Recycling of ferromanganese gas cleaning plant (GCP) sludge by novel agglomeration

Y. Rama Murthy a,⇑, Gajanan U. Kapure a, Sunil Kumar Tripathy a, G.P. Sahu b

a Ferro Alloy Mineral Research Group, Research and Development Division, Tata Steel Ltd., Jamshedpur-831001, India
b Ferro Alloy Plant, Joda, Odisha, India

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Abstract
Ferroalloy industries rely on minerals which are not available in pure form. The total value chain is very cost intensive and market driven. From mineral and environment conservation point of view every possible aspect must be explored for the economic utilisation of waste and low-grade ores. Unlike other metallurgical processes, Ferro alloys production generates a wide variety of waste rich in manganese/chrome which has a potential for recycling back. Fine size, high moisture content and presence of alkalis categorise this material into hazardous waste and economically non-viable. The challenge is to convert such anthropogenic material into a suitable feedstock for the furnace. In this article, the results of smelting trials carried out in electric arc furnace (EAF) using novel extruded briquettes (BREX) produced from the wastes of ferroalloy plant is discussed. The briquette produced by this technique exhibit high physical and metallurgical property and can replace the natural ore as a charge to some extent. Extruded briquettes (BREX) can be efficiently used as one of the essential charge component (up to 30% of the ore part of the charge) for the Silicomanganese smelting thus improving technical and economical parameters of the furnace and decreasing the self-cost of the Silicomanganese production.

1. Introduction

Manganese ore and its alloy play a critical role in both metallurgical and non-metallurgical industries. A major share of the mined manganese (high grade) ore gets converted directly into various grades of ferroalloys produced by pyrometallurgical route (i.e., in electric/submerge arc furnaces (EAF/SAF) and which is finally consumed in steelmaking in the form of manganese ferroalloy. Whereas, low to medium grade ores (<40%Mn) are processed through the conventional method of reduction roasting, magnetic separation followed by hydrometallurgical processing to produce chemical grade manganese dioxide (CMD), electrolytic manganese (EM) or electrolytic manganese dioxide (EMD) (Gaal et al., 2010). The rise in the demand for steel has increased the consumption of ferroalloy production proportionally. With an increase in the demand for manganese alloys and depletion in the primary resource of manganese ores, secondary manganese sources (like medium to low grade ore, Mn rich sludge, etc.), which cannot be economically processed by conventional (pyrometallurgical or pyro-pre-treatment) processes, become the prime source of manganese. Further the stringent environmental regulations (CO₂ emission, mine waste disposal, sludge reutilisation, etc.) pose a challenge for the sustainability of mining and manganese industries. Ferro manganese production generates a wide variety of products/by-products in the form of slag, off-gas, and sludge, rich in manganese with a potential to recycle back. Among the solid wastes generated, slag contributes major portion followed by sludge, which is generated when the off-gases from the furnace are cleaned with a wet scrubber which contains 35–40 wt% manganese (Zagirov et al., 2011). An overview of ferromanganese process is presented in Fig. 1.

Sludge generated from the gas cleaning plant (GCP) during the production of ferromanganese through submerged arc furnace (SAF) is considered as the important and recyclable source of manganese along with the slag. Sludge with fine particle size and high moisture content cannot be recycled as it is. Stringent pollution control board (PCB) norms, does not allow such material to sell off directly into the commercial market. Further, the regulations for the suppression of pollutants arising from ferroalloy industries and increasing costs of raw materials have made it imperative to recycle its waste. The challenge is to develop a process/technology which converts such anthropogenic waste into a suitable feedstock...
Diverse recycling methods/route for the utilization of fine and moist materials are available. Sintering, pelleting and briquetting (Fig. 2) are the three conventional technologies widely being used for agglomeration of ore fines. High capital investment coupled with the introduction of gangue through solid fuel prohibits the use of sintering process. While sintering is one of the proven processes for agglomeration of high grade manganese fines, it will be essential to locate the sintering plant near the ferromanganese plant to avoid undue fine generation by sinter handling. One of the most advanced and environmentally friendly ways are the production of manganese-ore pellets [Balashov et al., 2015]. These pellets are charged into SAF after preheating and pre-reduction in rotary kiln with waste gases [Ishitobi et al., 2010]. Though capital intensive, pelletization is preferred by large producer’s due to the possibility of utilizing high grade concentrates which are available in very fine form. In addition, the physical properties of the pellets are far superior to briquettes, which can withstand degradation during processing for preheating and for pre-reduction. Generally pelletization of manganese ore fine is generally not practised due to the requirement of high temperatures (<1150 to 1250°C) and fine grinding of ore which is not economical viable. Gaal et al. (2010) classified the utilization of ferromanganese waste into different process routes and categorised accordingly the process merits/demerits into:

(i) %waste deposited and manganese recovered (Fig. 3a)
(ii) % heavy metals (Zn, Pb)/alkalis back to furnace (Fig. 3b)
(iii) associated product and technological risks (Table 1).

Briquetting process and direct injection of fines have higher preferences among others for maximum utilisation of waste and recovery of manganese metal, though it is associated with poor furnace operation, which can be tackled with advanced agglomeration techniques.

3. Extrusion of ferrous and non-ferrous wastes

Extrusion is a well-established and extensively used process in ceramic industries for making various high-end ceramics. Stiff extrusion process consists of three sections i.e., primary mixer, secondary mixer and pug sealer with vacuum chamber. The extrusion process begins with feeding the charge mix into the hopper of the briquetting line, where from it goes through the homogenizing mixers to the extruder. The blended material from the feeder goes into the primary mixer where it is intensively sheared resulting in particle breakdown within the material mass. These mixers have a high-pressure screw section which intensively mixes the material to produce a homogeneous mixture which improves the extrudability of the material. The mix enters the vacuum chamber being partially agglomerated. The most unique aspect to stiff extrusion is the use of vacuum. This step involves subjecting the material to an intense vacuum prior to extrusion to remove all the compressible...
This results in a much stronger and more durable agglomerated product. The combination of high auger pressure and vacuum de-airing results in a tougher agglomerated product, even in the green state, that can withstand multiple handling operations. The vacuum is maintained throughout the working volume of the extruder. The dense mix is then extruded through die of required diameter and comes out as green extruded briquette. The application of vacuum is to lower the binder content of the briquette and achieve desired level of initial green strength as compared with the traditional briquetting technologies. This allows the transportation and the stockpiling of BREX for strengthening without any fines generation. The cross-sectional view of the extruder and extrusion process is presented in Fig. 4. Stiff extrusion relies on heavy duty augers to create high pressures for forging materials through forming dies. As the materials are pressed through the augers and dies under high pressure and shear there is a lot of particle breakdown that occurs within the material mass. The particle breakdown that occurs is unique to the stiff extrusion process and the affect is to produce a particle size distribution that improves the "extrudability" of the material. This high pressure and shear has the added benefit of intensively mixing the materials to a high degree of homogeneity. Experience of the stiff extrusion agglomeration was not studied systematically, which prevented its further popularity with metallurgists and in processing of metallurgical wastes. The first systematic successful trial in ferroalloy industry using novel agglomerates (BREX) as a feed was reported by Bizhanov et al. (2012a) to produce ferromanganese. In the same year (2012), the term BREX (briquettes of extrusion) has been introduced to distinguish these kinds of agglomerates from conventional briquettes (Bizhanov, 2013). Evaluation of metallurgical properties of extruded briquettes produced from blast furnace waste were studied and presented by Bizhanov et al. (2012b) and Kurunov and Bizhanov (2014). The results revealed that it is possible to use extruded briquettes in steelmaking process as a partial or 100% replacement of primary feed. Dalmia et al. 2012 reported successful production of pig iron via blast furnace route, with 80% BREX (blast furnace waste: iron-and-carbon-bearing) as a charge. This process eliminates the use of limestone and dolomite and reduced coke consumption by 150 kg/ton pig iron. These extruded briquettes were produced in an industrial line stiff-vacuum extruder from metallurgical sludge and dust at the metallurgical plant of Suraj Products Ltd., Rourkela, India.

