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(54) Title: CARBON INJECTION WITH THE CHARGED IRON OXIDE INSIDE DIRECT REDUCTION PLANT (DRP)-SHAFT FURNACES

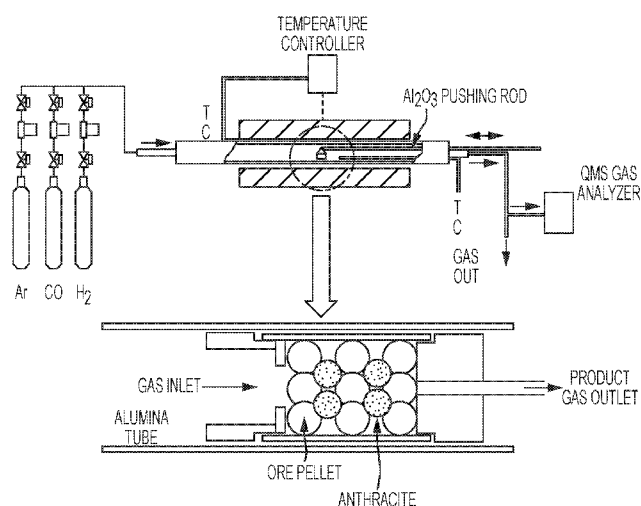


FIG. 1

(57) Abstract: Certain embodiments are directed to methods of producing direct reduced iron (DRI), the methods including contacting a target mixture comprising a solid carbonaceous reductant and an unreduced iron material (e.g., Fe_2O_3) with a reducing gas comprising carbon monoxide (CO) under conditions sufficient to produce reduced iron (Fe).

Published:

— *with international search report (Art. 21(3))*

CARBON INJECTION WITH THE CHARGED IRON OXIDE INSIDE DIRECT REDUCTION PLANT (DRP)-SHAFT FURNACES

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application claims the benefit of priority of U.S. Provisional Application No. 62/412,889, filed October 26, 2016, which is hereby incorporated by reference in its entirety.

BACKGROUND

[0002] Iron oxide-containing materials used in iron and steelmaking processes, such as taconite, typically contain high concentrations of gangue in addition to the iron oxide. It is known that the iron concentration may be enriched by finely grinding the iron oxide-containing materials and then magnetically separating the iron oxide from the gangue. The refined, finely ground iron is then formed into pellets. Firing the pellets in a reducing atmosphere, such as a mixture of carbon monoxide and hydrogen, converts iron oxide in the pellets to metallic iron. Pellets containing high concentrations of metallic iron are referred to as direct reduced iron (DRI) pellets. The highly metallized nature and high purity of direct reduced iron pellets makes it desirable to use direct reduced iron pellets for production of steel by smelting in electric furnaces or various other techniques.

[0003] DRI is then melted, preferably in an electric arc furnace (EAF), to produce molten iron and transformed into liquid steel. Direct reduction plants typically comprise a continuous moving bed reactor discharging hot or cold DRI. The term "cold DRI" is applied to DRI discharged at temperatures preferably below about 100 °C, and the term "hot DRI" is applied to DRI discharged at temperature typically above about 400 °C.

[0004] DRI, melted-down in electric arc furnaces, is usually mixed with scrap in selected proportions according to the economic cost of the charge materials and the attainable quality of the final steel products. The melting furnaces utilize both electrical and chemical energy for decreasing the tap-to-tap time thus increasing the productivity of the furnace. In this respect, DRI containing a high proportion of combined carbon (about 3%) is significantly beneficial, because this carbon chemically combines with oxygen injected into the furnace producing heat and a foamy slag resulting also a number of other advantages.

[0005] There remains a need for producing DRI in a more efficient and cost effective manner.

SUMMARY

[0006] The present invention provides a more efficient process for producing DRI and includes methods for producing direct reduced iron pellets using a process that partially replaces reducing gas by adding a solid carbon reductant (e.g., anthracite) in Direct Reduction Plants (DRP). The solid carbon reductant decreases the amount of natural gas (NG) consumption and results in a higher productivity (double reducing agent). The expected savings in NG can be used for extra production of Direct Reduced Iron (DRI). Studies described herein investigate the reduction behavior of the iron oxide pellets mixed with coke in presence of reducing gas mixture (H_2/CO) together with the associated effect on reduction rate of iron oxide pellets and carbon gasification rate of coke.

[0007] Certain embodiments are directed to methods of producing direct reduced iron (DRI), the methods including contacting a target mixture comprising at least 5, 10, 15, or 20 wt.% of a solid carbonaceous reductant and at least 70, 75, 80, 85, or 90 wt.% of an unreduced iron material (e.g., Fe_2O_3) with a reducing gas comprising carbon monoxide (CO) under conditions sufficient to produce reduced iron (Fe). Other embodiments are directed to methods of producing direct reduced iron (DRI), the methods comprising contacting a mixture comprising at least 5, 10, 15, or 20 wt.% of anthracite and at least 70, 75, 80, 85, or 90 wt.% an unreduced iron material (Fe_2O_3) with a reducing gas comprising carbon monoxide (CO) under conditions sufficient to produce reduced iron (Fe). Still other embodiments are directed to methods of producing direct reduced iron (DRI), the methods comprising contacting a mixture comprising 5 wt.% to 20 wt.% of anthracite and 80 to 95 wt.% an unreduced iron material (Fe_2O_3) with a reducing gas comprising carbon monoxide (CO) at 850 °C to 1100 °C to produce reduced iron (Fe).

