



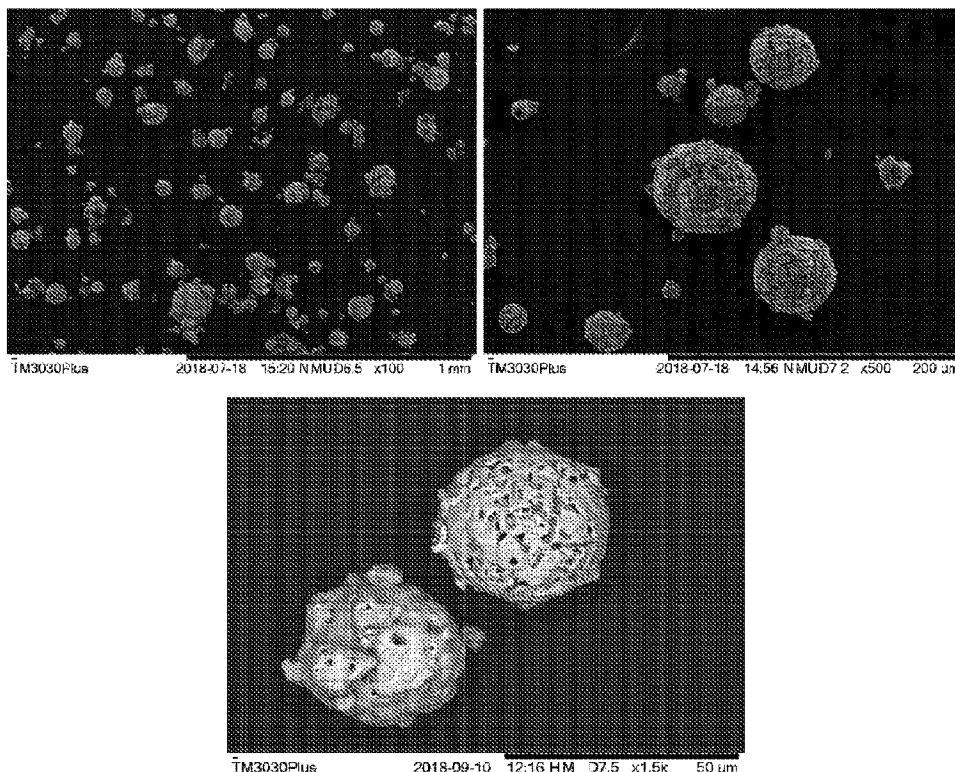
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(57) Abrégé/Abstract:

The present disclosure relates to a simple, economical, non-toxic and efficient method of producing spherical iron powder from iron oxide. The method comprises reacting iron oxide with a reducing agent to produce irregular iron powder, burning the obtained irregular iron powder in presence of oxygen to produce spherical iron oxide particles, and thereafter reacting the spherical iron oxide with a reducing agent to obtain the final spherical iron powder. The obtained spherical iron powder is characterized by a spherical morphology, high purity, finer particle size range and a porous structure across the surface of iron powder.

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(54) Title: A METHOD OF PRODUCING SPHERICAL IRON POWDER AND PRODUCTS THEREOF

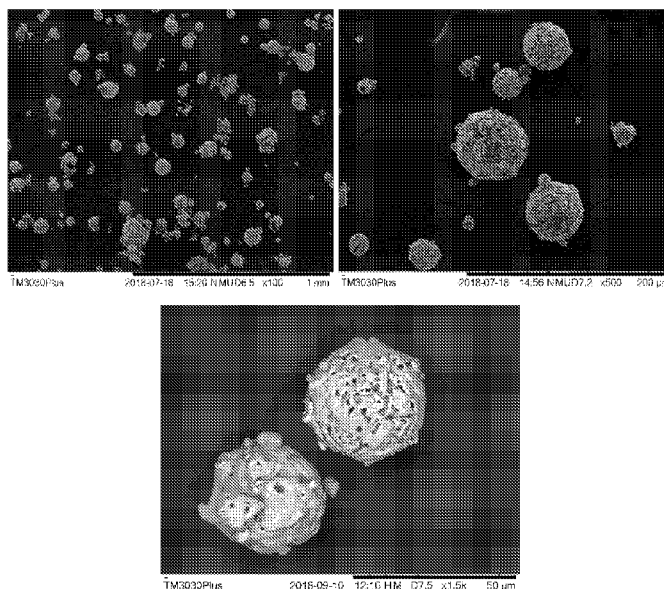


Figure 7

(57) Abstract: The present disclosure relates to a simple, economical, non-toxic and efficient method of producing spherical iron powder from iron oxide. The method comprises reacting iron oxide with a reducing agent to produce irregular iron powder, burning the obtained irregular iron powder in presence of oxygen to produce spherical iron oxide particles, and thereafter reacting the spherical iron oxide with a reducing agent to obtain the final spherical iron powder. The obtained spherical iron powder is characterized by a spherical morphology, high purity, finer particle size range and a porous structure across the surface of iron powder.

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“A METHOD OF PRODUCING SPHERICAL IRON POWDER AND PRODUCTS THEREOF”

TECHNICAL FIELD

5 The present disclosure is in the field of metallurgy, more particularly towards spherical iron powder production. The present disclosure provides a simple, economical, non-toxic and efficient method of producing spherical iron powder from iron oxide.

BACKGROUND OF THE DISCLOSURE

10 Aggregates of iron with a size range of about 20-200 μm are termed as iron powders. Metal powder manufacturing techniques are broadly classified into three methods: chemical (reduction/decomposition), physical (atomization/electrolytic) and mechanical (comminution). Iron powders on a large scale are usually manufactured by reduction (solid/gas) and atomization (air/water/gas) techniques. Reduction includes reducing iron oxide to iron powder
15 using solid or gaseous reducing agents. On the other hand, in the atomization technique, liquid iron melts are atomized by air or water or gas and further processed to get powdered form of iron. The selection of the method of production is always dependent on the type of application and desired property of the iron powder to be produced.

20 Iron powder prepared through reduction techniques and water atomization techniques are irregular in shape. Further, though iron powder produced via inert gas atomization is spherical in shape, said inert gas atomization is very expensive and includes a major limitation on the powder size that can be produced through atomization. For instance, fine powders cannot be produced through this route. Additionally, carbonyl processes can produce spherical iron
25 powders with fine particle size ranges, but said processes suffer from several disadvantages such as very high toxicity due to hazardous gases and the presence of unwanted impurities such as carbon and nitrogen in the iron powder product. Therefore, there is an immense need to develop a simple, cost-effective and non-toxic route/method to produce pure carbon-free spherical iron powders in the finer particle size ranges.

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STATEMENT OF THE DISCLOSURE

The present disclosure relates to a method of preparing spherical iron powder, comprising:

reducing iron oxide in presence of a carbon monoxide rich gas or a hydrogen rich gas or a solid carbonaceous reducing agent or any combination thereof, to obtain irregular iron powder,

burning the irregular iron powder in presence of oxygen rich gas, to obtain spherical iron oxide,

reducing the spherical iron oxide in presence of a carbon monoxide rich gas or a hydrogen rich gas or a combination thereof, to obtain the spherical iron powder.

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The present disclosure further relates to spherical iron oxide obtained by the method described above wherein said spherical iron oxide comprises a dense spherical morphology of iron oxide particles having an average particle diameter between 10 μm to 200 μm and an apparent density between 2.5 g/cc to 3 g/cc.

10

The present disclosure also provides spherical iron powder obtained by the method described above wherein said spherical iron powder has at least 98% metallic iron [Fe(m)] and comprises a dense spherical morphology of iron particles with an average particle diameter between 5 μm and 100 μm and an apparent density of 2.1 g/cc to 2.6 g/cc.