<table>
<thead>
<tr>
<th>Utilisation process</th>
<th>Product</th>
<th>Technological risk</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrometallurgy</td>
<td>Mn₂SO₄</td>
<td>Unknown</td>
</tr>
<tr>
<td>Briquetting/Direct Injection/Sinter</td>
<td>Mn alloy</td>
<td>Poor furnace operation</td>
</tr>
<tr>
<td>Solid state reduction</td>
<td>Mn ore substitute</td>
<td>Operation of RHF may be difficult</td>
</tr>
<tr>
<td>Oxyfines burner</td>
<td>High Mn Slag</td>
<td>O₂ is expensive. Fumes Not known</td>
</tr>
</tbody>
</table>

Table 1
Product and technological risk.

Fig. 3. Utilization of Ferromanganese dusts (Gaal et al., 2010).

Fig. 4. Cross sectional view of extruder.
4. Challenges in cold agglomerates

Blast furnace or electric arc furnaces are generally fed with raw ore or briquettes or pellets. Cold bonded agglomerates as a feed to furnace are not acceptable due to many reasons of which strength of agglomerate is critical one. The briquettes should not disintegrate too early inside the furnace, else it will lead to the generation of fine/dust and reduce the permeability to the gases. Secondly, appropriate binder is important to provide cohesion between the particles of the briquette and thereby increase the mechanical strength. Organic and inorganic binders have been used by many researchers attempted to manufacture briquettes with an adequate RDI value for use in blast furnaces. Organic binders decompose at high temperatures (>400 °C) and degrade the briquette, reducing the viscoelasticity observed at room temperature. Rheology phenomena may occur during the briquetting process depending on the force applied to the briquette. Granular materials interact with contact forces in this process and resist shearing stress to a certain level, maintaining the static configuration of the particles. The disadvantage is the disintegration in the granular zone in this reactor. This is due to the destruction of the binder phase during at higher temperatures. The overall challenges in novel agglomeration process are summarised Fig. 5.

5. Materials and method

Extruded briquettes were produced in a lab extruder (Fig. 6a and b) and industrial scale extruder (Steele make 25A Series Extruder) with a capacity of 20 tph. Three feed mixes with different sludge to fine ratio was prepared. For the lab extrusion process, feed consisting of fractions <3 mm size were used. Raw material mix was first homogenized in a laboratory Hobart mixture. The feed mix was then sheared in the extruder using shear plates. Shear mixing creates better water distribution and enhances plasticity and high degree of homogeneity. The laboratory extruder simulates the feed material through the sealing auger and die, into the vacuum chamber. The laboratory extruder consists of two chambers with a sealing die between. The rear chamber is fitted with a hopper for feeding the mix into the chamber. The feed mix passes through a shear plate into the second chamber. The second chamber contains vacuum facility. In this chamber the material is subjected to intense vacuum prior to extrusion in order to remove all of the compressible air from the material before it is compressed under high pressure. Extrusion was performed using a multi-hole 19.5 mm diameter die. The main components of the BREX are manganese sludge, manganese ore fines. The components of the mix are presented in Table 2.

Representative samples of GCP sludge and ore fines were collected and analysed for size and chemical constituents. The briquettes were subjected for cold compressive strength (CCS) analysis after 7 days of drying to know the strength of the briquette to withstand the load while handling transporting and storing in the bunker. Studies (Bulatov and Danishevskii, 1987; Jones, 1963) revealed that briquettes based on a cement–bentonite binder can be used as a charge component for a metallurgical furnace within 3 days of drying under natural conditions. As a result, the required sizes of briquette storage can be decreased. The threshold

There is a need to study the feasibility and optimize alternate briquetting techniques for fine and sludge material

Fig. 5. Challenges in novel agglomeration process.
level of compressive strength can be easily achieved by a simple increase in the fraction of this combined binder. The binder and plasticizer used were Portland cement and bentonite respectively. The cement–bentonite binder properties related to the formation of coagulation structures in the cement–bentonite–water system leads to the modification of the properties of the binder. Hydrated cement particles are gradually coated with an impermeable shell of flaky bentonite particles. The number of adsorbed bentonite particles, measuring particle size distribution and shape of pourable bulk materials in the range from 30 μm to 30 mm. It can be observed from the size analysis (Fig. 7) that major fraction of the sludge is having particles finer than 30 μm. Particles having such fineness generally contains higher inherent moisture content and is difficult to handle and store in different seasons.

