Abstract:
The invention relates to an improved method for processing high zinc iron ores for production of iron and steel comprising the steps of: producing agglomerate comprising a mixture of iron oxides, carbonaceous materials, and fluxes with mean-particle size respectively of 35 to 70, 25 to 60, and 45-85 microns, to form agglomerates of 8 to 15 mm size using combination of organic and inorganic binders and moisture to achieve the desired properties of the agglomerates; dezincificating and metalising the agglomerates in a furnace; smelting the reduced agglomerates, in hot/cold charging condition, to form hot metal (iron) in a furnace leading to production of crude steel; recovering zinc values from waste gas stream of the furnaces by carrying out conventional zinc extraction process.

Title: METHOD FOR SEPARATION OF ZINC AND EXTRACTION OF IRON VALUES FROM IRON ORES WITH HIGH CONCENTRATION OF ZINC
TITLE
Method for separation of zinc and extraction of iron, values from iron ores with high concentration of zinc

FIELD OF INVENTION
This invention relates to a two stage method for removal of zinc, reduction of iron ores and production of liquid metal using iron ores and process dust containing high zinc. The invention further relates to a selection of binders in agglomeration and thermal profile of the furnace through sequential adjustment of porosity which accelerates the removal of zinc vapors at high temperatures during reduction reaction. In first stage, non-shaft furnaces are used for zinc removal and direct reduction of iron ores and dust. In second stage, electric furnace is used to produce liquid metal and remove the remaining zinc from reduced metal by using innovative the slag chemistry.

BACKGROUND OF INVENTION
Blast Furnace process is used worldwide for production of hot iron metal using variety of iron ores. The volatile impurities such as alkalis, zinc, lead, etc. creates various operational problems in Blast furnace process. Therefore blast furnace process is not convenient route for processing iron ores with high zinc content. Alternative processes developed for iron and steel making, where shaft furnaces are used, are also not suitable for treatment of these high zinc ores. The boiling
point of zinc metal is -910°C and in oxidizing conditions it forms stable zinc oxide (solid phase). In furnaces where various temperature zones and oxidizing conditions exist, zinc recycles / accumulates inside the furnace. For example, in the shaft furnace zinc vapor coming from high temperature zone (bottom part) are condensed on the charge or furnace wall in the low temperature zone (T < 900°C) at the top, which results in recirculation of zinc within the system. The zinc recirculation increases the coke rate and creates many operations difficulties. Therefore, high zinc iron ores are rarely used in iron and steel industry.

OBJECTS OF INVENTION

It is therefore an object of the invention to propose an improved method for dezincification and metallization of iron ores with high zinc content by using solid state reduction in non shaft furnaces.

Another object of the invention is to propose a combination of binders in the improved method of agglomeration process for rapid removal of zinc vapor during reduction reaction, through sequential adjustment of porosity.

A further object of the invention is select a suitable combination of processes for production of iron and steel using product of first stage i.e. 'direct reduced iron.
A still further object of the invention is to propose a process for recovery of Zn values from waste gas stream for extraction of Zn metal.

**SUMMARY OF INVENTION**

Accordingly there is provided an improved method for processing high zinc iron ores for production of iron and steel comprising the steps of; producing of agglomerate comprising a mixture of iron oxides, carbonaceous materials, and fluxes with mean-particle size respectively of 35 to 70, 25 to 60, and 45-85 microns, to form agglomerates of 8 to 15 mm size using combination of organic and inorganic binders and moisture to achieve the desired properties of the agglomerates; dezincificating and metallising of agglomerates in a furnace; smelting the reduced agglomerates, in hot / cold charging condition, to form hot metal (iron) in a furnace leading to producing steel; recovering zinc values from waste gas stream of the furnace by carrying out conventional zinc extraction process.

**BRIEF DESCRIPTION OF THE ACCOMPANYING DRAWINGS**

The invention is explained in greater details with the accompanying drawing:
Figure 1, shows the plot of free energy change Vs temperature for reduction reactions of Zn & Fe oxides, which is used for development of the present invention.

Figure 2, presents the ZnFe2U4 - O2 phase diagram used for controlling the gaseous atmosphere inside various zones in Furnace and for separation of zinc from waste gas stream.