[0008] The target mixture can be contained or positioned in a direct reduction reactor. In certain aspects the iron containing component of the mixture is in the form of an iron oxide containing pellet. The solid carbonaceous reductant can be in the form of carbon containing particles, e.g., anthracite particles. In certain aspects the method is performed at a temperature of or about 800, 850, 900, 950 °C to 1000, 1050, 1100 °C. In certain aspects the method is performed at a temperature of about 925 °C.

[0009] The reducing gas comprising CO can further comprise H_2 . In certain aspects the reducing gas comprises 50, 60, 70 vol.% H_2 and 30, 40, 50 vol.% CO. The reducing gas can

be introduced at a flow rate of about or at least 500, 550, 600, 700, 800, 900, or 1000 ml/min. In certain aspects the reducing gas can be introduced at a flow rate of about 1000 ml/min.

[0010] The solid carbonaceous reductant can be anthracite or similar carbon containing materials. In certain aspects the target mixture can consist essentially of anthracite and iron ore. In certain aspects the unreduced iron material is iron ore. The unreduced iron material can be provided in pellet form. In certain aspects, increasing a weight ratio of anthracite in the target mixture can improve a reduction rate of Fe_2O_3 to Fe. Increasing a weight ratio of anthracite in the target mixture can also improve a gasification rate of the solid carbonaceous reductant, where the gasification produces carbon monoxide and further improves reduction rate of Fe_2O_3 to Fe. Furthermore, the increase of anthracite (coal) in the target mixture is capable of increasing hydrogen (H_2) concentration in the reducing gas in a substantially isothermal reduction of Fe_2O_3 to Fe. In certain aspects, the anthracite in the solid carbonaceous reductant may be capable of reducing a reduction time for Fe_2O_3 to Fe. The anthracite can also reduce clustering of the target mixture as a separator between pellets.

[0011] Other embodiments of the invention are discussed throughout this application. Any embodiment discussed with respect to one aspect of the invention applies to other aspects of the invention as well and vice versa. Each embodiment described herein is understood to be embodiments of the invention that are applicable to all aspects of the invention. It is contemplated that any embodiment discussed herein can be implemented with respect to any method or composition of the invention, and vice versa. Furthermore, compositions and kits of the invention can be used to achieve methods of the invention.

[0012] The use of the word “a” or “an” when used in conjunction with the term “comprising” in the claims and/or the specification may mean “one,” but it is also consistent with the meaning of “one or more,” “at least one,” and “one or more than one.”

[0013] Throughout this application, the term “about” is used to indicate that a value includes the standard deviation of error for the device or method being employed to determine the value.

[0014] The use of the term “or” in the claims is used to mean “and/or” unless explicitly indicated to refer to alternatives only or the alternatives are mutually exclusive, although the disclosure supports a definition that refers to only alternatives and “and/or.”

[0015] As used in this specification and claim(s), the words “comprising” (and any form of comprising, such as “comprise” and “comprises”), “having” (and any form of having, such as “have” and “has”), “including” (and any form of including, such as “includes” and “include”) or “containing” (and any form of containing, such as “contains” and “contain”) are inclusive or open-ended and do not exclude additional, unrecited elements or method steps.

[0016] In the context of the present invention, 16 embodiments are now described. Embodiment 1 is a method of producing direct reduced iron (DRI), the method comprising contacting a target mixture comprising at least 5 wt.% of a solid carbonaceous reductant and at least 85 wt.% of an unreduced iron material (Fe_2O_3) with a reducing gas comprising carbon monoxide (CO) under conditions sufficient to produce reduced iron (Fe). Embodiment 2 is the method of embodiment 1, wherein the target mixture is contacted with the reducing gas at a temperature of 800 °C to 1100 °C, preferably 925 °C. Embodiment 3 is the method of any of embodiments 1 or 2, wherein the reducing gas further comprises H_2 . Embodiment 4 is the method of any of embodiments 1, 2 or 3 wherein the reducing gas comprises 60 vol.% H_2 and 40 vol.% CO. Embodiment 5 is the method of any of embodiments 1 to 4, wherein the solid carbonaceous reductant is anthracite. Embodiment 6 is the method of any of embodiments 1 to 5, wherein the target mixture consists essentially of anthracite and iron ore. Embodiment 7 is the method of any of embodiments 5 and 6, wherein increase of a weight ratio of anthracite in the target mixture improves a reduction rate (RDR) of Fe_2O_3 and a gasification rate of the solid carbonaceous reductant, said gasification produces carbon monoxide and further improves reduction rate of Fe_2O_3 . Embodiment 8 is the method of any of embodiments 5 and 6, wherein increase of anthracite in the target mixture is capable of increasing hydrogen (H_2) concentration in the reducing gas in a substantially isothermal reduction of Fe_2O_3 . Embodiment 9 is the method of any of embodiments 5 and 6, wherein the anthracite in the target mixture is capable of reducing clustering in the target mixture. Embodiment 10 is the method of any of embodiments 5 and 6, wherein the anthracite in the target mixture is capable of reducing a reduction time for Fe_2O_3 . Embodiment 11 is the method of any of embodiments 1, 2, 5 and 6, further comprising introducing the reducing gas into a DRI reactor containing the target mixture.