6. Results and discussion

The chemical constituents of sludge and fines are presented in Table 4. It can be observed that the Mn content of the sludge is quite higher than that of fines. Though sludge contain bit higher amount K2O the Mn:Fe is higher. The sludge being ultrafine in nature, size analysis was carried out in a laboratory Camsizer XT. It is used for the measurement of fine powders and agglomerated particles, measuring particle size distribution and shape of pourable bulk materials in the range from 30 μm to 30 mm. It can be observed from the size analysis (Fig. 7) that major fraction of the sludge is having particles finer than 30 μm. Particles having such fineness generally contains higher inherent moisture content and is difficult to handle and store in different seasons.

6.1. Decrepitation Index (DI)

As the lump/feedstock land on top of the hot burden in the furnace and descent, they are subjected to thermal shock which may also cause breakdown of the lump/feed. The presence of hydrated and carbonate minerals as well as combined moisture is closely linked to the decrepitation of lump particles. It is therefore not surprising that thermally processed ores, such as sinter and pellets, do not decrepitate. DI quantifies the resistance of lump ore to thermal degradation. Porous ore does not decrepitate as much as dense ore even when it has high LOI. The phase transformation experienced by the oxides at 700 °C causes volumetric contractions, which yield anisotropy that induces stress in specific regions of the ore particle, leading to the formation and propagation of cracks. Two tests were carried out for DI in the laboratory muffle furnace with normal air at a temperature of 700 °C (Fig. 8). The results are presented in Table 5.

![Figure 7. Particle size analysis of sludge.](image-url)
After completion of test, there were no fines which indicate the BREX is more porous than lump ores. Average differential weight loss observed was 9.28%. A Decrepitation Index (DI) value of <15, is desirable for lump iron ore.

6.2. Reducibility Index (RI)

Reducibility parameters illustrate the possibility for removal of combined from the ore. A high reducibility is desirable, since it decreases the amount of reducing gas required to realise a given amount of metal from a given amount of ore. The test employs isothermal reduction of the sample (500 g), on a fixed bed at 950 °C introducing using reducing gases (40%CO + 60%N2) in the system. The results of RI test are presented in Table 6.

6.3. Reduction Disintegration Index (RDI)

This is test performed to measure the effect of reduction on the strength of raw material feed into a blast furnace. The generation of fines in percentage is called Reduction Degradation Index. Ores with lower RDI value are better. Higher the RDI value, accretion or agglomeration formation will more. There is no such standard method for manganese ores. The similar process is adopted for manganese ores and material was heated up to 550 °C for 30 min and gas composition (30:70 - CO:N2) and flow was kept similar as for iron ore testing. Comparative results obtained for BREX, Mn ores and other material is presented in Table 7.

- The RDI value for extruded briquettes is observed within the range observed for manganese ore lumps

6.4. Softening-Melting Test

This test is carried out to estimate the softening and melting behaviour of extruded briquettes. In the test, the required quantity of the ore is packed in graphite crucible and mixture of reducing gas is passed from the bottom of the crucible with subsequent rise in temperature. The result of softening and melting test is presented in Fig. 9 and Table 8.

Softening melting analysis has been carried out under reducing atmosphere with CO:70% and CO2:30% with constant gas flow rate of 2 lpm. Coke is added to the system on the top of the charge. Sized briquette fractions were put in graphite crucible which is heated from room temperature to 1400 °C to study the reduction and melting behaviour of manganese bearing charge. Initially, samples were heated with nitrogen till 350 °C followed by introduction of reducing gases till the experimental temperature. During initial period of heating, more of the bound and pore moisture is lost, subsequently there will be thermal decomposition of clay hydroxides minerals like gibbsite to alumina and water of hydration.