15

BRIEF DESCRIPTION OF THE ACCOMPANYING FIGURES

Figure 1 depicts X-ray powder diffraction (XRD) analysis of spray roasted iron oxide powder (raw material) employed in the present method of producing spherical iron oxide.

Figure 2 depicts XRD analysis of irregular iron powder obtained by reduction of spray roasted iron oxide during the present method of producing spherical iron oxide.

Figure 3 depicts microstructure of irregular iron powder obtained by reduction of spray roasted iron oxide during the present method of producing spherical iron oxide.

Figure 4 depicts XRD analysis of spherical iron oxide powder obtained after burning of irregular iron powder in presence of air.

Figure 5 depicts microstructure of spherical iron oxide powder obtained after burning of irregular iron powder in presence of air.

Figure 6 depicts XRD analysis of spherical iron powder obtained by reacting spherical iron oxide with hydrogen rich gas.

Figure 7 depicts microstructure of spherical iron powder obtained by reacting spherical iron oxide with hydrogen rich gas.

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DETAILED DESCRIPTION OF THE DISCLOSURE

With respect to the use of substantially any plural and/or singular terms herein, those having skill in the art can translate from the plural to the singular and/or from the singular to the plural as is appropriate to the context and/or application. The various singular/plural permutations may be expressly set forth herein for sake of clarity. The use of the expression “at least” or “at least one” suggests the use of one or more elements or ingredients or quantities, as the use may be in the embodiment of the disclosure to achieve one or more of the desired objects or results. Throughout this specification, the word “comprise”, or variations such as “comprises” or “comprising” or “containing” or “has” or “having” wherever used, will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

As used herein, the phrases ‘spherical iron’ or ‘spherical iron powder’ refer to high purity iron particles comprising a dense spherical morphology and a homogenous particle size along with a porous structure across the surface of iron powder, and having an average particle diameter between 5 μm and 100 μm with an apparent density of not more than 2.6 g/cc (gram per cubic centimeter).

As used herein, the phrases ‘spherical iron oxide’ or ‘spherical iron oxide powder’ refer to iron oxide particles comprising dense spherical morphology and a homogenous particle size, and having an average particle diameter between 10 μm and 200 μm with an apparent density of not more than 3 g/cc.

As used herein, the phrases ‘irregular iron’ or ‘irregular iron powder’ refer to iron particles characterized by irregular shape and inhomogeneity in particle size, and having an apparent density of 0.8-2.4 g/cc.

As used herein, the phrases ‘irregular iron oxide’ or ‘irregular iron oxide powder’ or ‘iron oxide powder (raw material)’ or ‘iron oxide (raw material)’ refer to iron oxide particles characterized by irregular shape and inhomogeneity in particle size.

The present disclosure is in relation to spherical iron powder production. An object of the present disclosure is to develop a simple, economical and non-toxic method of producing spherical iron powders in the finer particle size ranges.

Accordingly, the present disclosure provides a method of producing spherical iron powder, comprising steps of:

reducing iron oxide (raw material) in presence of a carbon monoxide rich gas or a hydrogen rich gas or a solid carbonaceous reducing agent or any combination thereof, to obtain irregular
5 iron powder,

burning the irregular iron powder in presence of oxygen rich gas, to obtain spherical iron oxide,

reducing the spherical iron oxide in presence of a carbon monoxide rich gas or a hydrogen rich gas or a combination thereof, to obtain the spherical iron powder.

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In an embodiment, the iron oxide (raw material) employed for reduction is spray roasted iron oxide powder which is an oxide form of iron, and said spray roasted iron oxide powder is obtained from spray roasting process during acid regeneration process of spent pickle liquor.

15 In an exemplary embodiment of the present disclosure, the spray roasted iron oxide powder comprises Fe(t) at a wt% of about 69.09, FeO at a wt% of about 0.26, SiO₂ at a wt% of about 0.08, CaO at a wt% of about 0.01, MgO at a wt% of about 0.027, MnO at a wt% of about 0.38, Al₂O₃ at a wt% of about 0.16, S at a wt% of about 0.01, C at a wt% of about 0.123. Said spray roasted iron oxide powder further comprises at least one or more additional
20 elements/components selected from Cr, Ti, Cu and P at various wt% to make up the final composition to 100 wt%.

In an embodiment of the present method, the carbon monoxide rich gas is selected from a group comprising coke oven gas, blast furnace gas, steel making converter gas, pure carbon monoxide
25 and combinations thereof.

In another embodiment of the present method, the hydrogen rich gas is selected from a group comprising cracked ammonia, hydrogen separated from coke oven gas, pure hydrogen and combinations thereof.

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In yet another embodiment of the present method, the solid carbonaceous reducing agent is selected from a group comprising carbon black, anthracite coal, coal tar pitch, coke breeze and combinations thereof.

In still another embodiment of the present method, the oxygen rich gas is selected from a group comprising pure oxygen, air, a mixture of oxygen and fuel gas, and combinations thereof. In an embodiment, the oxygen and fuel gas mixture is selected from a group comprising oxygen and acetylene, oxygen and propane, oxygen and butene, and combinations thereof.

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In an embodiment of the present method, the reduction of the iron oxide (raw material) to obtain irregular iron powder is carried out in a reduction furnace at a temperature of about 500°C to 1200°C and for a time-period of about 60 minutes to 360 minutes.

10 In exemplary embodiments of the present method, the reduction of iron oxide to irregular iron powder is performed at a temperature of about 500°C, about 600°C, about 700°C, about 800°C, about 900°C, about 1000°C, about 1100°C or about 1200°C. In preferred embodiments, the reduction of iron oxide to irregular iron powder is performed at a temperature of about 700°C, about 800°C, about 900°C or about 1000°C.

15

In another exemplary embodiment of the present method, the reduction of iron oxide to irregular iron powder is carried out for a time-period of about 60 minutes, about 80 minutes, about 100 minutes, about 120 minutes, about 140 minutes, about 160 minutes, about 180 minutes, about 200 minutes, about 220 minutes, about 240 minutes, about 260 minutes, about
20 280 minutes, about 300 minutes, about 320 minutes, about 340 minutes or about 360 minutes. In preferred embodiments, the reduction of iron oxide to irregular iron powder is carried out for a time-period of about 60 minutes, about 180 minutes, about 240 minutes, about 300 minutes or about 360 minutes.

25 In an embodiment of the present disclosure, the irregular iron powder obtained during the present method has an apparent density between 0.8 g/cc to 2.4 g/cc. In exemplary embodiments, the irregular iron powder possesses an apparent density of about 0.8 g/cc, about 0.9 g/cc, about 1 g/cc, about 1.6 g/cc, about 1.7 g/cc, about 1.8 g/cc, or about 2.2 g/cc.

30 In an embodiment of the present method, the burning of the irregular iron powder in presence of oxygen rich gas to obtain spherical iron oxide is carried out using a powder dispensing unit and gas-powder burning torch. In an exemplary embodiment, the burning of the irregular iron powder generates exothermic heat which melts and converts the irregular iron particles of the irregular iron powder into the spherical iron oxide particles due to surface tension.

In another embodiment of the present method, the reduction of the spherical iron oxide to obtain spherical iron powder is carried out in a reduction furnace at a temperature of about 500°C to 1200°C and for a time-period of about 60 minutes to 300 minutes.

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In exemplary embodiments of the present method, the reduction of spherical iron oxide to spherical iron powder is performed at a temperature of about 500°C, about 600°C, about 700°C, about 800°C, about 900°C, about 1000°C, about 1100°C or about 1200°C. In a preferred embodiment, the reduction of spherical iron oxide to spherical iron powder is performed at a temperature of about 500°C to 900°C. In another preferred embodiment, the reduction of spherical iron oxide to spherical iron powder is performed at a temperature of about 900°C to 1100°C.

