Hot briquetting tests with mixtures of direct reduced iron (DRI) pellets and residues of the Midrex direct reduction process are conducted on a hydraulic piston press with a closed die. The trials seek to test the feasibility of recycling these residues by introducing them directly into hot briquetted iron (HBI). The inclusion of residues is possible if a high pressure of 350 MPa and a high briquetting temperature of 800 °C are applied. It is ascertained that up to 20 wt% of the HBI can consist of residues and still meet the quality requirements for the safe transportation to the steel plant if the mixture contains sufficient HBI screened fines and HBI classifier dust. Under such conditions, the HBI briquettes have a compressive strength of >300 MPa, an abrasion resistance of >80%, and, most importantly, an apparent density of >5 g cm⁻³. It is further shown that the hot briquetting of Midrex residues is also possible without DRI pellets so that they can be reused as educt in the Midrex direct reduction process.

1. Introduction

The production of hot briquetted iron (HBI) with the Midrex process results in a variety of ferrous residues, including sludge, screened iron oxide fines, and HBI fines as well as dusts. All residues have a high iron oxide content and consist partly of metallic iron. The study investigated the possibility of briquetting these residues with pellets of direct reduced iron (DRI) into HBI so as to utilize the residue raw material potential and to avoid the disposal or depositing of these residues. The advantage of such an approach when compared with the independent cold briquetting of the residues for reuse in the Midrex process is that no additional binder is required. The metallic iron contained in the briquette acts as a binder during hot briquetting. Another advantage is that no additional briquetting plant is needed. This article describes a series of laboratory experiments designed to test the feasibility of adding the residues during hot briquetting with a hydraulic piston press. Three possible approaches were considered: first, the residue mixture was preheated and added to the hot, directly reduced pellets. Next, the study investigated whether it is possible to add the residue mixture in a cold state to the hot, directly reduced iron pellets. And finally, the study examined whether or not it is possible to hot briquette the residue mixture directly without reduced iron pellets. The most important target values for the hot briquettes are the apparent density of the briquettes as well as their pressure and abrasion resistance.

2. Midrex Process and Hot Briquetting

The Midrex process represents a commercially available alternative to the conventional blast furnace process for the production of DRI as a potential product for the production of crude steel in steelworks. The Midrex process consists of a shaft furnace containing a packed bed of iron oxide pellets. Pellets are fed at the top and exit at the bottom as hot or cold DRI. Hot reducing gas (H₂ and CO) is fed countercurrently into the shaft and passes upward, reducing the iron oxide pellets (these consist mainly of hematite) to the metallic phase. During the direct reduction of the iron oxide pellets, oxygen is released while the pellets remain in a solid state. This creates a sponge-like structure with many pores and a large surface area. The apparent density of the directly reduced pellets is about 2 g cm⁻³ and the specific surface area is about 1 m² g⁻¹. The DRI tends to reoxidize exothermically when the heat is released already under ambient conditions. That is why spontaneous ignition at low temperatures is a problem during the transportation of DRI. To decrease the specific surface area and, thus, make DRI safe for transportation, hot briquetting is needed to attain passivation. HBI is produced by pressing the DRI at temperatures of at least 650 °C to form briquettes with an apparent density of at least 5 g cm⁻³. Industrially, a roller press is used for this purpose. The hot DRI as reduced pellets is forced by a vertical screw feeder into the nip between two counterrotating rollers with matched pockets. A specific linear pressing force of 150 kN cm⁻¹ is needed to attain the required apparent density in the briquettes.
3. Residues and Recycling Possibilities