**DETAILED DESCRIPTION OF THE INVENTION**

Common zinc mineral is sphalerite, ZnS. However, zinc also found in the form of Franklinite [(Zn₁,Fe,Mn)(Fe,Mn)₂O₄], a oxide mineral. In magnetite lattice Zn²⁺ can replace Fe²⁺ cations forming stable (Fe₃U₄ - ZnF₂U₄) solid solution phase. The Zn²⁺ cation is smaller in size than Fe²⁺ so replacement of Fe²⁺ by Zn²⁺ reduces the lattice dilation and strain energy. Therefore dissolution of ZnO in magnetite increases thermodynamic and structural stability. Although, the crystal structure of hematite (HCP) does not favors dissolution of ZnO, structure can accommodate partial replacement of Fe³⁺ by Zn²⁺ cations by vacancy formation and lattice dilation. Therefore, high zinc concentration is detected in hematite minerals (as in case of oxidized ores of Thach Khe, Vietnam). It is not possible to remove this lattice zinc in iron ores by conventional beneficiation techniques such as magnetic separation, gravity separation, etc. Therefore present invention
provides a method capable of removing zinc locked in the iron mineral lattice and also produces reduced iron, which can be integrated with conventional iron & steel making process.

Important reduction reactions of ZnO, Fe₃O₄ and ZnFe₂O₄ with solid carbon and CO gas are listed below and a plot of free energy change Vs temperature for these reduction reactions is presented in Figure 1.

\[
\begin{align*}
ZnO + C &\rightarrow Zn + CO \quad -(RI) \\
ZnO + CO &\rightarrow Zn + CO₂ \quad -(KI) \\
Fe₃O₄ + AC &\rightarrow 3Fe + ACO \quad -(R3) \\
Fe₃O₄ + ACO &\rightarrow 3Fe + ACO₂ \quad -(RA) \\
ZnFe₂O₄ + AC &\rightarrow Zn + 2Fe + ACO \quad -(R5) \\
ZnFe₂O₄ + ACO &\rightarrow Zn + 2Fe + ACO₂ \quad -(R6)
\end{align*}
\]

It is observed from the Figure 1 that Zinc oxide reduces to zinc metal by solid carbon (reaction RI) above 900°C temperature, whereas reduction of pure magnetite by carbon (reaction R3) starts above 710°C temperature. Zinc ferrite reduction to metallic zinc and iron (reaction R5) is possible (\(\Delta G < 0\)) above 750°C.
temperature by solid carbon, as thermodynamic activity of zinc oxide changes significantly when it is present in the form of zinc ferrite. The Figure 1 also confirms that gaseous reduction of ZnO or ZnFe2O4 by carbon monoxide requires high temperatures i.e. above 1200°C. The phase diagram of ZnFe2O4 - O2 computed using FACT-Sage program is shown in Figure 2, which shows the boundary lines for thermodynamic stability of various phase (solid, liquid and gas) Zn - Fe - O compounds. Therefore solid state reduction above 750°C temperature and below 10^-6 oxygen partial pressure, are the critical thermodynamic conditions for removal of zinc from magnetite lattice.

In magnetite lattice, Zn²⁺ and Fe³⁺ cations have zero octahedral site preferential energy therefore the site occupancy of cations is mainly decided on basis of cationic radii and charge. As a result, smaller Zn²⁺ and Fe³⁺ cations occupy both tetrahedral & octahedral sites, whereas large Fe²⁺ cations preferentially occupy octahedral sites in magnetite lattice. In reducing atmosphere, the imposed oxygen chemical potential promotes the diffusion of Fe and Zn cations through oxygen anions on CCP lattice in the magnetite, toward the reaction interface on the surface of the grain. The vacancies generated during this process diffuse inward and promote the cation diffusion toward a reaction interface. Because Fe³⁺ and Zn²⁺ cations have zero OSPE, these cations jump from one octahedral site to another via a vacant neighboring tetrahedral site. The charge and ion balanced diffusion path is energetically more favorable to direct jump between two
octahedral sites, which are separated by anions. At high temperatures, zinc is homogeneously distributed in lattice (T > immiscibility dome), the rate of zinc removal depends on the diffusion of Zn through anion lattice. The detail studies of the reaction kinetics and reduction mechanism also revealed that the rate limiting process is diffusion of cations through oxygen anion lattice. Therefore reaction temperatures are higher than thermodynamic conditions for dezincification.