In another exemplary embodiment of the present method, the reduction of spherical iron oxide to spherical iron powder is carried out for a time-period of about 60 minutes, about 80 minutes, about 100 minutes, about 120 minutes, about 140 minutes, about 160 minutes, about 180 minutes, about 200 minutes, about 220 minutes or about 240 minutes.

In an embodiment, the present method further involves steps of cooling, crushing, pulverizing and screening to obtain the irregular iron powder or the spherical iron powder.

In another embodiment of the present disclosure, the method steps/processes including: (i) burning of the irregular iron powder in presence of oxygen rich gas to obtain spherical iron oxide, and (ii) further reduction of said spherical iron oxide in presence of a carbon monoxide rich gas and/or a hydrogen rich gas, are critical to obtain the final highly pure spherical iron powder with fine particle size range.

In an exemplary embodiment of the present disclosure, the method for preparing spherical iron powder from iron oxide comprises:

- 30 a) heating the iron oxide (raw material) in a reduction furnace to a temperature of about 500°C to 1200°C and reducing the iron oxide in presence of a carbon monoxide rich gas or a hydrogen rich gas or a solid carbonaceous reducing agent or any combination thereof, for a time-period of about 60 minutes to 360 minutes to form a loosely sintered iron powder cake,

- b) cooling the loosely sintered iron powder cake in the furnace to room temperature under inert atmosphere, followed by crushing, pulverizing and screening said iron powder cake to obtain the irregular iron powder,
- c) burning the irregular iron powder in the presence of oxygen rich gas to generate exothermic heat to melt and convert the irregular iron particles of the irregular iron powder into the spherical iron oxide powder due to surface tension,
- d) heating the spherical iron oxide powder in a reduction furnace to a temperature of about 500°C to 1200°C and reducing the spherical iron oxide powder in presence of a carbon monoxide rich gas or a hydrogen rich gas or a combination thereof, for a time-period of about 60 minutes to 300 minutes to form loosely sintered spherical iron powder cake, and
- e) cooling the loosely sintered spherical iron powder cake in the furnace to room temperature under inert atmosphere, followed by crushing, pulverizing and screening said spherical iron powder cake to obtain the spherical iron powder.

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In an embodiment of the above described method, the step of cooling under inert atmosphere is performed by passing nitrogen gas or argon gas, preferably nitrogen gas.

In another exemplary embodiment of the present disclosure, the method for preparing spherical iron powder from iron oxide comprises the following steps:

20

- a) heating the iron oxide (irregular spray roasted iron oxide powder) in a reduction furnace to the temperature of about 500°C to 1200°C,
- b) reduction (removal of oxygen) of the iron oxide in presence of reducing agent selected from:
 - (i) carbon monoxide rich gas such as coke oven gas or blast furnace gas or steel making converter gas,
 - or
 - (ii) hydrogen rich gas such as cracked ammonia or pure hydrogen or converted coke oven gas (i.e. hydrogen separated from coke oven gas),
 - or
 - (iii) carbonaceous solid reducing agent such as carbon black or coal tar pitch or anthracite coal or coke breeze,

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at a temperature of about 500°C to 1200°C and for about 60 minutes to 360 minutes to reduce the iron oxide to form a loosely sintered iron powder cake of apparent density 0.8-2.4 g/cc,

- 5 c) cooling the loosely sintered iron powder cake in the furnace to room temperature by passing nitrogen gas,
- d) crushing, pulverizing and screening the iron powder cake to obtain irregular iron powder,
- e) burning the irregular iron powder using a powder dispensing unit and gaseous powder burning torch carried by oxygen rich gas (pure oxygen, or a mixture of oxygen and fuel
10 gas) to generate exothermic heat to melt and convert the irregular iron particles into spherical iron oxide particles due to the surface tension, wherein the obtained spherical iron oxide particles have an apparent density of not more than 3 g/cc and an average particle diameter between 10 µm and 200 µm, preferably between 40 µm and 100 µm,
- f) heating the spherical iron oxide in a reduction furnace to a temperature of about 500°C
15 to 1200°C,
- g) reduction (removal of oxygen) of the spherical iron oxide in presence of a reducing agent viz. hydrogen rich gas such as cracked ammonia or pure hydrogen or converted coke oven gas (i.e. hydrogen separated from coke oven gas), at a temperature of about 500°C to 1200°C and for about 60 minutes to 300 minutes to reduce the spherical iron
20 oxide to form loosely sintered spherical iron powder cake,
- h) cooling the loosely sintered spherical iron powder cake in the furnace to room temperature by passing nitrogen gas, and
- i) crushing, pulverizing and screening the spherical iron powder cake to produce spherical iron powder with a chemical purity of at least 98% metallic iron and having an apparent
25 density of not more than 2.6 g/cc and an average particle diameter between 5 µm and 100 µm, preferably between 30 µm and 60 µm.

The present disclosure further relates to products obtained during the spherical iron powder production method described herein, including spherical iron oxide and spherical iron powder.

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The present disclosure thus provides spherical iron oxide powder obtained by the method described herein, wherein said spherical iron oxide comprises a dense spherical morphology of iron oxide particles having an average particle diameter between 10 µm to 200 µm and an apparent density of not more than 3 g/cc. In a preferred embodiment, the spherical iron oxide

possesses an average particle diameter between 40 μm to 100 μm . In another preferred embodiment, the spherical iron oxide possesses an apparent density between 2.5 g/cc to 3 g/cc. In exemplary embodiments, the spherical iron oxide possesses an apparent density of about 2.5 g/cc, or about 2.6 g/cc, or about 2.8 g/cc.

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In an embodiment, the spherical iron oxide obtained by the present method comprises Fe(t) at a wt% of about 70.3 to 71.7, FeO at a wt% of about 18.2 to 23.99, Fe(m) at a wt% of about 1.3 to 1.8, CaO at a wt% of about 0.01 to 0.014, SiO₂ at a wt% of about 0.01 to 0.9, MgO at a wt% of about 0.026 to 0.04, MnO at a wt% of about 0.28 to 0.415, Al₂O₃ at a wt% of about 0.2 to 0.54, C at a wt% of about 0.03 to 0.034, and S at a wt% of about 0.002 to 0.004. Said spherical iron oxide further comprises at least one or more additional elements/components selected from Cr, Ti, Cu and P at various wt% to make up the final composition to 100 wt%.

In an exemplary embodiment, the spherical iron oxide obtained by the present method comprises Fe(t) at a wt% of about 71.7, FeO at a wt% of about 23.99, Fe(m) at a wt% of about 1.8, CaO at a wt% of about 0.01, SiO₂ at a wt% of about 0.09, MgO at a wt% of about 0.026, MnO at a wt% of about 0.36, Al₂O₃ at a wt% of about 0.204, C at a wt% of about 0.033, and S at a wt% of about 0.002.

The present disclosure further provides spherical iron powder obtained by the method described herein, wherein said spherical iron powder has high chemical purity with at least 98% metallic iron and comprises a dense spherical morphology of iron particles with an average particle diameter between 5 μm and 100 μm and an apparent density of not more than 2.6 g/cc. In a preferred embodiment, the spherical iron powder possesses an average particle diameter between 30 μm to 60 μm . In another preferred embodiment, the spherical iron powder possesses an apparent density between 2.1 g/cc to 2.6 g/cc. In exemplary embodiments, the spherical iron powder possesses an apparent density of about 2.3 g/cc, or about 2.25 g/cc, or about 2.1 g/cc.

In an embodiment, the spherical iron powder obtained by the present method has a purity or metallic iron content of about 98% to 98.8 %.