A large number of mostly valuable residues are generated during the production of iron and steel in steel plants. This includes such processes as the preparation of the raw material during the shaft furnace process for the production of reduced iron. These residues are also generated in electric arc furnaces and basic oxygen furnaces as well as during the further processing of steel.\textsuperscript{[5–6]} These residual materials are present in the form of dust, slag, and sludge. A number of efforts are undertaken to process the residual materials and to reuse them for other purposes, to recover valuable materials, or to return them to the process after treatment.\textsuperscript{[5,7–9]} This article deals with the treatment options for residues created especially during the Midrex direct reduction process. The factors that determine the mass of iron oxide pellets required to produce 1 ton of DRI in the Midrex process include the loss of oxygen through the reduction, the addition of carbon, oxide fines losses from screening, the processing of the iron oxide pellets in the reduction furnace, and the screening of DRI/HBI products. Figure 1 is a process diagram of the Midrex process which shows the generated residuals. Typical fines losses from the reduction furnace to the off-gas are 1–1.5 wt%. The losses due to oxide screening can be up to 10 wt% depending on the grain size. And there are also losses during the briquetting of DRI in the form of dust and screened fines. HBI briquettes leave the roller press as a continuous string and therefore have to be broken into individual briquettes, creating fines. The losses from HBI crushing and screening amount 1–3 wt%.\textsuperscript{[10]} Remet is material which is created during furnace start-ups and shutdowns which is not fully metalized or produced when a briquetting machine is first being started up. The remet is recycled to the furnace, and the remet fines are the fines which are screened from this remet before it is reused. Some of the residual materials (especially screened oxide fines and HBI screened fines) can be returned to the process without treatment or sold elsewhere. That is why the proportion of these materials is lower than the data for the quantities produced. On the basis of practical experience, it can be assumed that the amount of residues to be reprocessed will be in the range of 3–6 wt% of the input material and consists of approximately 40 wt% dried sludge, 30 wt% oxide screened fines, 15 wt% HBI screened fines, 5 wt% HBI classifier dust, 5 wt% remet screened fines, and 5 wt% process classifier dust.

Landfilling or the disposal of the products should be avoided to use the raw material potential of these residues. So far, briquetting with binding agents has been considered and practiced for the recycling of the residual materials. The residual materials are briquetted with a binder in a roller press and used together with iron oxide pellets as feed material for the Midrex process. The briquettes are reduced in the reduction shaft and then processed into HBI together with the directly reduced pellets. This article presents an alternative method. The aim is to integrate the residues directly into the hot briquetting of DRI. As the accumulation rate of residues is low and the material is partly reduced, the fundamental briquette quality should not be negatively influenced with regard to the chemical composition. Furthermore, the use of an additional binder (and, thus, additional gangue) can be dispensed with because the metallic iron of the DRI acts

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure1.png}
\caption{Schematic representation of the Midrex direct reduction process and the resulting residual materials.}
\end{figure}
as a binder. This would mean that the purchase and operation of an additional briquetting plant could be dispensed with and the existing roller press could be used for hot briquetting. Two procedures are considered for adding the residues to DRI pellets during hot briquetting. The first procedure feeds hot reduced pellets and dry, cold residual materials into the roller gap of the roller press for combined hot briquetting. The second procedure feeds hot reduced pellets and dry residual materials preheated to the press temperature into the roller gap of the roller press for combined hot briquetting.

Many facilities for the direct reduction of iron oxide pellets have or had either no options or only limited means for residue recycling. These materials frequently lie in the open on a pile. For the residue stock, hot briquetting without sponge iron pellets could be of interest to save binder costs. That is why tests for such a procedure were also included in the test program. Ideally, the briquettes should be of small size because they are to be fed into the Midrex shaft furnace together with the iron oxide pellets. If their share of the overall shaft furnace feed is not too high, inclusion into HBI ought to be practicable.

4. Binding Mechanisms

During the hot briquetting of DRI, the necessary strength is mainly provided by the solid bridges of alpha iron between the particles. These can result from sintering processes and melt flow. The mobility of atoms and molecules is stimulated by the high temperatures during hot briquetting (2/3 of the melting temperature of the solid are necessary); and due to diffusion processes, a solid transport to the contact points of the particles occurs which results in the formation of sinter bridges. The process can be intensified by increasing the temperature and increasing the particle contact. A further increase in temperature leads to melting phenomena. This can be achieved not only through heating, but also friction and plastic deformation generate heat and, thus, contribute to partial melting. Initially, liquid bridges are formed by capillary forces. As they cool, the liquid bridges transform into solid bridges. In addition, such molecular forces as van der Waals forces contribute to the binding. Due to the increased pressure plasticity of the reduced iron at high temperatures and under high pressure, there is a strong approximation and larger contact surfaces are created between the particles. These forces can be very high at extremely small distances between the adhesion partners. The forces decrease as the distance between the particles increases – with the maximum van der Waals interaction being at a distance range of 100 nm. \[11\]

The presence of sponge iron fines improves the briquetting potential of sponge iron pellets as tiny particles fill the voids in the pellets’ bulk during briquetting. \[12\] The residues should, therefore, have a high content of metallic iron to avoid a negative influence on the quality of the briquettes in mechanical and chemical terms.