[0017] Embodiment 12 is a method of producing direct reduced iron (DRI), the method comprising contacting a mixture comprising 5 wt.% to 20 wt.% of anthracite and 80 to 95 wt.% an unreduced iron material (Fe_2O_3) with a reducing gas comprising carbon monoxide

(CO) at 850 °C to 1100 °C to produce reduced iron (Fe). Embodiment 13 is the method of embodiment 12, wherein the target mixture is contacted with the reducing gas at a temperature of 800 °C to 1100 °C. Embodiment 14 is the method of any of embodiments 12 and 13, wherein the reducing gas comprises 60 vol.% H₂ and 40 vol.% CO. Embodiment 15 is the method of any of embodiments 12 and 13, wherein the target mixture consists essentially of anthracite and iron ore. Embodiment 16 is the method of any of embodiments 12 and 13, wherein reducing gas is introduced at a flow rate of 500 to 1000 ml/min.

[0018] Other objects, features and advantages of the present invention will become apparent from the following detailed description. It should be understood, however, that the detailed description and the specific examples, while indicating specific embodiments of the invention, are given by way of illustration only, since various changes and modifications within the spirit and scope of the invention will become apparent to those skilled in the art from this detailed description.

DESCRIPTION OF THE FIGURES

[0019] The following drawings form part of the present specification and are included to further demonstrate certain aspects of the present invention. The invention may be better understood by reference to one or more of these drawings in combination with the detailed description of the specification embodiments presented herein.

[0020] FIG. 1. Schematic of one embodiment of a direct reduction system.

[0021] FIG. 2. Shows an analysis of iron ore pellets used in certain embodiments.

[0022] FIG. 3. Shows an analysis of anthracite used in certain embodiments.

[0023] FIG. 4. Shows representative reduction rate data.

[0024] FIG. 5. Shows representative gasification rate data.

[0025] FIG. 6. Shows gas composition of pellet in 10% coal isothermal reduction reaction.

[0026] FIG. 7. Shows representative reduction rate and gasification data for an isothermal reduction reaction.

[0027] FIG. 8. Shows data relating to coal char formation from heating anthracite.

DESCRIPTION

[0028] The present invention includes a process for producing direct reduced iron pellets. The direct reduced iron pellets are prepared by metallizing pellets under reducing conditions at elevated temperatures.

5 **[0029]** The present invention has the ability to utilize a variety of iron oxide-containing materials regardless of gangue, ferric iron, ferrous iron, and total iron content. Iron ore, such as is obtained from taconite, may be utilized in the iron oxide-containing material. The iron oxide-containing material may also include reverts that are used alone or in conjunction with iron ore. Reverts that are particularly suitable for use with the present invention include a
10 variety of furnace dusts and sludges, such as basic oxygen furnace dust, blast furnace dust, and millscale. Other similar mineral wastes, such as iron oxide waste produced from illminite processing, may also be used in producing direct reduced iron pellets according to the present invention. The iron oxide-containing material is capable of being formed from any blend of iron ore and reverts ranging from pure iron ore to pure reverts.

15 **[0030]** When iron ore is used in formulating the iron oxide-containing material, the iron content in the iron ore can be, but need not be, enriched using conventionally known comminution and beneficiation processes. These processes preferably include ball-mill pulverizing the iron ore to minus 325 mesh. As used herein all references to mesh refer to U.S. mesh classification set forth in ASTM E-11-61.

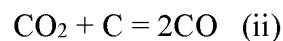
20 **[0031]** The internal fixed carbon content is the fixed carbon content internal to the pellet before firing. Internal fixed carbon content is distinguished from residual carbon, which refers to the fixed carbon content internal to the pellet after firing. Internal fixed carbon content is also distinguished from external carbon, which is distinct from the pellet.

[0032] Insufficient internal fixed carbon content can lead to direct reduced iron pellets
25 with low metallization. Higher internal fixed carbon content can result in direct reduced iron pellets with a greater degree of metallization as well as a higher level of residual carbon. However, excessive internal fixed carbon content causes the direct reduced iron pellets to exhibit unacceptably low fired compressive strengths and causes unacceptable pellet degradation. Preferably, the internal fixed carbon content is between about 5 and 20 wt.%,
30 and all values and ranges there between, including 5 to 8 wt.%, 8 to 11 wt.%, 11 to 14 wt.%,

14 to 17 wt.%, and 17 to 20 wt.%. Unless referred to otherwise, all references to percent in this application refer to percent by weight.

[0033] The internal fixed carbon content may also be adjusted by formulating the pellets with reverts, such as blast furnace dust and blast furnace sludge, that contain significant levels of carbon. Additionally, carbonaceous materials, such as petroleum coke and carbon-bearing, battery waste, can be used in producing pellets with a desired internal fixed carbon content.

[0034] Increase of a weight ratio of the internal carbon content (*e.g.* anthracite content) in the target mixture improves a reduction rate (RDR) of Fe_2O_3 and a gasification rate of the solid carbonaceous reductant (internal carbon) during the process of reducing Fe_2O_3 . The gasification can produce carbon monoxide and further improve reduction rate of Fe_2O_3 . The main reactions of gasification herein can include:



where CO and H_2 are included in the reducing gas for producing direct reduced iron from unreduced iron material (Fe_2O_3); water (H_2O) and CO_2 are formed by reacting H_2 and CO with Fe_2O_3 , respectively. Thus, the increase of C (*e.g.* anthracite) in the target mixture can increase hydrogen (H_2) concentration in the reducing gas in a isothermal reduction process of Fe_2O_3 to Fe. Overall, increase of carbon content (such as anthracite content) can reduce the reduction time for Fe_2O_3 . Furthermore, functioning as a separator among the pellets, anthracite in the solid carbonaceous reductant is capable of reducing clustering in the target mixture.