In this innovative method, iron ores with high Zn content are mixed with carbonaceous materials, as a reductant and other fluxes. The mixture is then agglomerated in the form of pellets or briquettes. Desired properties of the agglomerates comprises wet-drop number, dry-drop number, green crushing strength and dry-crushing strength which respectively ranges 6 to 8, 10 to 15, 1.5 kg pellet, and 15kg/pellet. The green agglomerates are dried to remove the moisture. A non shaft furnaces such as rotary hearth furnace is used for solid state reduction and dezincification, However present invention does not exclude operation in the other type of furnaces. In the rotary hearth furnace the agglomerated feed is charged continuously in layers to maintained appropriate height of the burden on the hearth. The agglomerates are heated in different zones of furnace. The heat required for endothermic reduction reactions is supplied by the combustion products and radiation energy from the furnace heaters / burners. The air : fuel ratio of the furnace burners is kept at appropriate levels in different zones to maintain at desired reducing conditions in
various zones of furnace. The carbonaceous material in the agglomerate acts as reductant and also maintain reducing atmosphere close to reaction interface. The ratio of ore to carbonaceous materials is adjusted to provide to C required for reduction and for maintaining reducing atmosphere at reaction interface. Temperature profile of the furnace is maintained as desired to form a molten slag at appropriate time and also allow coalesce of reduced metal. In cooling zone, the reduced pellets are cooled to 800°C to 1000°C required for subsequent processes. If required, the DRI are agglomerated by hot briquetting process. The atmosphere of cooling zone and subsequent hot process is controlled to minimize the reoxidation of newly formed metallic iron. In this innovative process, 70 - 95% degree of metallization with 80 - 95% dezincification can be achieved in the temperature range from 1100 to 1400°C and 10 - 60 minutes heating cycle. The process also generates DRI with very low silicon (0.1 - 0.9%) and carbon (0.3 - 1.5%) content.

In the present invention, it is preferred to maintain furnace conditions in such a way that the product gas flushes / carries the zinc vapors out of furnace as shown in Figure 2. In the preferred case the gas flow follows the charge / heart movement so that hot gas will not come in contact with low temperature charge, on which Zn vapors can deposit and recirculate / accumulate inside the furnace. Other option used in this innovative process is to collect the gas from high temperature zone and cool to temperatures below 900°C to separate the Zn vapors and then recycle in the furnace to maintain the reducing atmosphere.
However, other applications of hot furnace gas (such as air and fuel preheating) are not excluded in this invention.

According to a further advantageous embodiment of this invention, it is preferred to adjust the porosity of dry carbon composite pellets to enhance the vaporization of zinc near reaction interface and rapid transport of Zn vapor to outlet gas stream. This is achieved by using combination of the hybrid binders and moisture in the agglomeration. The inorganic binders doses ranges between 0.5 to 2%, and wherein the organic binders are used in the doses between 1 to 5%. The volume ratios of iron ore, coal, binder and moisture are adjusted in innovative way to generate porosity within agglomerate as reduction and dezincification reaction proceeds. The binder vaporization and coal utilization is sequenced in such a way to compensate the shrinkage of the agglomerate during reduction reaction and also to achieve the required strength in reduced pellets. The composite pellets are dried in the temperature range from 110 to 300°C to remove the moisture and thereby generate the porosity (primary pores). In this invention, the combination of organic and inorganic binders is used so that the organic binder enhances the strength of dry pellets / briquettes whereas inorganic binder provides strength at high temperature inside the furnace during reduction reactions. The organic binder vaporizes in the early stages of reduction reaction which increases 5 - 10% porosity (secondary pores) of the pellets / briquettes. These porous channels (primary and secondary pores)
generated at lower temperature enhance the rapid transport of Zn vapors formed during solid state reduction of magnetite and zinc ferrite solid solution phase above 800°C temperature. As the reduction reaction progresses, carbon / reductant gets consumed which also maintains pore channels i.e. the high porosity for rapid gas phase transport from reaction interface to furnace atmosphere.

According to a further advantageous embodiment of this invention, particle size and size distribution of feed (iron ore, reductant, flux and binders) used for agglomeration process are adjusted to achieve the required green and dry pellet strength, to generate the pore channels for rapid transport of gaseous products and to enhance the rate of reduction reaction (topochemical). Iron oxides, carbonaceous materials, and fluxes are prepared to achieve mean-particle size respectively of 35 to 70, 25 to 60, and 45-85 microns, to form agglomerates of 8 to 15 mm size. As a result, high productivity (tones / hour / m²) is achieved in this short time reduction process (heating and cooling cycle).