In another embodiment, the spherical iron powder obtained by the present method comprises Fe(t) at a wt% of about 98.04 to 98.9, FeO at a wt% of about 0.04 to 0.32, Fe(m) at a wt% of

about 98 to 98.8, CaO at a wt% of about 0.039 to 0.052, SiO₂ at a wt% of about 0.078 to 0.97, MgO at a wt% of about 0.001 to 0.042, MnO at a wt% of about 0.3 to 0.48, Al₂O₃ at a wt% of about 0.217 to 0.56, C at a wt% of about 0.02 to 0.03, and S at a wt% of about 0.001 to 0.005. Said spherical iron powder further comprises at least one or more additional elements/components selected from Cr, Ti, Cu and P at various wt% to make up the final composition to 100 wt%.

In an exemplary embodiment, the spherical iron powder obtained by the present method comprises Fe(t) at a wt% of about 98.8, FeO at a wt% of about 0.13, Fe(m) at a wt% of about 98.7, CaO at a wt% of about 0.048, SiO₂ at a wt% of about 0.078, MgO at a wt% of about 0.001, MnO at a wt% of about 0.48, Al₂O₃ at a wt% of about 0.32, C at a wt% of about 0.02, and S at a wt% of about 0.001.

In an embodiment, the foregoing descriptive matter is illustrative of the disclosure and not a limitation. While considerable emphasis has been placed herein on the particular features of this disclosure, it will be appreciated that various modifications can be made, and that many changes can be made in the preferred embodiments without departing from the principles of the disclosure. Those skilled in the art will recognize that the embodiments herein can be practiced with modification within the spirit and scope of the embodiments as described herein.

EXAMPLES

EXAMPLE 1: Production of spherical iron powder by employing irregular spray roasted iron oxide powder as a raw material and coke oven gas as a reducing agent

This example demonstrates the reduction of irregular spray roasted iron oxide powder using coke oven gas (carbon monoxide rich gas) as reducing agent, and burning the resultant irregular iron powder to produce spherical iron oxide powder which is further reduced to obtain spherical iron powder.

The chemistry/composition of the raw material (spray roasted iron oxide powder) used in the present experiment is provided in Table 1 below.

Table 1: Chemical composition of spray roasted iron oxide raw material used for reduction

Composition (wt %)	Fe(total) [Fe(t)]	FeO	CaO	SiO ₂	S	MgO	MnO	Al ₂ O ₃	C
Spray Roasted Iron Oxide	69.09	0.26	0.01	0.08	0.01	0.027	0.38	0.16	0.123

100 g of iron oxide obtained from spray roasting of pickle liquor was placed in the reduction chamber of furnace and heated at a rate of about 10 °C/min, to reach to a temperature of about 900°C. When the desired temperature was reached, coke oven gas was introduced into the furnace with the flow rate of about 2 litres per minute for about 240 minutes. The reduction lasted for about 240 minutes at a temperature of 900°C resulting in the formation of loosely sintered iron powder cake. Thereafter, to eliminate the pyrophoric nature of the iron powder, the loosely sintered iron powder cake was then subsequently allowed to cool in an inert environment by passing nitrogen gas to further lower the oxygen content. The nitrogen cooling was carried out at a flow rate of about 0.5 liters per minute till the temperature of the furnace reached to atmospheric temperature, thus yielding about 70 grams of iron powder cake. The cake was then crushed and pulverized into desired size to produce irregular iron powder having apparent density of 1 g/cm³. The XRD analysis of the raw material (irregular spray roasted iron oxide powder) and the irregular iron powder obtained after reduction are shown in Figure 1 and Figure 2, respectively. Figure 3 shows the microstructure of the irregular iron powder after reducing the spray roasted iron oxide powder.

The obtained irregular iron powder is then subjected to combustion (burned in the presence of air) to generate exothermic heat to melt and convert the irregular iron particles into spherical iron oxide particles due to the surface tension. The resultant iron oxide obtained after burning is spherical in shape. The XRD analysis and microstructure of the spherical iron oxide are shown in Figure 4 and Figure 5, respectively, and the chemistry/composition is shown in Table 2. The average particle diameter of spherical iron oxide is between 10 and 200 µm with an apparent density of not more than 3 g/cc.

Table 2: Chemical composition of spherical iron oxide produced after combustion

Composition (wt %)	Fe (total)	FeO	Fe (metal)	CaO	SiO ₂	C	S	MgO	MnO	Al ₂ O ₃
Spherical Iron Oxide	71.7	23.99	1.8	0.01	0.09	0.033	0.002	0.026	0.36	0.204

The spherical iron oxide is then reduced in hydrogen rich gas (pure hydrogen) in a reduction furnace, to the temperature of about 500°C to 900°C for about 60 to 240 minutes duration. The resultant loosely sintered spherical iron powder cake is cooled in the furnace to room temperature by passing nitrogen gas. The sintered spherical iron powder cake is then crushed, pulverized and screened to produce spherical iron powder. Figure 6 and Figure 7 shows the XRD analysis and microstructure of spherical iron powder, respectively. Further, the chemistry/composition of the spherical iron powder product is provided in Table 3.

Table 3: Chemical composition of spherical iron powder produced after reduction of spherical iron oxide

Composition (wt %)	Fe (total)	FeO	Fe (metal)	CaO	SiO ₂	C	S	MgO	MnO	Al ₂ O ₃
Spherical Iron powder	98.8	0.32	98.5	0.052	0.12	0.028	0.005	0.042	0.386	0.259

As observed from Table 3, the spherical iron powder contained at least 98.5 wt% metallic iron.

The spherical iron powder had an average particle diameter between 5 and 100 µm with an apparent density of 2.6 g/cc. Said results indicate the achievement of high purity spherical iron powder with finer particle size range by employing the present method.

EXAMPLE 2: Production of spherical iron powder by employing irregular spray roasted iron oxide powder as a raw material and cracked ammonia gas as a reducing agent

Synthesis of spherical iron powder was carried out similar to the procedure described in Example 1. Here, the irregular spray roasted iron oxide powder having the composition as defined in Example 1 was reduced using cracked ammonia gas (hydrogen rich gas) at about 800°C for a reduction time of about 180 minutes to produce irregular iron powder with an apparent density of 0.9 g/cm³. The subsequent steps/conditions of burning to produce spherical iron oxide and further reduction to obtain the final spherical iron powder was performed as described in Example 1.

The average particle diameter of the obtained spherical iron oxide in this example was between 40 μm and 100 μm with an apparent density of 2.6 g/cc. The chemistry/composition of the obtained spherical iron oxide is provided in Table 4.

5

Table 4: Chemical composition of spherical iron oxide produced after combustion

Composition (wt %)	Fe (total)	FeO	Fe (metal)	CaO	SiO ₂	C	S	MgO	MnO	Al ₂ O ₃
Spherical Iron Oxide	71.4	20.2	1.5	0.012	0.01	0.03	0.002	0.03	0.34	0.2

Further, the chemistry/composition of the obtained spherical iron powder in this example is provided in Table 5.

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Table 5: Chemical composition of spherical iron powder produced after reduction of spherical iron oxide

Composition (wt %)	Fe (total)	FeO	Fe (metal)	CaO	SiO ₂	C	S	MgO	MnO	Al ₂ O ₃
Spherical Iron powder	98.8	0.13	98.7	0.048	0.078	0.02	0.001	0.001	0.48	0.32

As observed from Table 5, the reduced spherical iron powder contained at least 98 wt% (i.e. about 98.7 wt%) metallic iron. Further, the spherical iron powder had an average particle diameter between 30 μm and 60 μm with an apparent density of 2.3 g/cc. Said results indicate the achievement of high purity spherical iron powder with finer particle size range by employing the present method.