[0035] The agglomerate of iron oxide-containing materials can be formed into pellets using either extrusion or other conventionally known pelletizing techniques. When extrusion is used to form the pellets, the extruder is preferably selected with a diameter of less than about 1 or 2 centimeters. Pellets with this nominal size allow the firing process to be completed within a commercially feasible time period.

[0036] A variety of furnace configurations may be used to fire the pellets and thereby convert the pellets into direct reduced iron pellets. Factors affecting the firing temperature

include refractory type, fuel type, external reductant type, pellets degradation (dusting), and low melting point phases such as wustite and fayalite (ferrous oxide and iron silicate). For most compositions firing the pellets at temperatures of between about 800 °C and 1,000 °C produces desirable metallization rates. Preferably, firing is performed at a temperature of approximately 925 °C. When a firing temperature of about 925 °C is used, the pellets can be fired for approximately 1 hour.

EXAMPLES

[0037] The following examples as well as the figures are included to demonstrate preferred embodiments of the invention. It should be appreciated by those of skill in the art that the techniques disclosed in the examples or figures represent techniques discovered by the inventors to function well in the practice of the invention, and thus can be considered to constitute preferred modes for its practice. However, those of skill in the art should, in light of the present disclosure, appreciate that many changes can be made in the specific embodiments which are disclosed and still obtain a like or similar result without departing from the spirit and scope of the invention.

EXAMPLE 1

[0038] Reduction based on Quadruple mass spectroscopy QMS measurements for iron ore pellets or iron containing pellets (“iron pellets”) with anthracite in absence/presence of H₂/CO gas mixture was determined. Iron/carbon (Fe₂O₃/C) mixtures were prepared having 0, 5, 10, and 15 wt% C. Iron pellets were formed having a pellet size or average pellet size of about or at least 10.0 to 12.5 mm (FIG. 2). Anthracite grains were used having a grain size or average grain size of 5 to 10 mm (FIG. 3). The components were exposed to reduction temperatures of 850 and 1000 °C. A reducing gas was contacted with the iron/anthracite components, the reducing gas having a H₂/CO volumetric ratio of 0 and 60/40. In certain instances the gas flow rate remained constant. The sample size of the iron pellets was 100 g. A schematic of the experimental set up is provided in FIG. 1.

[0039] *Optimum flow rate to remove gas boundary diffusion.* Reduction rate shows significant difference at 1000 ml/min compare to other flow rate (FIG. 4). Gasification rate shows that change of flow rate does not significantly affect the gasification (FIG. 5).

[0040] *Reduction of iron ore Pellet mixed with 10% Coal using H₂/CO gas mixture at 1000 °C.* Increase of CO₂ concentration at beginning of the reduction shows that reduction proceeds at a high rate - reduction from Fe₂O₃ → Fe₃O₄ → FeO. Further reduction from FeO to M-Fe proceeds at a lower rate as shown by a gradual decrease of CO₂ concentration. The QMS result shows clear increase in H₂ gas during isothermal reduction of Fe₂O₃ (FIG. 6). The reduction rate (RDR) and gasification rate (RCS) show a high value in the beginning of reduction and gradually decrease with time. The curves are similar in shape, which means that both reduction and gasification have mutual behavior (FIG. 7).

[0041] *Non-isothermal gasification of coal char in CO₂ by TG-DSC.* Coal char produced from heating (5 °C/min) anthracite coal in Ar atmosphere at 1000 °C shows higher reactivity start at ~927°C (FIG. 8).

CLAIMS

1. A method of producing direct reduced iron (DRI), the method comprising contacting a target mixture comprising at least 5 wt.% of a solid carbonaceous reductant and at least 85 wt.% of an unreduced iron material (Fe_2O_3) with a reducing gas comprising carbon monoxide (CO) under conditions sufficient to produce reduced iron (Fe).
2. The method of claim 1, wherein the target mixture is contacted with the reducing gas at a temperature of 800 °C to 1100 °C, preferably 925 °C.
3. The method of any of claims 1 and 2, wherein the reducing gas further comprises H_2 .
4. The method of any of claims 1 and 2, wherein the reducing gas comprises 60 vol.% H_2 and 40 vol.% CO.
5. The method of claim 1, wherein the solid carbonaceous reductant is anthracite.
6. The method of claim 1, wherein the target mixture consists essentially of anthracite and iron ore.
7. The method of any of claims 5 and 6, wherein increase of a weight ratio of anthracite in the target mixture improves a reduction rate (RDR) of Fe_2O_3 and a gasification rate of the solid carbonaceous reductant, said gasification produces carbon monoxide and further improves reduction rate of Fe_2O_3 .
8. The method of any of claims 5 and 6, wherein increase of anthracite in the target mixture is capable of increasing hydrogen (H_2) concentration in the reducing gas in a substantially isothermal reduction of Fe_2O_3 .
9. The method of any of claims 5 and 6, wherein the anthracite in the target mixture is capable of reducing clustering in the target mixture.
10. The method of any of claims 5 and 6, wherein the anthracite in the target mixture is capable of reducing a reduction time for Fe_2O_3 .
11. The method of any of claims 1,2, 5 and 6, further comprising introducing the reducing gas into a DRI reactor containing the target mixture.