In present invention, a combination of fluxes is used to form slag with desired liquidus. The rate of reduction reaction is also adjusted in innovative way to produce desired amount of FeO oxide in the charge during reduction reaction which forms molten slag. The fluxes used in the charge impart desired physico-chemical properties in molten slag and also control loss of Fe in the slag phase. The slag properties are adjusted to dissolve gangue phases and also maintain
desired viscosity so that molten slag will not block the pores and thereby hinder the flow of Zn vapors and product gases. At high temperatures, when desired level of dezincification is achieved the designed slag chemistry forms fluid slag which accelerate coalesce of reduced metallic particles and better separation of slag and metal. Thus in this invention, rapid dezincification and better slag-metal separation is achieved by innovative flux chemistry and heating cycle / rate.

In present invention, the higher degree of metallization and dezincification was also achieved by using appropriate grade of iron ore concentrate. The increase in Fe content of iron ore enhances the degree of metallization and removal of gangue components help to reduce the flux requirement. However, the higher % metallization and lower slag content decrease the cold crushing strength of the reduced pellets. Therefore heating cycle, porosity, feed size, and slag chemistry are adjusted to achieve the desired properties of reduced pellets / briquettes.

The process described in this invention was used for processing Iron ore containing ~ 0.07 % Zinc. The agglomerates were prepared using anthracite coal, iron ore fines and flux, using combination of binders as discussed in the invention. The agglomerates were reduced in a furnace using desired heating profile in the temperature range from 1100 to 1400 C. The DRI with metallization in the range of 70 - 95% and zinc less than 0.01 % were produced by the process. The DRI was used to produce liquid metal in electric furnace.
In the second step of this inventive process, the hot DRI is directly melted in electric furnace to form either a) hot metal, which can be used of BOF steel making, by adjusting the C, Si, S, P levels, or b) directly steel by using double slag practice. Production process options will be dictated by local economics.

One of the embodiments of the present invention is recovery of zinc. The zinc vaporized during reduction in the furnace is carried away by the waste gas stream. The zinc vapors are condensed by reducing the temperature below 900°C and by readjusting the oxygen partial pressure of the gas stream, if required. The waste gas stream from arc furnace used for iron and steel making is also treated in similar way to recover the zinc values. The zinc oxide condensed in the condenser is collected. Since, coal is used as a reductant, the zinc oxides dust also contents many impurities which needs to be removed. When the concentration of the zinc in the dust is > 40 % then dust is used directly for zinc extraction. In this invention carbo-thermic reduction of zinc oxide is carried out to extract metallic zinc which, is then purified by conventional electrolysis technique. On the other hand dust with zinc concentration lower than 40% are reduced in separate campaign to separate the iron and produce high zinc dusts. The other method used for zinc enrichment of dust is smelting of furnace dust in electric arc furnace which generate high zinc dust which can then be treated by conventional routes.
WE CLAIM

1. An improved method for processing high zinc iron ores for production of iron and steel comprising the steps of:

- producing agglomerate comprising a mixture of iron oxides, carbonaceous materials, and fluxes with mean-particle size respectively of 35 to 70, 25 to 60, and 45-85 microns, to form agglomerates of 8 to 15 mm size using combination of organic and inorganic binders and moisture to achieve the desired properties of the agglomerates;

- dezincificating and metallising the agglomerates in a furnace;

- smelting the reduced agglomerates, in hot / cold charging condition, to form hot metal (iron) in a furnace leading to production of crude steel;

- recovering zinc values from waste gas stream of the furnaces by carrying out conventional zinc extraction process.

2. The method as claimed in claim 1, wherein the desired properties of the agglomerates comprises wet-drop number, dry-drop number, green crushing strength and dry-crushing strength which respectively ranges 6 to 8, 10 to 15, 1.5 kg pellets, and 15kg/pellets.
3. The method as claimed in claim 1, wherein the dezincificating and metallising comprises:

- sequentially adjusting the porosity (primary pores) in agglomerates during water evaporation at temperature 80° - 150°C;

- evaporating the organic binders at temperature between 130° to 300°C to create secondary pores; and

- consuming the carbonaceous materials in reduction at a temperature between 500° to 1200°C to create tertiary pores.

4. The method as claimed in claims 1 or 3, wherein the dezincificating and metallising further comprises:

- providing a pore-channel for rapid transportation of gaseous products through selection of mean-particle sizes of the ingredients forming the agglomerate.