EXAMPLE 3: Production of spherical iron powder by employing irregular spray roasted iron oxide powder as a raw material and pure hydrogen gas as a reducing agent

Synthesis of spherical iron powder was carried out similar to the procedure described in Example 1. Here, the irregular spray roasted iron oxide powder having the composition as defined in Example 1 was reduced using pure hydrogen gas at about 700°C for a reduction time of about 60 minutes to produce irregular iron powder with an apparent density of 0.8 g/cm³. The subsequent steps/conditions of burning to produce spherical iron oxide remain the same as described in Example 1. The spherical iron oxide was then reduced in hydrogen rich gas

(cracked ammonia) in a reduction furnace at about 700°C for about 180 minutes duration. The resultant loosely sintered spherical iron powder cake was cooled in the furnace to room temperature by passing nitrogen gas. The sintered spherical iron powder cake was then crushed, pulverized and screened to produce spherical iron powder. The chemistry/compositions of the obtained spherical iron oxide and spherical iron powder of this example is provided in Tables 6 and 7, respectively.

Table 6: Chemical composition of spherical iron oxide produced after combustion

Composition (wt %)	Fe (total)	FeO	Fe (metal)	CaO	SiO ₂	C	S	MgO	MnO	Al ₂ O ₃
Spherical Iron Oxide	71.6	23.5	1.6	0.014	0.01	0.034	0.004	0.04	0.415	0.201

The average particle diameter of the obtained spherical iron oxide in this example was between 80-200 µm with an apparent density of 2.5 g/cc.

Table 7: Chemical composition of spherical iron powder produced after reduction of spherical iron oxide

Composition (wt %)	Fe (total)	FeO	Fe (metal)	CaO	SiO ₂	C	S	MgO	MnO	Al ₂ O ₃
Spherical Iron powder	98.9	0.13	98.8	0.039	0.049	0.02	0.003	0.001	0.475	0.217

The obtained spherical iron powder primarily contained metallic iron at about 98.8 wt % and possessed an average particle diameter between 60-100 µm with an apparent density of 2.1 g/cc. Said results indicate the achievement of high purity spherical iron powder with finer particle size range by employing the present method.

EXAMPLE 4: Production of spherical iron powder by employing irregular spray roasted iron oxide powder as a raw material and coke breeze as a reducing agent

Synthesis of spherical iron powder was carried out similar to the procedure described in Example 1. Here, the irregular spray roasted iron oxide powder having the composition as defined in Example 1 was reduced using very fine carbonaceous coke breeze as reducing agent at reducing temperature of about 900°C for a reduction time of about 300 minutes to produce irregular iron powder with an apparent density of 1 g/cm³. The subsequent steps and rest all

parameters to produce the final spherical iron powder remain the same as described in Example 3. The chemistry/compositions of the obtained spherical iron oxide and spherical iron powder of this example is provided in Tables 8 and 9, respectively.

5 Table 8: Chemical composition of spherical iron oxide produced after combustion

Composition (wt %)	Fe (total)	FeO	Fe (metal)	CaO	SiO ₂	C	S	MgO	MnO	Al ₂ O ₃
Spherical Iron Oxide	70.3	18.2	1.3	0.014	0.9	0.032	0.003	0.04	0.28	0.54

The average particle diameter of the obtained spherical iron oxide was between 70-180 μm with an apparent density of 2.8 g/cc.

10 Table 9: Chemical composition of spherical iron powder produced after reduction of spherical iron oxide

Composition (wt %)	Fe (total)	FeO	Fe (metal)	CaO	SiO ₂	C	S	MgO	MnO	Al ₂ O ₃
Spherical Iron powder	98.04	0.04	98	0.039	0.97	0.03	0.004	0.001	0.3	0.56

The spherical iron powder primarily contained metallic iron at about 98 wt % with very few impurities and possessed an average particle diameter between 30-80 μm and an apparent density of 2.25 g/cc. Said results indicate the achievement of high purity spherical iron powder with finer particle size range by employing the present method.

The present disclosure is thus successful in providing a simple, cost-effective, non-toxic and efficient method of producing highly pure iron powder of fine particle size range. Said iron powder with high purity is immensely useful and can be employed in different industries, including powder metallurgy, diamond tools, welding and chemical industry. Highly pure iron powder especially finds applications in porous electrodes, metal injection molding (MIM), water treatment, food agriculture, catalyst, electromagnetic and metal fuel applications.

WE CLAIM:

1. A method for producing spherical iron powder, comprising steps of:
 - reducing iron oxide in presence of a carbon monoxide rich gas or a hydrogen rich gas or a solid carbonaceous reducing agent or any combination thereof, to obtain irregular iron powder,
 - burning the irregular iron powder in presence of oxygen rich gas, to obtain spherical iron oxide,
 - reducing the spherical iron oxide in presence of a carbon monoxide rich gas or a hydrogen rich gas or a combination thereof, to obtain the spherical iron powder.
2. The method of claim 1, wherein the iron oxide employed for reduction to obtain the irregular iron powder is a spray roasted iron oxide powder.
3. The method of claim 2, wherein the spray roasted iron oxide comprises total iron Fe(t) at a wt% of about 69.09, FeO at a wt% of about 0.26, SiO₂ at a wt% of about 0.08, CaO at a wt% of about 0.01, MgO at a wt% of about 0.027, MnO at a wt% of about 0.38, Al₂O₃ at a wt% of about 0.16, S at a wt% of about 0.01, C at a wt% of about 0.123, along with at least one or more additional elements or components selected from Cr, Ti, Cu and P at various wt% to make up the final composition to 100 wt%.
4. The method of claim 1, wherein the carbon monoxide rich gas is selected from the group consisting of coke oven gas, blast furnace gas, steel making converter gas, pure carbon monoxide and combinations thereof.
5. The method of claim 1, wherein the hydrogen rich gas is selected from the group consisting of cracked ammonia, hydrogen separated from coke oven gas, pure hydrogen and combinations thereof.
6. The method of claim 1, wherein the solid carbonaceous reducing agent is selected from the group consisting of carbon black, anthracite coal, coal tar pitch, coke breeze and combinations thereof.
7. The method of claim 1, wherein the oxygen rich gas is selected from the group consisting of pure oxygen, air, a mixture of oxygen and fuel gas, and combinations

thereof; and wherein the mixture of oxygen and fuel gas is selected from one or more from the group consisting of oxygen and acetylene, oxygen and propane and oxygen and butene.

8. The method of claim 1, wherein the reduction of the iron oxide to obtain irregular iron powder is carried out in a reduction furnace at a temperature of about 500°C to about 1200°C and for a time-period of about 60 minutes to about 360 minutes.
9. The method of claim 1, wherein the burning of the irregular iron powder is carried out using a powder dispensing unit and gas-powder burning torch; and wherein the burning of the irregular iron powder generates exothermic heat which melts and converts the irregular iron particles of the irregular iron powder into the spherical iron oxide particles due to surface tension.
10. The method of claim 1, wherein the reduction of the spherical iron oxide to obtain spherical iron powder is carried out in a reduction furnace at a temperature of about 500°C to about 1200°C and for a time-period of about 60 minutes to about 300 minutes.
11. The method of claim 1, wherein the method further comprises additional steps of cooling, crushing, pulverizing and screening to obtain the irregular iron powder or the spherical iron powder.
12. The method of claim 1, wherein the obtained spherical iron oxide comprises a dense spherical morphology of iron oxide particles having an average particle diameter between 10 µm to 200 µm and an apparent density between 2.5 g/cc to 3 g/cc.
13. The method of claim 1, wherein the obtained spherical iron powder has at least 98% metallic iron [Fe(m)] and comprises a dense spherical morphology of iron particles having an average particle diameter between 5 µm and 100 µm and an apparent density of 2.1 g/cc to 2.6 g/cc.
14. The method according to any one of claims 1 to 13, wherein the method comprises:
 - (a) heating the iron oxide in a reduction furnace to a temperature of about 500°C to about 1200°C and reducing the iron oxide in presence of the carbon monoxide rich gas or the hydrogen rich gas or the solid carbonaceous reducing agent or any

combination thereof, for a time-period of about 60 minutes to about 360 minutes to form a loosely sintered iron powder cake,

- (b) cooling the loosely sintered iron powder cake in the furnace to room temperature under inert atmosphere, followed by crushing, pulverizing and screening said iron powder cake to obtain the irregular iron powder,
- (c) burning the irregular iron powder in the presence of the oxygen rich gas to generate exothermic heat to melt and convert the irregular iron particles of the irregular iron powder into the spherical iron oxide powder due to surface tension,
- (d) heating the spherical iron oxide powder in a reduction furnace to a temperature of about 500°C to about 1200°C and reducing the spherical iron oxide powder in presence of the hydrogen rich gas, for a time-period of about 60 minutes to about 300 minutes to form loosely sintered spherical iron powder cake, and
- (e) cooling the loosely sintered spherical iron powder cake in the furnace to room temperature under inert atmosphere, followed by crushing, pulverizing and screening said spherical iron powder cake to obtain the spherical iron powder.

