nozzle there exists a central passageway comprised of converging and diverging sections 24 and 25 and throat 26 through which supersonic velocity is imparted to the hydrocarbon feed and a series of passageways 29 through which velocities less than supersonic are imparted to a portion of the hydrocarbon feed. If desired, an annulus can be substituted for the series of passageways. Also, if desired, a centrally positioned opening can be used for that feed entering at subsonic velocities and at least one peripherally-positioned passageway can be employed to introduce feed at supersonic velocities. The annulus or the individual passageways can be positioned parallel to the centerline of the nozzle or at an angle thereto.

75 The method of this invention produces, under the predefined operating conditions, carbon blacks which are significantly different than those blacks produced by multi-stream nozzles emitting at less than supersonic velocities.

80 Those nozzles shown in FIGURES 1 and 5 of the drawing were employed in the production of a large particle carbon black, and a multiport subsonically-emitting nozzle was employed for comparison purposes, in a vertical reactor in a best mode of practicing the invention. Air as oxidant was introduced into the reactor through its circumferential periphery upstream at about the locus of the discharge of the feed nozzle and also downstream thereof at a distance approximately equal to the diameter of the reactor. The reactor pressure was 19 psia in all runs.

90 Results were as follows:

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Nozzle	Figure 1	Figure 5	Subsonic Multistream
Hydrocarbon Feed, GPH	96	93	88
Atomizing Air, SCFH	6,000	6,000	6,000
Axial Air, SCFH	5,000	5,000	5,000
Circumferential Air, Upstream, SCFH	15,000	15,000	15,000
Circumferential Air, Downstream, SCFH	15,000	15,000	15,000
Circumferential Gas Rate, Upstream, SCFH	1,000	1,000	1,000
Product Analysis			
Photometer	90	89	92
N ₂ Surface Area, m ² /g	16.2	20.2	22.6
Structure, cc/100 gm (DPB)	32	29	30
Yield, #/gal.	5.06	4.67	4.72
Carbon Deposit on Reactor	None	None	Film

Nozzle of FIGURE 1 had convergent included angle of 38° and a divergent included angle of 30°. Nozzle of FIGURE 5 had a convergent included angle of 60° and a divergent included angle of 30°.

The above data illustrate the operability of the method and apparatus of this invention. They further indicate that the employment of the supersonic nozzles was carried out without carbon deposition on the reactor whereas, in contrast, carbon was deposited on the reactor when employing the subsonic nozzle under otherwise substantially identical conditions. Accordingly, it is seen that operations with the supersonic nozzles will extend the operating cycle of the system.

WHAT WE CLAIM IS:—

1. A process for the production of carbon black by pyrolytic decomposition of a hydrocarbon feedstock, which comprises introducing the feedstock as a mass of finely-divided particles downwardly into a pyrolysis zone containing a mass of hot combustion gas produced by the combustion of a fuel in the presence of a free oxygen-containing gas supplied to the pyrolysis zone in an amount equivalent to from 240 to 450 standard cubic feet of air

per U.S. gallon of feedstock, conducting the reaction mass so formed downwardly through the pyrolysis zone at a velocity in the range 3.5 to 11 feet per second and at a temperature in the range 2400°F to 2500°F, and recovering the carbon black product from the effluent from said zone, wherein at least part of the feedstock is introduced into said zone at supersonic velocity.

2. A process according to claim 1, wherein the feedstock is introduced into said zone as a plurality of separate streams at least one of which has a supersonic velocity.

3. A process according to claim 1 or 2, wherein the whole of the feedstock is introduced into said zone at supersonic velocity.

4. A process according to claim 2, wherein at least one of the remaining streams has a subsonic velocity.

5. A process according to claim 4, wherein the feedstock is introduced into said zone as a plurality of peripheral streams of subsonic velocity arranged around a central stream of supersonic velocity directed along the axis of said zone.

6. A process according to claim 1, substantially as hereinbefore described.

7. A carbon black reactor comprising a

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Hydrocarbon Feed, GPH	96	93	88
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Axial Air, SCFH	5,000	5,000	5,000
Circumferential Air, Upstream, SCFH	15,000	15,000	15,000
Circumferential Air, Downstream, SCFH	15,000	15,000	15,000
Circumferential Gas Rate, Upstream, SCFH	1,000	1,000	1,000
<u>Product Analysis</u>			
Photometer	90	89	92
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per U.S. gallon of feedstock, conducting the reaction mass so formed downwardly through the pyrolysis zone at a velocity in the range 3.5 to 11 feet per second and at a temperature in the range 2400°F to 2500°F, and recovering the carbon black product from the effluent from said zone, wherein at least part of the feedstock is introduced into said zone at supersonic velocity.

2. A process according to claim 1, wherein the feedstock is introduced into said zone as a plurality of separate streams at least one of which has a supersonic velocity.

3. A process according to claim 1 or 2, wherein the whole of the feedstock is introduced into said zone at supersonic velocity.

4. A process according to claim 2, wherein at least one of the remaining streams has a subsonic velocity.

5. A process according to claim 4, wherein the feedstock is introduced into said zone as a plurality of peripheral streams of subsonic velocity arranged around a central stream of supersonic velocity directed along the axis of said zone.

6. A process according to claim 1, substantially as hereinbefore described.

7. A carbon black reactor comprising a

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- substantially cylindrical pyrolysis chamber disposed about a vertical axis and having inlets in the upper end for hydrocarbon feed, fuel, and free oxygen-containing gas and an outlet in the lower end for the recovery of product carbon black, and comprising a hydrocarbon feed introduction nozzle fitted in the upper end of the said chamber and connected to the said hydrocarbon feed inlet, the said nozzle comprising at least one port adapted to inject the feed at supersonic velocity.
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8. A reactor according to claim 7, in which the nozzle comprises at least one other port adapted to inject the feed at subsonic velocity.
9. A reactor according to claim 8, in which the nozzle comprises a single port adapted to inject feed at supersonic velocity and a plurality of ports adapted to inject feed at subsonic velocity arranged around said single port.
10. A reactor according to claim 8, in which the nozzle comprises a single port adapted to inject feed at supersonic velocity and a single port adapted to inject feed at subsonic velocity, the latter taking the form of an annulus arranged around the former.
11. A reactor according to any one of claims 7—10, in which the supersonic port or ports each comprises a convergent entry passageway and a divergent exit passageway separated by a throat, the walls of the entry passageway converging at an angle of from 38° to 40° and the walls of the exit passageway diverging at an angle of about 30°.
12. A reactor according to claim 7, comprising a hydrocarbon feed introduction nozzle substantially as hereinbefore described with reference to Figs. 1 and 2, or 3 and 4, or 5 and 6, of the accompanying drawings.
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