With introduction of reducing gases there will be reduction of iron bearing oxides mainly hematite into lower oxidation states. Phase transformation of manganese at a pO2 10⁻⁸ atm transforms from Mn(IV) state to Mn(III) state. As with transformation of pyrolusite to bixbyte, there is volume expansion which leads to formation of fines and leads to lower sticky mass between a temperature region of 500–600 °C, (Fig. 9) in the region I. Another reason which might be possible is that the fines generated lead to block the gas flow from the system.

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### Table 5
Test results of decrepitation index (DI).

<table>
<thead>
<tr>
<th>Sl. no.</th>
<th>Sample</th>
<th>BREX value</th>
<th>Mn Ores</th>
<th>Iron ore</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Temp. 700 °C; 30 min.</td>
<td>No fines</td>
<td>2–6%</td>
<td>15–20%</td>
</tr>
</tbody>
</table>

### Table 6
Test results of RDI.

<table>
<thead>
<tr>
<th>Sl. no.</th>
<th>Sample</th>
<th>BREX Value</th>
<th>Mn Ores</th>
<th>Iron ore Pellets &amp; Sinter</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Temp. 700 °C; 30 min.</td>
<td>~75.016%</td>
<td>57–85%</td>
<td>Sinter – 60–70% Pellet – 50–60%</td>
</tr>
</tbody>
</table>

MnO₂ - 500 °C - Mn₂O₃ - 900 °C - Mn₃O₄ - 1700 °C - MnO - 1700 °C – Mn.


### Table 7
Test results of RDI.

<table>
<thead>
<tr>
<th>Sl. no.</th>
<th>BREX value</th>
<th>Mn Ores</th>
<th>Iron ore Pellets &amp; Sinter</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>17%</td>
<td>14.40–28.20%</td>
<td>Iron ore - 15–25% Pellet - 1.1–3% Sinter - 20–30%</td>
</tr>
</tbody>
</table>


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path and hence increase in pressure drop across the bed. In the region II, further reduction of manganese oxides takes place at a temperature range of 700–850 °C. Further, there is softening of material due to formation of CaO-Al₂O₃-SiO₂-Fe(T) slag system which melts at lower temperatures and leads to the increase in pressure drop across the bed. A similar phenomenon is also observed in the region III. As the temperature of the bed reaches above 930 °C, there is a sharp increase in the pressure drop of 10 mm WC. Secondly, during the test there is a constant load of 0.5 kg applied from the top.

For the regions I–III, with generation of particles and constant load there is a positive increase in pressure drop. This attribute can be explained with a decrease in mean free path for the gas to flow and velocity decrease which leads to the increase in pressure drop across the bed. A similar phenomenon is also observed in the region III. As the temperature of the bed reaches above 930 °C, there is a sharp increase in the pressure drop of 10 mm WC. Secondly, during the test there is a constant load of 0.5 kg applied from the top.

### 6.5. Heating microscope

The heating microscope is completely automatic test and can simulate industrial heating treatments up to 1750 °C on the specimen, with heating rates up to 80 °C/min. or even instantaneous heating. This microscope simulates the following characteristic temperatures (Sintering Beginning, Softening, Sphere, Half Sphere, and Melting). Sintering beginning (sintering temperature): The temperature at which the sample has reached a dimensional variation corresponding to a defined percentage with reference to the first image acquired, considered as 100%. Softening temperature: The temperature at which the rounding of the corners of the sample and the smoothing of the walls of the sample occurred. Sphere temperature: The temperature at which the height of the sample is equal to the width of the base according to the standards or at which the diameter is the double of the ray of a hypothetical circumference. Half Sphere temperature: The temperature at which the height of the sample is half the width of the base. Melting temperature: The temperature at which the height of the sample shrinks to under a third of the base and the sample is completely liquefied. The results are presented in Table 9 and Fig. 10. A comparison of properties of novel extruded briquettes vis-a-vis conventional feed to the submerged arc furnace i.e., manganese natural ore/briquettes is presented in Table 10.