5. Requirements on HBI

The needed quality characteristics of HBI result mainly from the requirements for the safe transportation of the briquettes and the requirements of the steel plants for further processing. For the shipping and handling of the briquettes, a high mechanical strength is needed (less fines generation; fines and chips <5 wt%; a minimum compressive strength of 50 MPa; an abrasion resistance of \(R_{10}(300)\) of >80%) along with an apparent density exceeding 5 g cm\(^{-3}\) to prevent reoxidation and spontaneous self-ignition. For use in electric arc furnace (EAF) steel plants, an apparent density of more than 5 g cm\(^{-3}\) is needed to also permit the rapid sinking into the furnace slag layer. In addition, the chemical properties are of particular importance for any further processing as they influence the melting properties. A high degree of metallization is important while the amount of gangue should be low. Unwanted accompanying elements must be removed in the steel plants with a high energy input. \[10\] [11]

6. Experimental Section

6.1. Test Material

Material data of the residues and DRI pellets which were used in the tests are shown in Table 1–3. The chemical analyses were performed according to ISO 9516-1 and ISO 16878 (Fe-total). The sponge iron pellets (reference pellets) which were used in these tests belong to the lower end of the quality spectrum with an Fe-total of 84 wt% and a rank of metallization of 93.5%. The rank of metallization describes the ratio of metallic iron to the total iron content. This mixture was tested to ascertain the possibility of using combined hot briquetting with Midrex residues for lower grade sponge iron pellets. Characteristically, the size of the reference pellets is between 4 and 16 mm. The proportion of broken pellets is very low. The various residual materials are characterized by different degrees of reduction.

Table 1. Chemical composition of the residues and the DRI pellets.

| Component            | \(\text{Fe}_{\text{tot}}\) [wt\%] | \(\text{Fe}_2\text{O}_3\) [wt\%] | \(\text{FeO}\) [wt\%] | \(\text{Fe}_{\text{eq}}\) [wt\%] | C [wt\%] | \(\text{SiO}_2\) [wt\%] | \(\text{CaO}\) [wt\%] | \(\text{Al}_2\text{O}_3\) [wt\%] | \(\text{MgO}\) [wt\%] | \(\text{K}_2\text{O}\) [wt\%] | \(\text{TiO}_2\) [wt\%] | P [wt\%] |
|----------------------|-------------------------------|-------------------------------|---------------------|-------------------------------|----|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|-------|
| Oxide fines          | 67.20                         | 96.10                         | 0.00                | 0.00                          | 0.03| 1.59                 | 0.91                 | 0.53                 | 0.141                | 0.014                | 0.075                | 0.029 |
| Dried sludge         | 74.00                         | 63.60                         | 0.00                | 29.53                         | 2.01| 2.18                 | 1.03                 | 0.85                 | 0.244                | 0.019                | 0.103                | 0.061 |
| Process classifier dust | 70.08                        | 79.40                         | 0.00                | 14.51                         | 0.26| 3.07                 | 1.00                 | 1.01                 | 0.114                | 0.090                | 0.096                | 0.030 |
| HBI classifier dust  | 83.94                         | 34.51                         | 2.10                | 58.17                         | 0.93| 2.06                 | 1.28                 | 0.60                 | 0.183                | 0.014                | 0.090                | 0.063 |
| HBI screened fines   | 88.71                         | 17.79                         | 1.73                | 74.92                         | 1.02| 2.12                 | 1.03                 | 0.54                 | 0.250                | 0.020                | 0.097                | 0.030 |
| Remet fines          | 84.87                         | 30.94                         | 4.00                | 59.81                         | 0.40| 2.21                 | 1.06                 | 0.82                 | 0.200                | 0.023                | 0.106                | 0.032 |
| DRI pellets          | 84.01                         | –                              | –                   | 78.55                         | 4.01| –                   | –                   | –                   | –                    | –                   | –                   | –     |
The median particle size $d_{50}$ and the mean particle size $d_m$ of the residues are within the range of 0.1 and 2 mm.

For the briquetting tests, a mixture consisting of residual materials is used in every experiment. The M1 mixture is used for the first and the second part of the combined pressing tests applied to the residual material mixture with directly reduced pellets. During the third part of the tests, the residual material mixture is pressed without DRI pellets so as to test the suitability of three different mixtures for briquetting (mixture M2, M3, M4). Table 4 shows the composition of the residue mixtures that were used in these tests. Table 5 shows the material characteristics of the DRI pellets and the residue mixtures.