15. The method of claim 14, wherein said spherical iron oxide comprises Fe(t) at a wt% of about 70.3 to about 71.7, FeO at a wt% of about 18.2 to about 23.99, Fe(m) at a wt% of about 1.3 to about 1.8, CaO at a wt% of about 0.01 to about 0.014, SiO₂ at a wt% of about 0.01 to about 0.9, MgO at a wt% of about 0.026 to about 0.04, MnO at a wt% of about 0.28 to about 0.415, Al₂O₃ at a wt% of about 0.2 to about 0.54, C at a wt% of about 0.03 to about 0.034, and S at a wt% of about 0.002 to about 0.004, along with at least one or more additional elements or components selected from Cr, Ti, Cu and P at various wt% to make up the final composition to 100 wt%.

16. The method of claim 15, wherein said spherical iron powder comprises Fe(t) at a wt% of about 98.04 to about 98.9, FeO at a wt% of about 0.04 to about 0.32, Fe(m) at a wt% of about 98 to about 98.8, CaO at a wt% of about 0.039 to about 0.052, SiO₂ at a wt% of about 0.078 to about 0.97, MgO at a wt% of about 0.001 to about 0.042, MnO at a wt% of about 0.3 to about 0.48, Al₂O₃ at a wt% of about 0.217 to about 0.56, C at a wt% of about 0.02 to about 0.03, and S at a wt% of about 0.001 to 0.005, along with at least one or more additional elements or components selected from Cr, Ti, Cu and P at various wt% to make up the final composition to 100 wt%.