12. A method of producing direct reduced iron (DRI), the method comprising contacting a mixture comprising 5 wt.% to 20 wt.% of anthracite and 80 to 95 wt.% an unreduced iron material (Fe_2O_3) with a reducing gas comprising carbon monoxide (CO) at 850 °C to 1100 °C to produce reduced iron (Fe).
13. The method of claim 12, wherein the target mixture is contacted with the reducing gas at a temperature of 800 °C to 1100 °C.
14. The method of any of claims 12 and 13, wherein the reducing gas comprises 60 vol.% H_2 and 40 vol.% CO.
15. The method of any of claims 12 and 13, wherein the target mixture consists essentially of anthracite and iron ore.
16. The method of any of claims 12 and 13, wherein reducing gas is introduced at a flow rate of 500 to 1000 ml/min.

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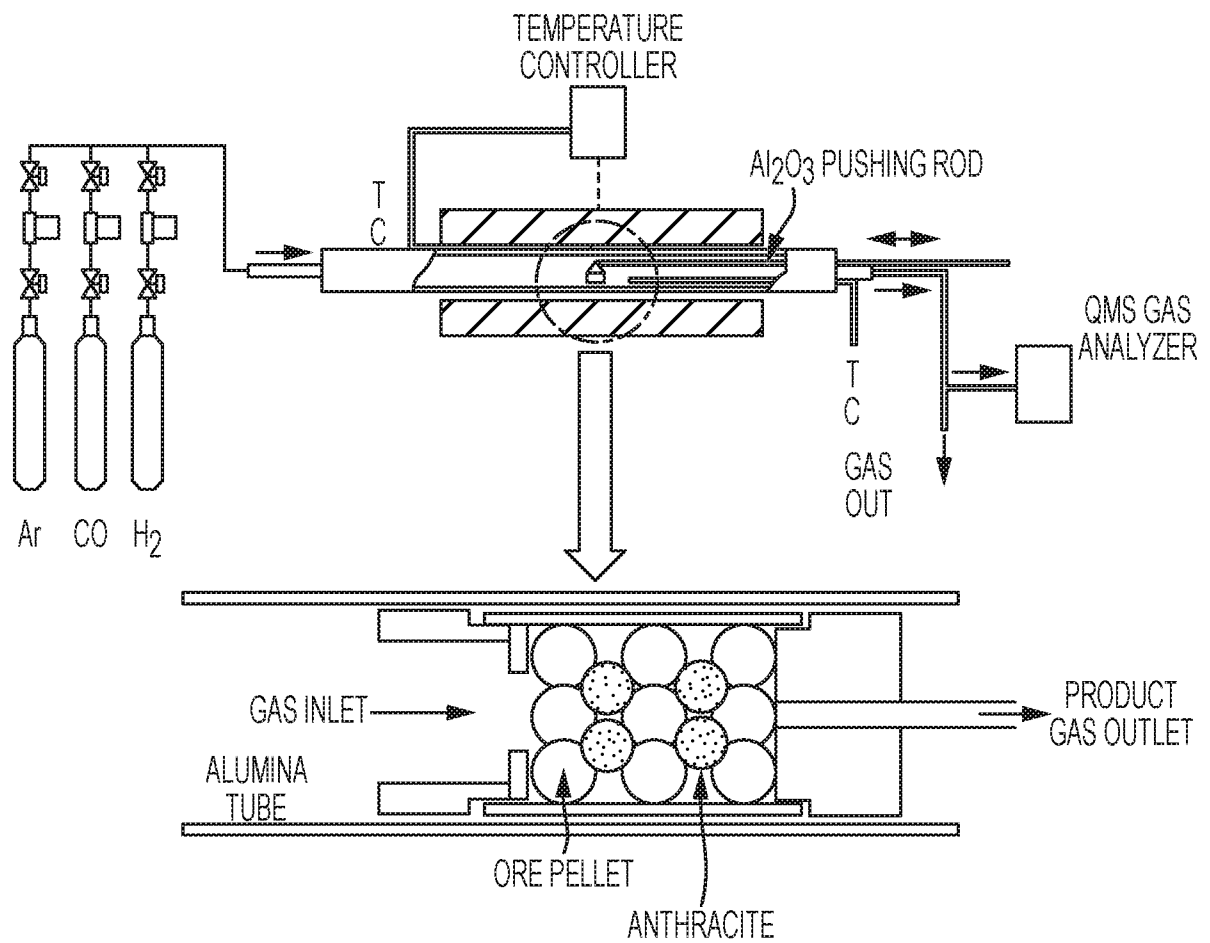


FIG. 1

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CHEMICAL COMPOSITION OF PELLET (wt%)							
T-Fe	Fe ₂ O ₃	FeO	SiO ₂	Al ₂ O ₃	CaO	K	P
68.260	96.279	1.182	1.310	0.368	0.712	0.008	0.039