If the use of these materials improves the briquetting success, then it should also be useful to briquette these materials. Mixtures M3 and M4 were examined to investigate the influence of the mixture composition. M1 is similar to M2, with the difference being that HBI screened fines are not used because M1 is added to DRI.

Table 2. Grain size of the residues.

| Unit | Oxide fines | Dried sludge | Process classifier dust | HBI (screened fines) | HBI (classifier dust) | Remet fines |
|------|-------------|--------------|-------------------------|----------------------|-----------------------|-------------|
| $d_0$ (10 mm) | wt% | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| $d_0$ (6.3 mm) | wt% | 99.6 | 100.0 | 99.5 | 100.0 | 98.6 |
| $d_0$ (4 mm) | wt% | 97.4 | 99.9 | 99.3 | 99.8 | 98.4 |
| $d_0$ (2 mm) | wt% | 86.1 | 99.0 | 92.5 | 48.1 | 85.5 |
| $d_0$ (1 mm) | wt% | 71.4 | 97.2 | 55.2 | 28.1 | 21.5 |
| $d_0$ (0.63 mm) | wt% | 63.5 | 96.1 | 16.3 | 19.5 | 18.6 |
| $d_0$ (0.5 mm) | wt% | 60.9 | 95.1 | 3.0 | 16.8 | 0.9 |
| $d_0$ (0.315 mm) | wt% | 60.8 | 94.5 | 1.2 | 16.7 | 0.2 |
| $d_0$ (0.125 mm) | wt% | 44.4 | 74.1 | 0.1 | 5.3 | 0.1 |
| $d_0$ (0.09 mm) | wt% | 34.7 | 61.9 | 0.0 | 4.1 | 0.0 |
| $d_0$ (0.063 mm) | wt% | 24.8 | 43.8 | 0.0 | 2.9 | 0.0 |
| $d_0$ (0.03 mm) | wt% | 1.2 | 1.67 | 0.2 | 0.9 | 0.1 |
| $d_0$ (0.01 mm) | wt% | 0.32 | 0.33 | 0.32 | 0.32 | 0.32 |
| $d_0$ | mm | 0.764 | 0.140 | 1.122 | 1.865 | 1.416 |
| $d_0$ | mm | 0.190 | 0.072 | 0.951 | 2.074 | 1.445 |

Table 3. Grain size of the DRI pellets.

| Unit | DRI pellets |
|------|-------------|
| $d_0$ (18 mm) | wt% | 100.0 |
| $d_0$ (16 mm) | wt% | 98.92 |
| $d_0$ (14 mm) | wt% | 86.53 |
| $d_0$ (12.5 mm) | wt% | 71.15 |
| $d_0$ (10 mm) | wt% | 29.00 |
| $d_0$ (8 mm) | wt% | 10.14 |
| $d_0$ (6.3 mm) | wt% | 6.38 |
| $d_0$ (4 mm) | wt% | 1.34 |
| $d_0$ (2 mm) | wt% | 0.33 |
| $d_0$ (1 mm) | wt% | 0.32 |
| $d_0$ | mm | 10.844 |
| $d_0$ | mm | 11.246 |

Table 4. Composition of the residue material mixtures used in the briquetting tests.

| Fe-carrier | Unit | M1 | M2 | M3 | M4 |
|-----------|------|----|----|----|----|
| Oxide fines | wt% | 30 | 30 | 30 | 0 |
| Dried sludge | wt% | 55 | 40 | 60 | 60 |
| Process classifier dust | wt% | 2.5 | 5 | 0 | 5 |
| HBI screened fines | wt% | 0 | 15 | 10 | 15 |
| HBI classifier dust | wt% | 10 | 5 | 0 | 10 |
| Remet screened fines | wt% | 2.5 | 5 | 0 | 10 |

Table 5. Material characteristics of the DRI pellets and the residue mixtures.