5. The method as claimed in claim 1, wherein the dezincificating and metallising further comprises:

- controlling the visocity of the formed slag through combination of the fluxes so as to avoid blocking the pore channels which enables smooth exit of the gaseous products.
6. The method as claimed in claim 1, wherein the furnace is selected from the types of Rotary hearth furnace, non-shaft furnace and multi-hearth furnace.

7. The method as claimed in claim 1, wherein said iron oxides are iron ores containing high zinc concentration in the range of 0.01 to 1 % from iron ores, EAF dust, plant wastes and their combination.

8. The method as claimed in claim 1, wherein said carbonaceous materials comprises of anthracite coal, bituminous coal, coking coal, pet coal, coke breeze, other carbonaceous materials and their combinations.

9. The method as claimed in claim 1, wherein said binders comprises of inorganic, organic binders and their combinations, wherein the inorganic binders is used in the doses between 0.5 to 2%, and wherein the organic binders are used in the doses between 1 to 5%.

10. The method as claimed in claim 1 or 9, wherein the organic binders comprise of dextrins, celluoses, starches, flours and their combination, mono and polyacrylics and acrylamides and their combinations, different gums like guar gum.

11. The method as claimed in claim 1 or 9, wherein the inorganic binders,
comprise bentonite, colloidal silica, expanding clays and their combinations, cement, sodium, silicate.

12. The method as claim in claim 1, wherein the step of producing agglomeration comprises:

- preparing a feed (iron ores, coal, binders, and fluxes) to achieve required particle size and size distribution including surface area;

- blending, mixing and prewetting of the feed fines to achieve required mix for agglomeration;

- preparing the agglomerate in a disc / drum pelletizer or briquetting machines with desired moisture level and processing parameters to achieve required properties / quality of agglomerates; and

- drying the agglomerates in a temperature range from 110 to 300°C to remove the moisture and formation of primary pores

13. The method as claimed in claims 1 to 5, wherein the dezincification and
metallization is carried out in a furnace where different temperature is maintained in different zones thereby removing the organic binder in early stages to form interconnected pore channels and then reducing and vaporizing zinc at higher temperatures.

14. The method as claimed in claims 1 to 5, wherein the fluxes minimize the Fe loss in the slag.

15. The method as claimed in claims 1, 12, and 14, wherein the fluxes comprise oxides of CaO, MgO and SiO₂ and their compounds.

16. The method as claimed in claim 1, wherein the zinc values are separated from waste gas stream of the furnace by reducing the temperature of the waste gas below 900°C and by adjusting CO/CO₂ ratio of the waste gas by introducing air.

17. The method as claimed in claim 16, wherein the zinc values separated are processed in a furnace to enrich the zinc concentration in the product collected from waste gas stream, wherein the compounds collected from the waste gas stream, having more than 40% zinc content, and being adapted for extraction of zinc by conventional processes.
An improved method for processing high zinc iron ores for production of iron and steel as substantially described and illustrated herein with reference to the accompanying drawings.
Figure 1:

R1 $\rightarrow$ ZnO + C $\rightarrow$ Zn + CO
R2 $\rightarrow$ ZnO + CO $\rightarrow$ Zn + CO$_2$
R3 $\rightarrow$ Fe$_3$O$_4$ + 4C $\rightarrow$ 3Fe + 4CO
R4 $\rightarrow$ Fe$_3$O$_4$ + 4CO $\rightarrow$ 3Fe + 4CO$_2$
R5 $\rightarrow$ ZnFe$_2$O$_4$ + 4C $\rightarrow$ Zn + 2Fe + 4CO
R6 $\rightarrow$ ZnFe$_2$O$_4$ + 4CO $\rightarrow$ Zn + 2Fe + 4CO$_2$

Figure 2
INTERNATIONAL SEARCH REPORT

A CLASSIFICATION OF SUBJECT MATTER
IPC®: CZZB 1/24Z (2006.01); CZZB 5/10 (2006.01); CIZB 5/16 (2006.01); CZZB 19/04 (2006 01), CZ1B 3/02 (2006.01), CZ1B 3/04 (2006.01)
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B FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC®: C22B, 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)
EPODOC, WPI, X-FULL, IPDL

C DOCUMENTS CONSIDERED TO BE RELEVANT

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<td>AU 489386 B2 (Board of Control of Michigan Technological University) 27 March 1975 (27.03.1975) Description, p. 5, p. 6-12, p. 13, p. 1, Fig. 1, Claims 1, 2, 5, 6, 9, 11</td>
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### INTERNATIONAL SEARCH REPORT

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