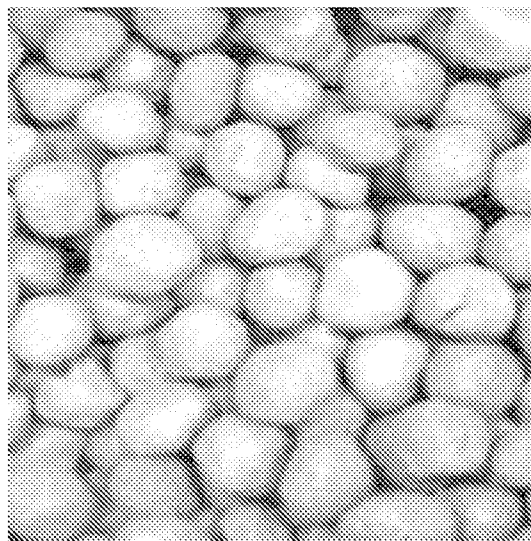
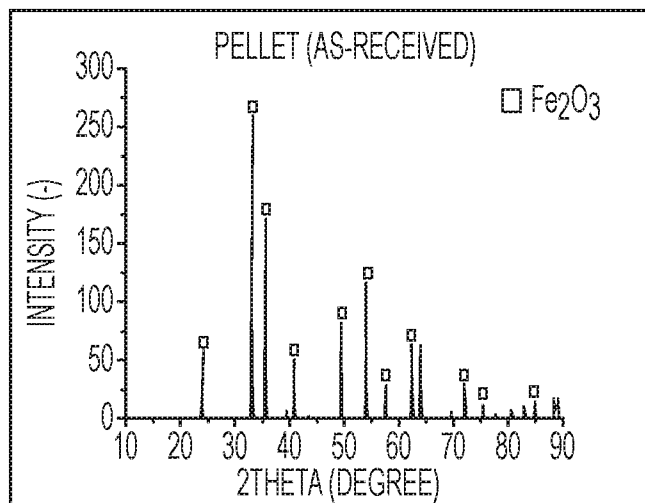


FIG. 2

ANALYSIS OF ANTHRACITE:

PROXIMATE ANALYSIS OF ANTHRACITE (wt%)			
F.C.	V.M.	ASH	I.M.
81.59	5.70	7.24	5.47

ULTIMATE ANALYSIS OF ANTHRACITE (wt%)				
C	H	N	O	S
95.13	0.84	0.39	3.32	0.32

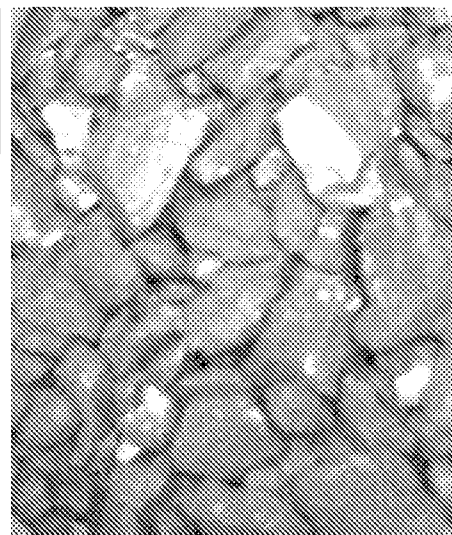


FIG. 3

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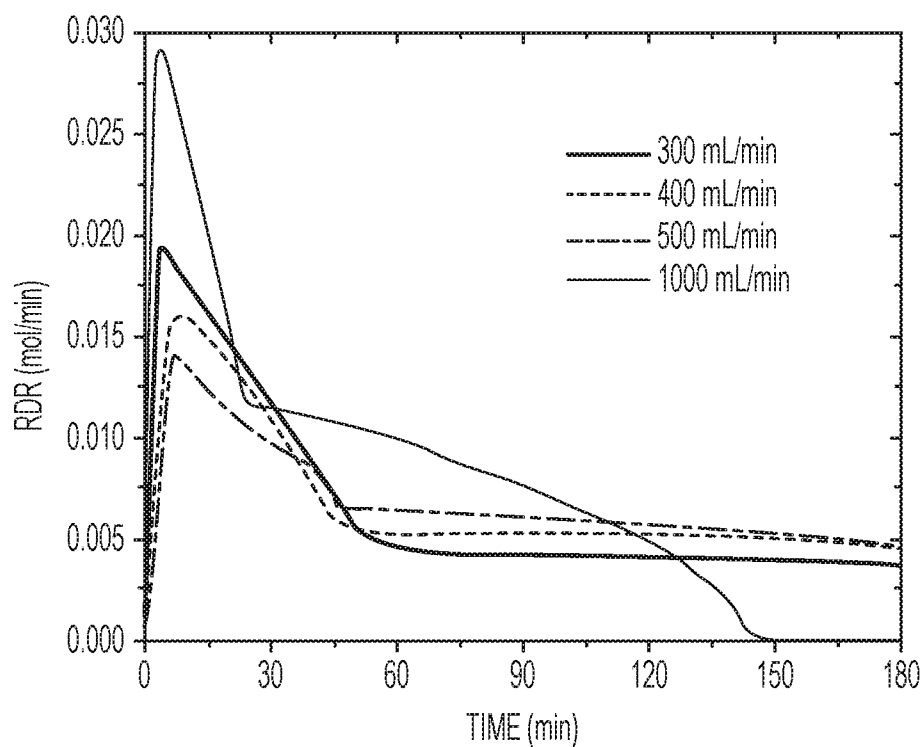