| Parameter | Unit | DRI pellets | M1 | M2 | M3 | M4 |
|-----------|------|-------------|----|----|----|----|
| Mean particle size | mm | 10.844 | 0.542 | 1.12 | 0.703 | 1.104 |
| Median particle size | mm | 11.246 | 0.15 | 0.929 | 0.141 | 0.169 |
| Bulk density | g cm$^{-3}$ | 1.336 | 2.070 | 2.013 | 1.988 | 1.753 |
| True density | g cm$^{-3}$ | 7.023 | 5.152 | 5.515 | 5.214 | 5.445 |
| Apparent density | g cm$^{-3}$ | 2.835 | – | – | – | – |
| Porosity | % | 59.63 | – | – | – | – |
| Fe$_{total}$ | wt% | 84.01 | 73.60 | 75.01 | 73.43 | 78.09 |
| Degree of metallization | % | 93.50 | 34.77 | 39.56 | 34.33 | 53.12 |
| Metallic Fe | wt% | 78.55 | 25.59 | 29.67 | 25.21 | 34.33 |
| Carbon | wt% | 4.01 | 1.22 | 1.05 | 1.32 | 1.51 |
6.2. Test Program and Test Procedure

The briquetting tests were conducted with a hydraulic piston press that has a closed die. Figure 2 is a schematic representation of the test sequence depicting the preheating of the samples and the pressing of the briquettes in a hydraulic piston press. The hydraulic piston press was refitted for hot briquetting. All the tests used round briquettes (diameter: $\varnothing = 50$ mm, height $h$ corresponding to the compression ratio) to determine the briquette quality. Briquetting tests with the hydraulic piston press are a cost saving method to determine the effects of the relevant influences under a broad variety of conditions. The quality of the test results is transferable to the briquetting process with roller briquetting machines and is, thus, relevant for commercial briquetting.

The first series (hot DRI pellets and hot residues) of the hot briquetting tests were conducted with preheated pellets and a mixture of preheated Midrex residues (M1). The tests were conducted as follows (see Figure 2): A) The weighed pellet sample and M1 mixture are filled into conical steel boxes (approximately 10 cm high and wide) which have a lower and an upper lid (airtight to avoid reoxidation). The boxes are inserted into the batch furnace which was preheated to 900 °C and kept there for a residence time of 20–30 min in the furnace. These special boxes are closed during the heating (upper and lower lid). The temperature in the oven is measured, but not the temperature of the sample. Experience has shown that the temperature of the sample corresponds to the temperature of the furnace chamber after a residence time of 20–30 min in the furnace. B) The boxes with DRI are manually removed from the oven with oven tongs and placed on the die of the hydraulic piston press. The die is filled with pellets by removing the lower lid of the box. C) The pellet box is removed from the die and placed with the hot M1 mixture on the die. The die is filled with the preheated particles of the M1 mixture after the lower lid of the box is removed. The fine particles trickle into the voids between the pellets. This is supported by slightly stirring the mixture manually with a metal rod. D) Once the M1 box has been removed from the die, the pressing process starts immediately. The applied briquetting parameters are a pressure of $p = 350$ MPa and a residence time of $t = 3$ s under maximum pressure. Pressure is set at a high level which can, though, be attained by roller briquetters. This is done to compensate for the expected negative effect of the residues on the briquette quality. No vibration is applied during compaction. The sample material experiences a temperature loss of about 70–80 °C (hot–hot) as it is moved from the furnace to the die. The press itself is not heated. The die is cooled with cooling water to prevent the deformation of the die and to reduce the material’s adhesiveness. Consequently, the effective press temperature for the tests is approximately 800 °C. The next step involves extruding the hot briquettes from the die and putting the briquettes on a grid for air cooling.

The tests of series 1 (hot DRI pellets and hot residues) were conducted with varying amounts of M1 added to the mixture consisting of pellets and M1. More precisely, 0, 3, 5, 10, 20, 30, 40, and 50 wt% of the tested mixtures consisted of M1. During the second test series (hot DRI pellets and cold residues), the M1 mixture is added in a cold state (ambient temperature) to the hot pellets. Otherwise, the test procedure is the same as described earlier. The second procedure is less expensive because the residues are not heated separately. The effective press temperature of the DRI is also reduced through the addition of the cold mixture (hot–cold). But the conditions for hot briquetting worsen a bit due to the cooling effect of the residue material mixture. The tests of series 2 (hot DRI pellets and cold residues) were conducted with the M1 mixture equaling 0, 3, 5, and 10 wt% of the pellet and M1 mixture. During the third test series (briquetting of residue mixture without pellets), the M2, M3, and M4 mixtures are preheated and briquetted similar to the test procedure described for series 1, but without DRI pellets.