### 6.6. Plant trials in submerged arc furnace (SAF)

Based on the comparative physical and metallurgical properties of the briquettes, a trial campaign was undertaken to understand the furnace behaviour using secondary feed input material i.e. extruded briquettes. The trials were carried out in batch mode to gain the confidence. The overall furnace performance of furnace w.r.t. manganese metal and MnO in the slag and basicity of the system is presented in Fig. 11. It can be observed that the furnace operated smoothly, with a constant and uniform current load during briquette use. The results show that the trial program of smelting with the use of extruded briquettes in batch charge of an industrial submerged arc furnace was successful, demonstrating the high efficiency of extruded briquettes in the process of ferromanganese smelting.
Table 10
Comparison of novel agglomerate (BREX) properties with ore.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Effect</th>
<th>Mn ore</th>
<th>Measured Value for BREX</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tumbler Index (TI) % + 6.3 mm fraction</td>
<td>Generation of fines while handling, transportation and feeding to furnace</td>
<td>70–83%</td>
<td>41.60%</td>
<td>Criteria: 70%- TI; &lt;5%- AI To understand why the value is low. Standardization of process is required</td>
</tr>
<tr>
<td>Abrasion Index (AI) % – 0.5 mm</td>
<td>Characterize material susceptibility to: Breakage Degradation</td>
<td>Not available</td>
<td>(+10 mm) – 79.5% (+5mm) – 83.5%</td>
<td>The shatter test shows a good strength of BREX. It indicates that it can withstand the breakage due to multiple handling</td>
</tr>
<tr>
<td>Shatter Index</td>
<td>Characterize material susceptibility to: Breakage Degradation</td>
<td>Not available</td>
<td>114.89 kgf (Axial); 55.3 kgf (Horizontal)</td>
<td>Conventional Briquettes - 90 – 100: Par with conventional briquettes Good feed</td>
</tr>
<tr>
<td>Cold Compressive Strength (CCS) Porosity</td>
<td>Withstand load without breaking It affects the degree of preproduction of the ores. The water content and cold strength also affect the burden properties</td>
<td>Mn ore – 6 to 43% (captive mines) MOIL Ores: 23% Gabonese ore: 38% Comilog ores: 30–48%, BHP: 7–10%</td>
<td>40.01% Good feed</td>
<td></td>
</tr>
<tr>
<td>Decrepitation Index (DI)</td>
<td>Effect thermal degradation fine generation (thermally processed ores, such as sinter and pellets, do not decrepitate) DI quantifies the resistance of lump ore to thermal degradation</td>
<td>DI vary between 2 and 6% 0 (loss in wt. Avg.9.27%)</td>
<td>A DI value of &lt;15, is desirable for lump iron ore</td>
<td></td>
</tr>
<tr>
<td>Reduction Disintegration Index (RDI)</td>
<td>Measure the effect of reduction on strength of raw material feed into blast furnace</td>
<td>Mn Ore - 14.40–28.20% Pellet - 1.1–3% Sinter – 20–30%</td>
<td>17% The RDI value for extruded briquettes is observed within the range observed for manganese ore lumps</td>
<td></td>
</tr>
<tr>
<td>Reducibility Index (RI)</td>
<td>Reducibility parameters illustrate the possibility for removal of combined from the ore. A high reducibility is desirable, since it decreases the amount of reducing gas required to realize a given amount of metal from a given amount of ore</td>
<td>Mn - 57–85% Sinter – 60–70% Pellet – 50–60%</td>
<td>75%</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 11. Furnace performance using briquettes.
7. Conclusions

- Fine and moist GCP sludge and ore fines can be efficiently agglomerated using novel stiff vacuum extrusion technology.
- These briquettes exhibit good mechanical property (strength) which ensures that they will remain intact during storage, transport, and handling, as well as when they are in a layer of other charge materials inside metallurgical furnaces before they are melted.
- Briquette exhibit lower DI (zero fines) and RDI value of 17% indicating the probability of generation of fines will be less while entering the heating zone of furnace. The reducibility index (RI) 75% is a comparable value.
- This study exhibits the possibility of using extruded briquette (Mn sludge and fine) in SAF in the form of a suitable agglomerate, for partial to complete replacement of lump ore.
- This technology will address the pertinent problem of sludge and fine utilisation of FAP plant.

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