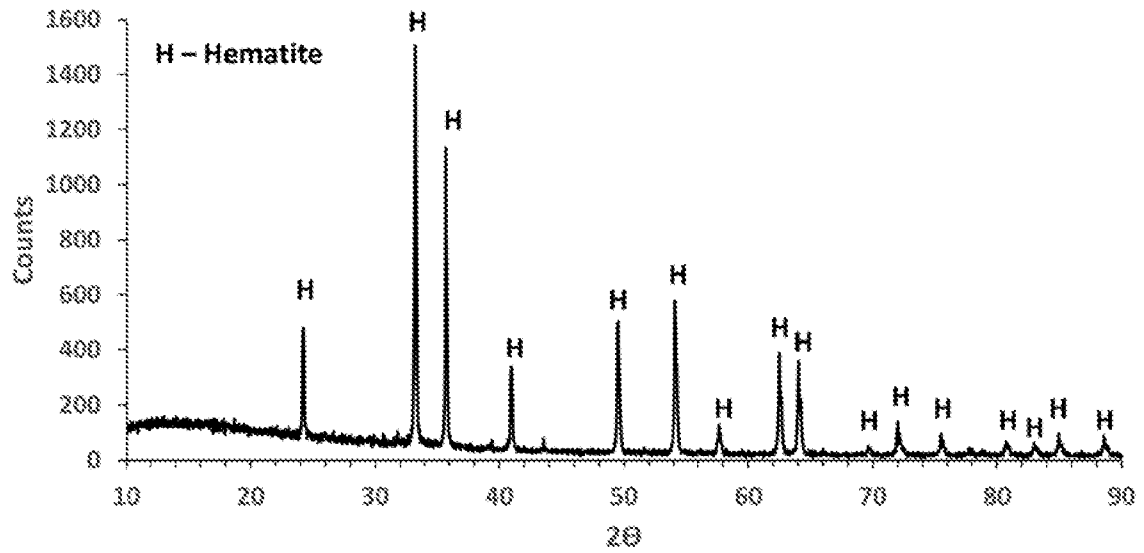


Figure 1

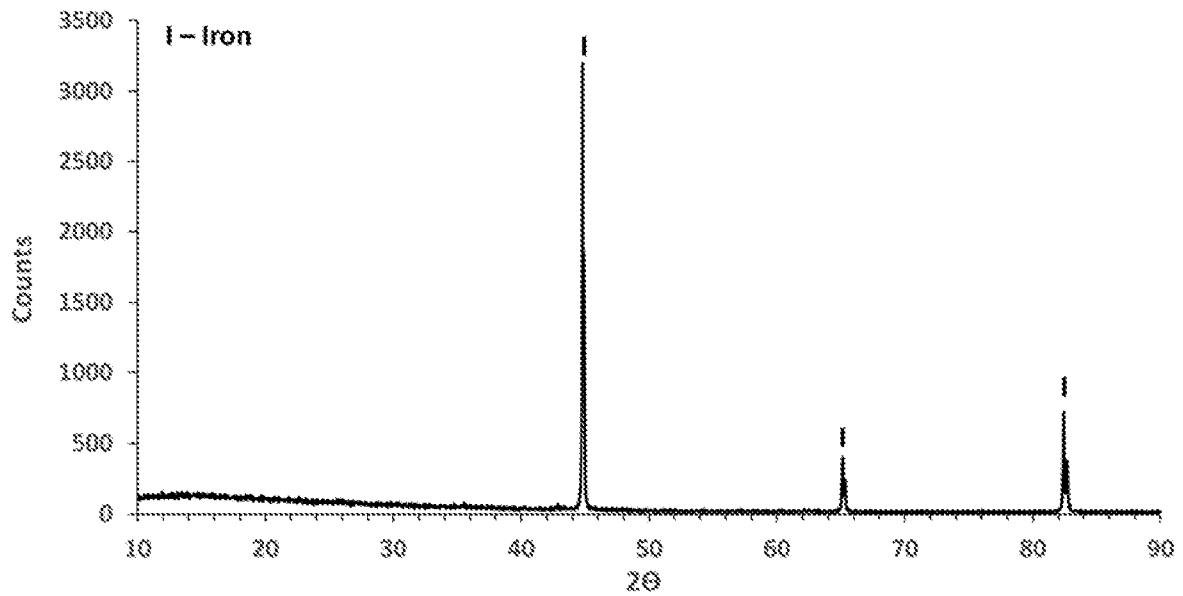


Figure 2

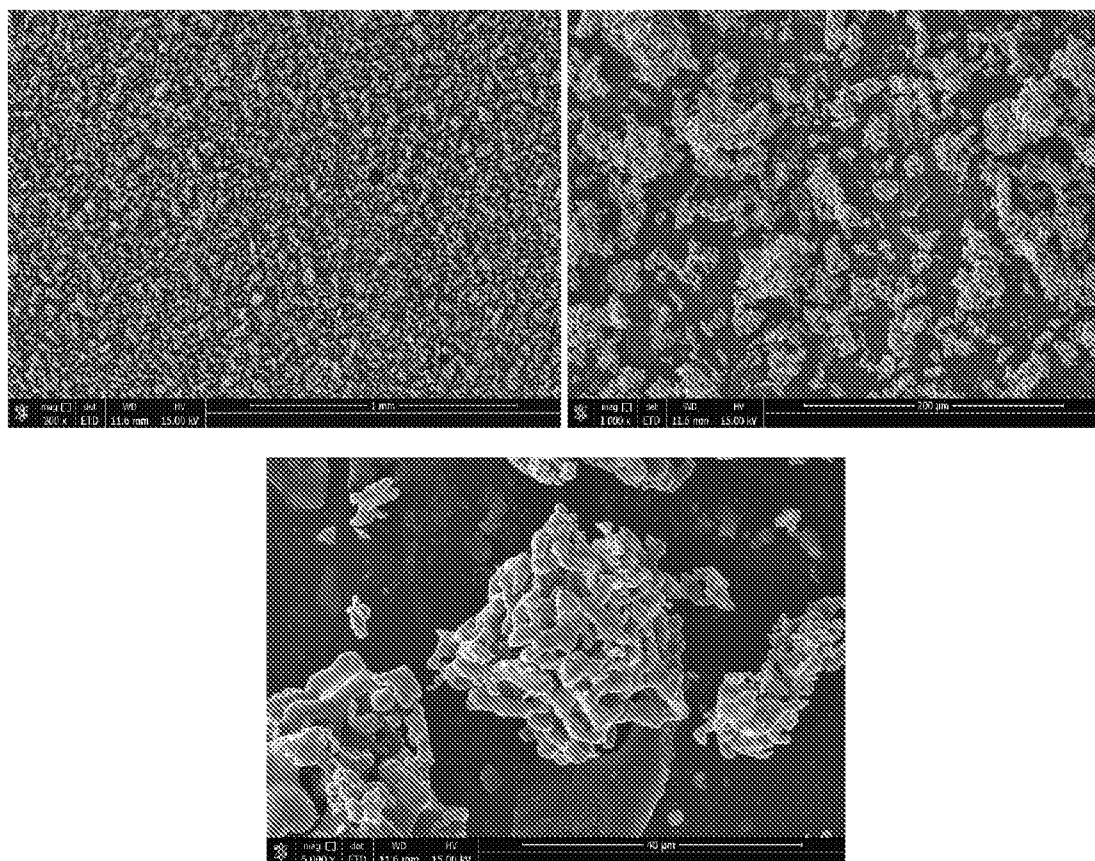


Figure 3

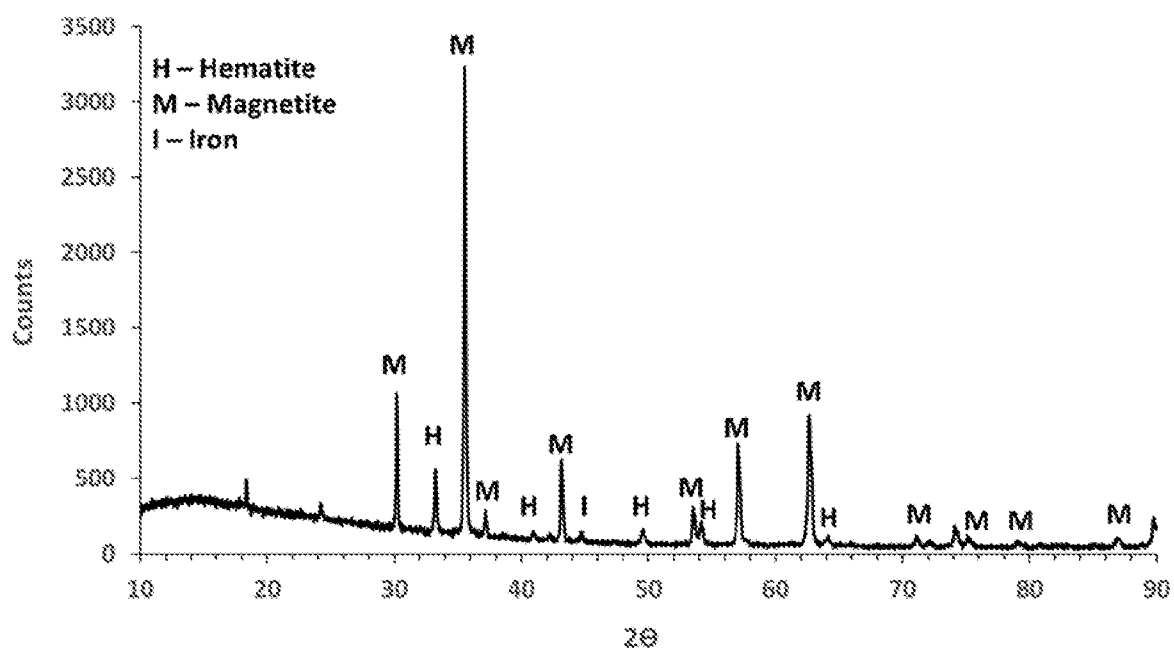


Figure 4

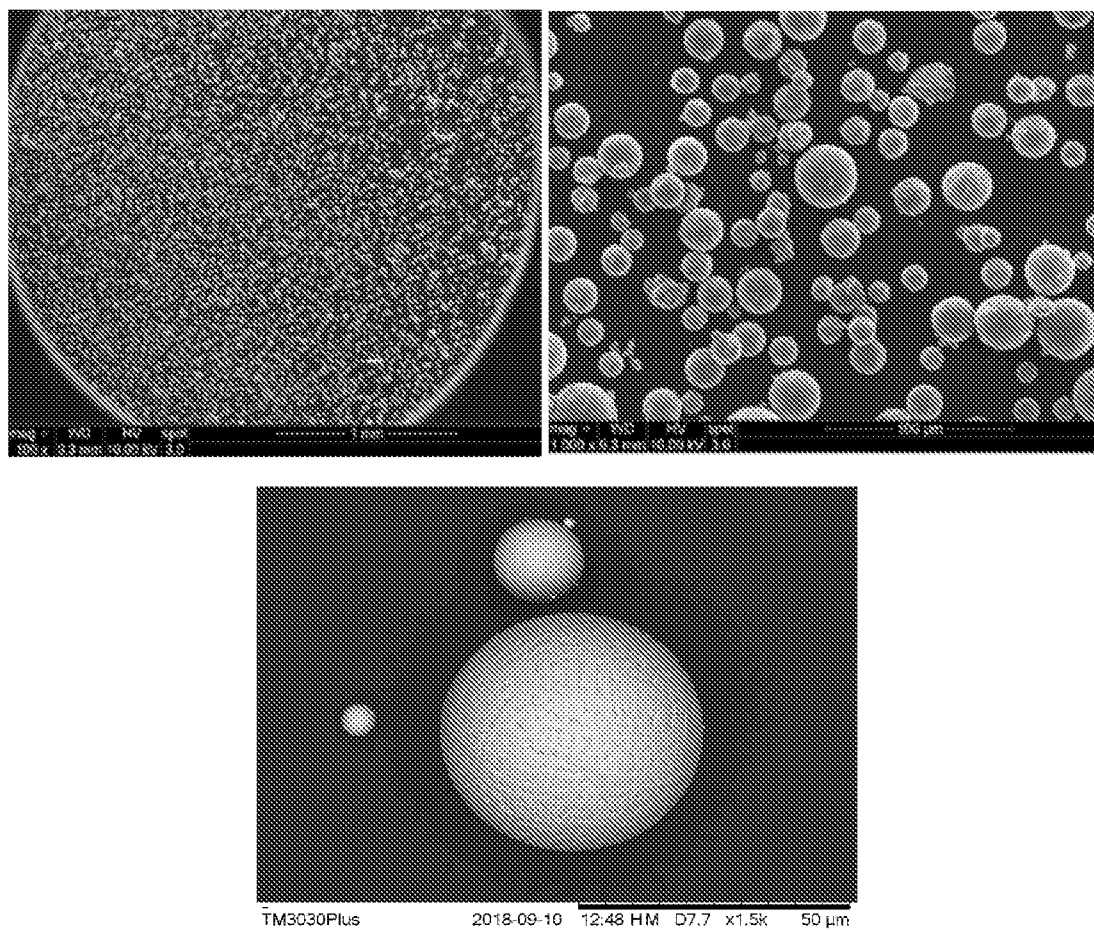


Figure 5

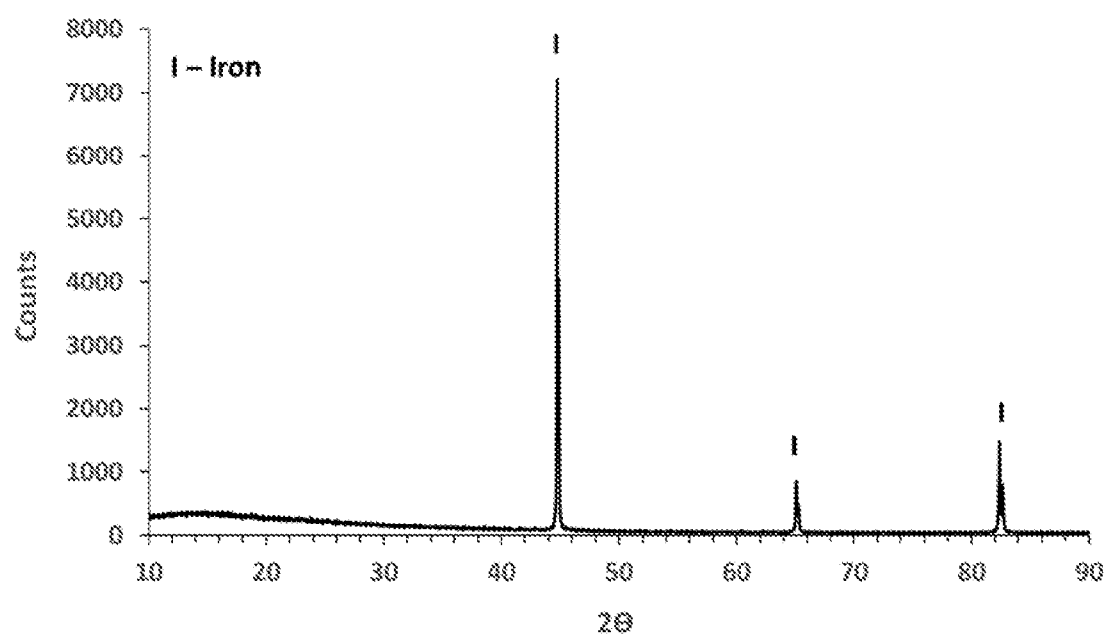


Figure 6