A total of six to nine briquettes are produced per test parameter combination. The subsequent briquette quality and process parameters were then determined. The apparent density of the briquettes $\rho_{app,b}$ was calculated from the mass and volume of the briquettes. The compressive strength was determined according to TGL 9491 (abbreviation of a former technical standard of the German Democratic Republic). The briquettes were tested with the universal testing machine UH-500kNA made by the Shimadzu Corporation. Pressure was applied by two compression pistons on the briquettes until the briquettes broke. The maximum compressive stress in MPa is an indicator of the compressive strength $\sigma_P$. The abrasion resistance was determined according to DIN 51717 by the defined stress which is imposed on the briquettes inside a drum. The drum has a diameter of

Figure 2. Schematic illustration of the test sequence with regard to preheating the samples in a batch furnace and pressing the briquettes with the hydraulic piston press: 1, furnace; 2, cylindrical die with a 5 cm inner diameter; 3, moving piston; 4, fixed piston; 5, DRI in a special conical box with a lower and an upper lid; 6, residual mixture M1 in a special conical box with a lower and an upper lid; 7, water cooling; 8, briquette (HBI).
50 cm, a length of 50 cm, and a rotation speed of 25 revolutions per minute. The drum has four blades on the inside that are 8 cm long and set at 90°. Three briquettes were assessed in each test run; they had a total weighed mass of about 500 g. The R10(100), R10(300), and R30(300) values for abrasion resistance were determined. For example, R10(100) describes the residue mass on the 10 mm sieve after a sample of three briquettes had trembled for 100 rotations inside the test drum in relation to the total mass of inserted briquettes. The elastic springback of briquettes (height expansion) is calculated as follows

$$\Delta h = \frac{h - h_{p,\text{max}}}{h_{p,\text{max}}} \quad (1)$$

where \( h \) is the briquette height after the pressure release, and \( h_{p,\text{max}} \) is the briquette height at maximum pressure. The compression ratio \( K \) is the ratio of the dumping height in the die (\( h_{\text{b,d}} \)) to the height of the briquettes at maximum pressure (\( h_{p,\text{max}} \)). The densification energy \( W_v \) of the briquetting process is the integral of the pressure-displacement diagram. The densification ratio \( \Phi_v \) is calculated from the apparent density of the briquettes \( (\rho_{\text{app},b}) \) and the bulk density of the briquetting mixture \( (\rho_{\text{bulk},bm}) \).

$$\Phi_v = \frac{\rho_{\text{app},b}}{\rho_{\text{bulk},bm}} \quad (2)$$

The densification efficiency \( \eta \) is calculated from the densification ratio \( \Phi_v \) and the densification energy \( W_v \).

$$\eta = \frac{\Phi_v}{W_v} \quad (3)$$

To determine the elastic springback of the briquettes, the height of the hot briquettes was measured immediately after they had been expelled from the die. All other briquette quality parameters were measured when the briquettes had reached the ambient temperature. The obtained results were then considered for the briquetter design, including the roll diameter, the feeding system for the briquetting material, the shape of the ring or segment molds, and the drive system.[14]

### 7. Results and Discussion

#### 7.1. Combined Briquetting of Residues and DRI Pellets

The results for test series 1 (hot DRI pellets and hot residues) and test series 2 (hot DRI pellets and cold residues) are compiled in Table 6 and 7. Figure 3 shows the briquettes (hot DRI pellets and hot residues) with different proportions of the M1. The results of the tests with 0 wt% M1 (100 wt% DRI pellets) represent the optimum that can be achieved for the strength parameters and other characteristic values.

All briquettes having a mixture consisting of DRI pellets and the residue mixture M1 had a good shape (Figure 3) irrespective of the proportion of M1 mixture in the briquette. At a low M1 content, the grain boundaries of the pellets are still visible on the briquette surface, and shrinking cracks run along the grain boundaries. As the M1 content increases, the pellet grain boundaries disappear more and more while shrinking cracks now frequently pass right through the pellets. And the cracks are wider.