FIG. 4

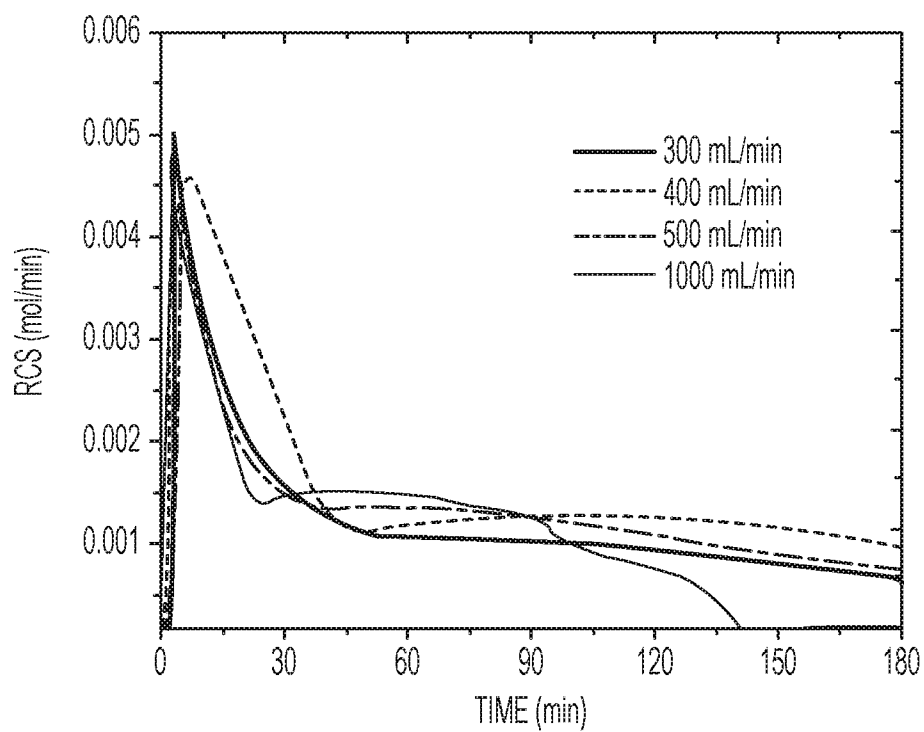


FIG. 5

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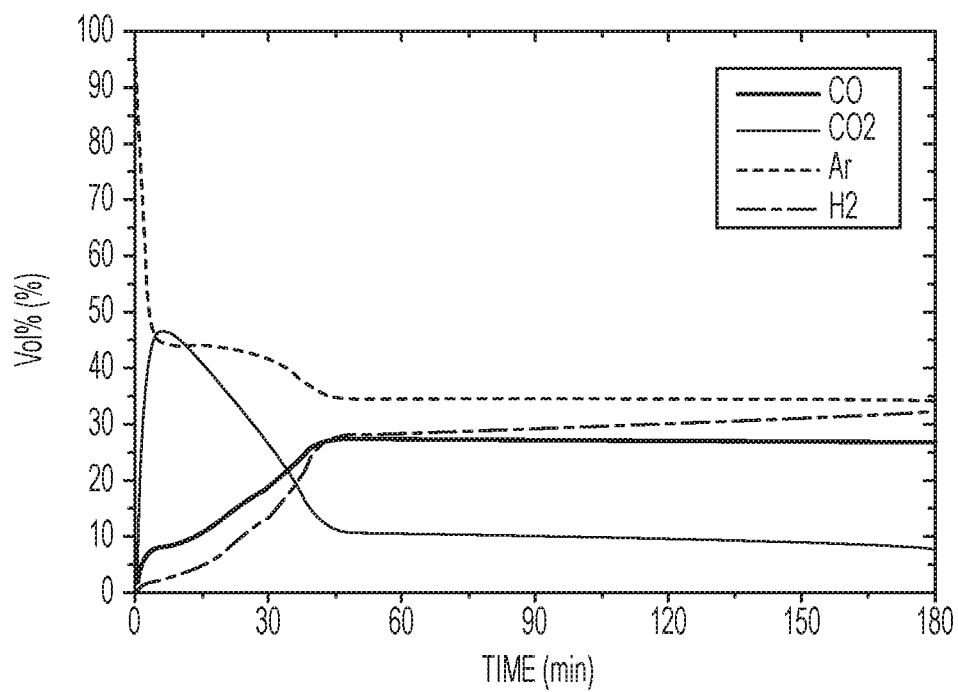