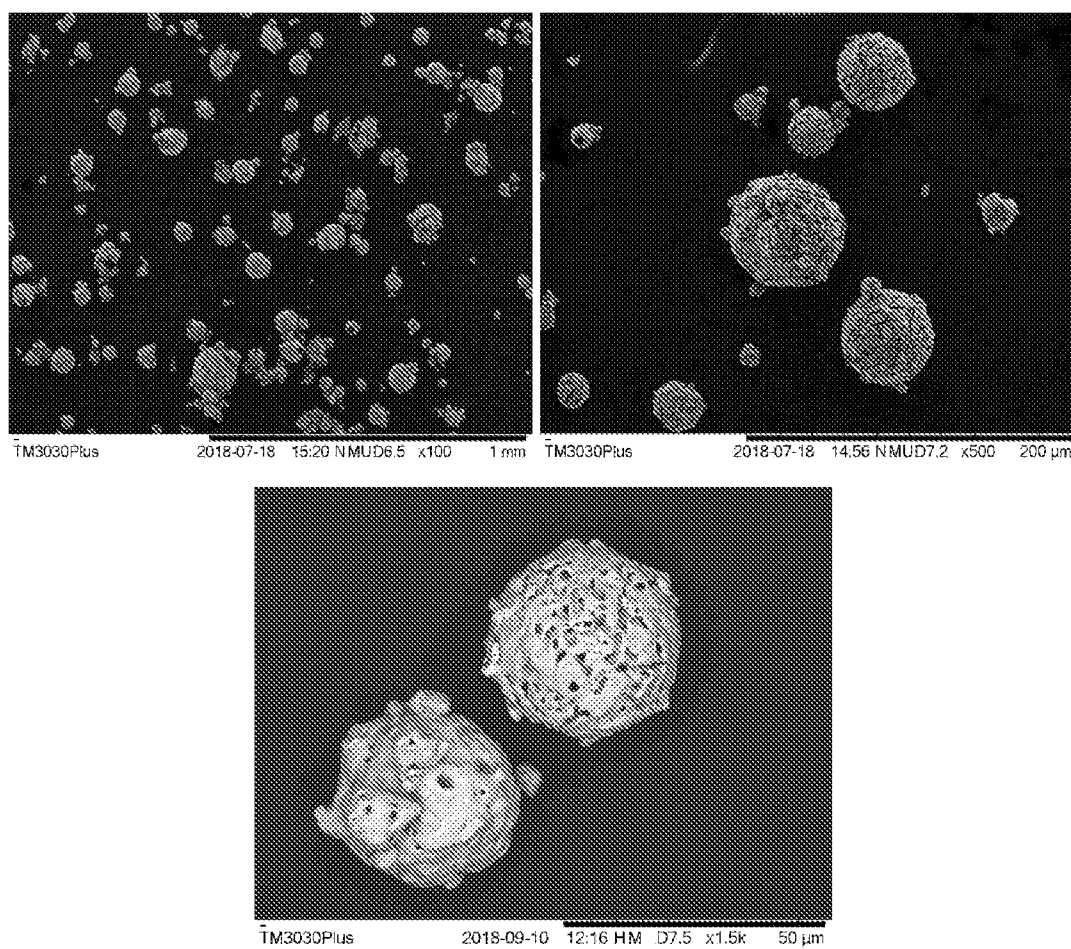
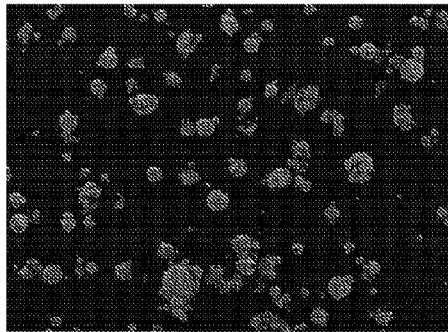
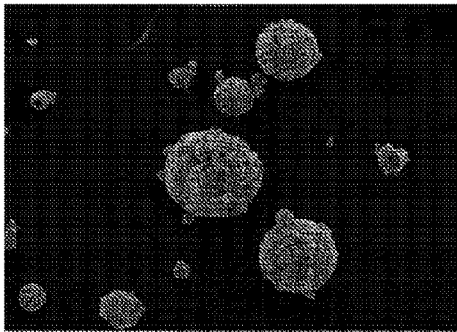


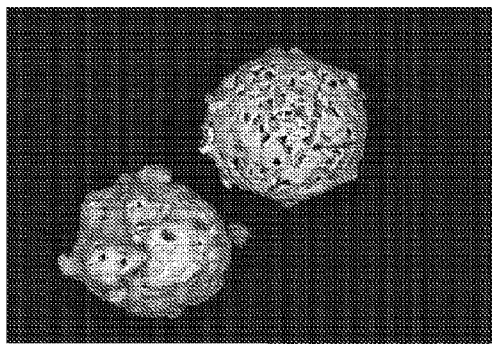
Figure 7



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TM3030Plus 2018-07-18 14:56 NMUD7.2 x500 200 μ m



TM3030Plus 2018-09-10 12:16 HM D7.5 x1.5k 50 μ m