| Amount of M1 [wt%] | Apparent density [g cm\(^{-3}\)] | Compressive strength [MPa] | Abrasion resistance R10(100) [%] | Abrasion resistance R10(300) [%] | Abrasion resistance R30(100) [%] |
|-------------------|----------------------------------|-----------------------------|--------------------------------|-------------------------------|-------------------------------|
| 0                 | 5.930                            | 704.34                      | 99.4                           | 98.1                          | 98.1                          |
| 3                 | 5.447                            | 539.8                       | 94.8                           | 92.8                          | 55.4                          |
| 5                 | 5.388                            | 612.5                       | 88.4                           | 84.3                          | 35.0                          |
| 10                | 5.313                            | 557.3                       | 86.0                           | 82.4                          | 18.5                          |
| 20                | 5.285                            | 510.5                       | 83.7                           | 79.8                          | 22.4                          |
| 30                | 5.139                            | 442.6                       | 83.3                           | 78.2                          | 54.8                          |
| 40                | 4.932                            | 446.9                       | 79.9                           | 72.3                          | 26.8                          |
| 50                | 4.802                            | 382.3                       | 77.4                           | 71.6                          | 15.3                          |

| Amount of M1 [wt%] | Apparent density [g cm\(^{-3}\)] | Compressive strength [MPa] | Abrasion resistance R10(100) [%] | Abrasion resistance R10(300) [%] | Abrasion resistance R30(100) [%] |
|-------------------|----------------------------------|-----------------------------|--------------------------------|-------------------------------|-------------------------------|
| Hot DRI pellets and hot residues procedure | | | | | |
| 3                 | 5.384                            | 571.1                       | 93.9                           | 86.6                          | 46.4                          |
| 5                 | 5.310                            | 549.5                       | 90.4                           | 89.0                          | 25.0                          |
| 10                | 5.136                            | 520.0                       | 87.1                           | 80.2                          | 12.5                          |

But more importantly, it can be seen that the particles of the M1 materials are incorporated very well into the briquette matrix irrespective of their proportion. As far as the apparent density, compressive strength, and abrasion resistance are concerned (these are the main quality parameters of HBI briquettes), there is, as was to be expected, a clear negative influence of the M1 content (Figure 4–6). In Figure 4, the error bars represent the standard deviation for six briquettes. This applies to both the hot–hot and the hot–cold procedures. Obviously, the lower metal content of the residues, which means lower plasticity, hinders densification as well as the formation of Fe-metal bridges, therefore downgrading the mechanical stability of the briquettes. The influence of the actual procedure (hot–hot/hot–cold) is fairly marginal up to a M1 content of 10 wt%. This is a good test result because it means that it is not necessary to heat the residues/residue mixtures.

The tests showed that it is possible to produce high-quality briquettes that meet the transportation and safety requirements of pellet/M1 mixtures under the process conditions which were applied in these tests. This is clearly valid for the hot–hot procedure and is also very likely so for the hot–cold method. The compressive strength of the briquettes was never under a value of 300 MPa in all the tests – this is more than sufficient. Up to an M1 content of 20 wt%, the abrasion resistance R10(300) is ≥80% which permits transportation over a long distance, the storage in a pile, and the handling without major briquette disintegration. Within that residue content range, the apparent density does not fall below a level of 5 g cm\(^{-3}\). The risk of self-ignition can, thus, be ruled out. The briquetting tests confirm a direct correlation between the compression ratio and the apparent density. The values of both parameters fall with an increased M1 content.

No clear trend was determined for the elastic springback, but it can be seen that the height of the briquettes at maximum pressing pressure (\( h_{p,\text{max}} \)) and the height of the briquettes after pressure release (\( h \)) increase both with an increasing proportion of.
M1. This is linked to a lower maximum pressure at the contact points of the pellets due to the residue particles found between the pellets. In addition, this process parameter is also linked to the shrinkage of the hot briquettes once they are removed from the hot press die and placed on a grid to cool in the air. The higher variance of that parameter may have something to do with the time it takes to determine the height of the rapidly cooling hot briquettes. This could have been due to the tester’s unintentional variation in the handling of the material.

Both the compression ratio and the elastic springback are relevant for the design of the pillow shape molds of the roll briquetter. They indicate that with the addition of the M1 material to the pellets, the mold can be deeper. This supports a pressure build-up in the roller gap and makes the briquetting process more stable.