FIG. 6

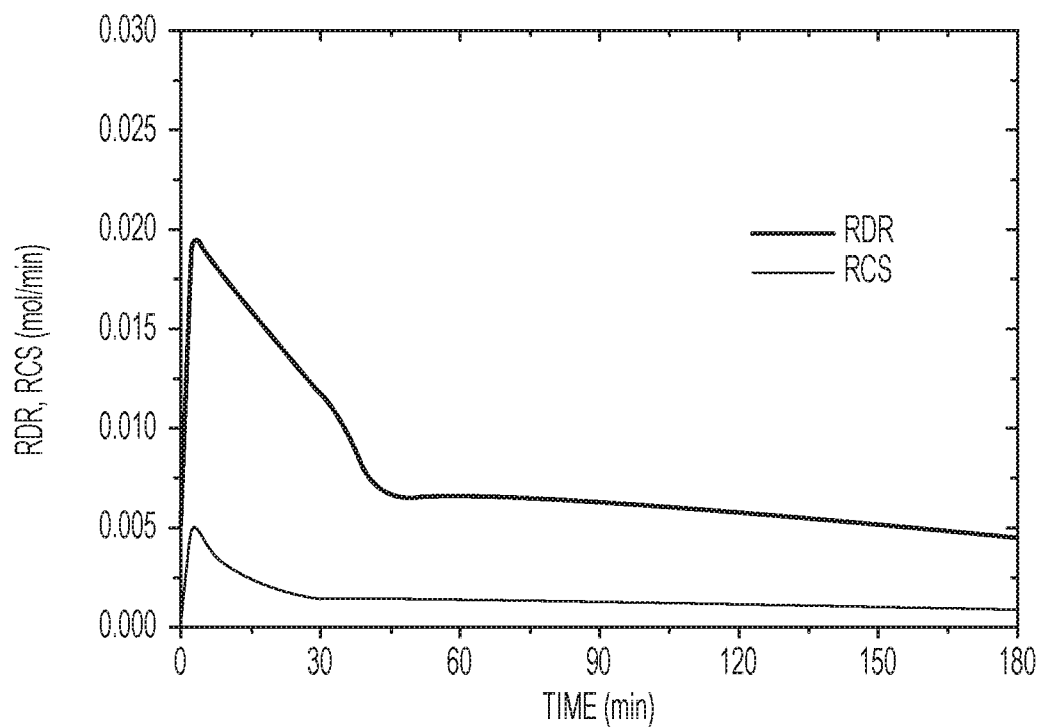


FIG. 7

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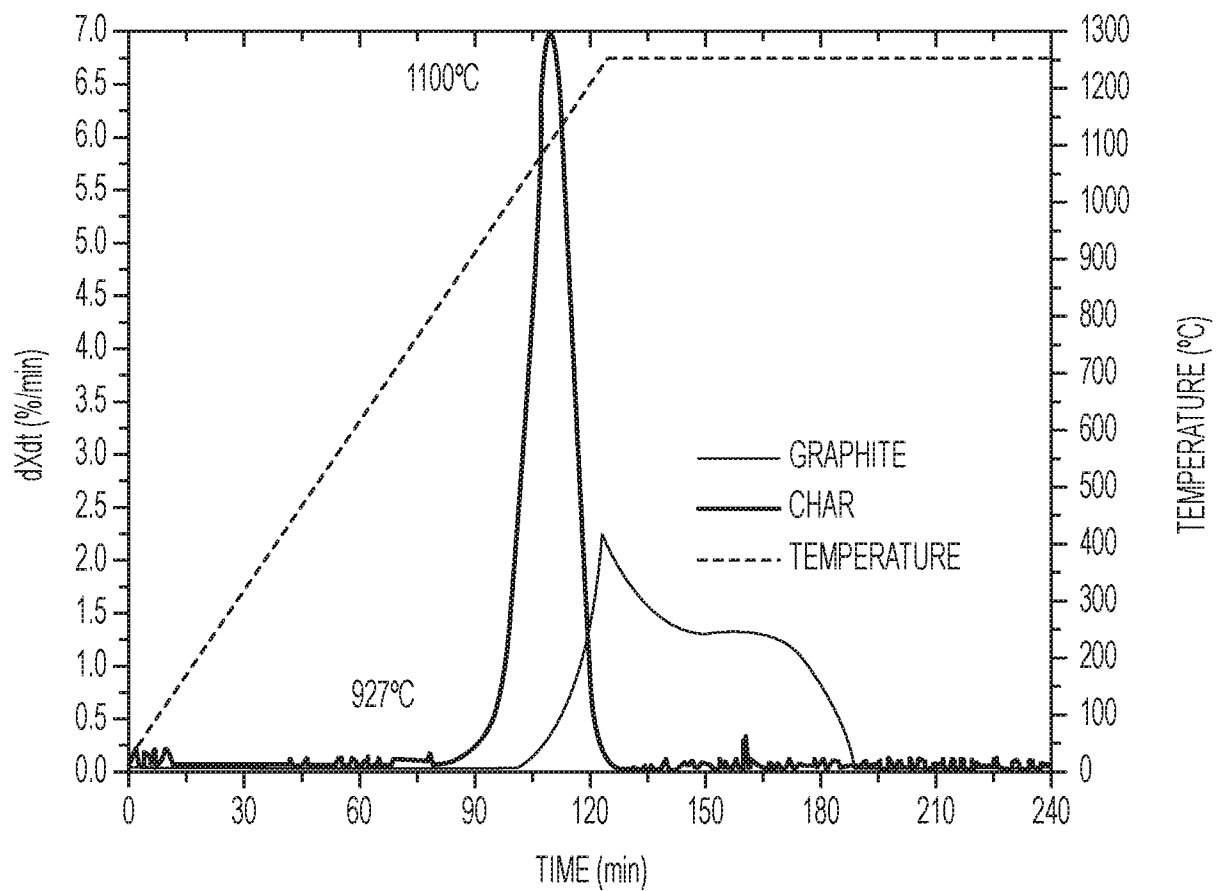


FIG. 8

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2017/056291

A. CLASSIFICATION OF SUBJECT MATTER
INV. C21B13/00 C21B13/02
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C21B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2015/114546 A1 (SAUDI BASIC IND CORP [SA]) 6 August 2015 (2015-08-06) paragraphs [0002], [0003], [0030], [0031], [0038], [0043], [0050], [0056], [0068], [0075] ----- -/--	1-16



Further documents are listed in the continuation of Box C.



See patent family annex.

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Date of the actual completion of the international search

23 January 2018

Date of mailing of the international search report

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INTERNATIONAL SEARCH REPORT

International application No
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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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