Figure 3 shows the selected pressure-displacement diagrams for the hot–hot procedure at an M1 content of 0, 10, 20, and 30 wt%. As all of these pressure-displacement diagrams indicate, the densification energy generally declines as the M1 content increases. This is due to the changing plastic deformation required to attain the preset maximum pressure (350 MPa). Within the M1 content range >10 wt%, the plasticity of the pellets gradually loses its dominating influence. As a consequence, the briquette height rises under maximum pressure. The decline of the densification ratio is a further confirmation of the residues-induced plasticity loss of the briquetting material.

Table 7. Results of the briquetting tests using the pellets/M1 mixtures (part 2).

| Amount of M1 [wt%] | Briquette height at maximum pressure [mm] | Briquette height after pressure release [mm] | Elastic spring-back [%] | Compression ratio [−] | Densification energy [kN m] | Densification ratio [−] | Densification efficiency [(kN m)−1] |
|-------------------|------------------------------------------|---------------------------------------------|------------------------|----------------------|-----------------------------|------------------------|---------------------------------|
| Hot DRI pellets and hot residues procedure |
| 0                 | 13.7                                     | 15.4                                       | 12.3                   | 4.82                 | 4.08                        | 4.44                   | 1.09                           |
| 3                 | 15.0                                     | 16.7                                       | 11.6                   | 4.30                 | 4.55                        | 3.96                   | 0.87                           |
| 5                 | 14.8                                     | 16.8                                       | 13.5                   | 4.15                 | 4.28                        | 3.80                   | 0.89                           |
| 10                | 14.4                                     | 16.7                                       | 8.7                    | 3.71                 | 4.18                        | 3.62                   | 0.86                           |
| 20                | 15.6                                     | 16.7                                       | 8.6                    | 3.57                 | 4.03                        | 3.30                   | 0.82                           |
| 30                | 15.8                                     | 17.4                                       | 9.7                    | 3.17                 | 3.75                        | 2.96                   | 0.79                           |
| 40                | 16.3                                     | 18.1                                       | 11.1                   | 2.71                 | 3.88                        | 2.64                   | 0.68                           |
| 50                | 16.9                                     | 18.1                                       | 7.3                    | 2.36                 | 4.01                        | 2.40                   | 0.60                           |
| Hot DRI pellets and cold residues procedure |
| 3                 | 15.3                                     | 16.7                                       | 9.3                    | 4.07                 | 4.15                        | 4.03                   | 0.97                           |
| 5                 | 15.5                                     | 16.8                                       | 8.1                    | 4.22                 | 4.31                        | 3.86                   | 0.90                           |
| 10                | 15.7                                     | 17.3                                       | 10.4                   | 3.56                 | 4.14                        | 3.63                   | 0.88                           |

Figure 4. The apparent density of briquettes made from DRI pellets and the M1 mixture (the error bars represent the standard deviation for six briquettes); target value apparent density >5 g cm−3.

Figure 3. Briquettes consisting of DRI pellets and M1 mixture (series 1: Hot DRI pellets and hot residues, $p = 350$ MPa, $\vartheta_p = 800$ °C).

0 % M1

10 % M1

30 % M1

50 % M1

50 mm

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All in all, the hot briquetting tests confirm the possibility of including M1 residual materials in HBI up to a content of approximately 20 wt%. The lower energy costs of the compactor are another ancillary plus point. Normally, this is also reflected in the densification efficiency diagrams. This is, though, not the case here due to the decreasing densification ratio. The densification efficiency can be seen as the cost–benefit ratio that evaluates the briquetting process while taking the bulk density of the initial briquetting mixture into account. Consequently, this parameter can be used to establish a ranking when comparing the briquetting properties of different sponge iron pellets and different residue mixtures.

The test results show that it is possible to use residual materials in the combined briquetting process with regard to the mechanical properties and safe transportation. However, it is still necessary to investigate the influence on the chemical properties which are relevant for steel conversion. The chemical properties of HBI depend on the chemical properties of the DRI pellets as well as on the proportion and the chemical composition of the residues. During the briquetting process itself, the chemical properties are not influenced. Consequently, the chemical properties can be affected and predicted by the selective use of the already prereduced material (adjustment of the mixture composition of the residues) and the amount used. As far as the chemical properties are concerned, it is not possible to add 20 wt% of residual materials. However, the amount of the metallurgical residual materials that are produced is normally much lower. When using DRI pellets with a total iron content of 91 wt%, for example, the total iron content is reduced to 90.13 wt% by adding 5 wt% of the M1 mixture.

### 7.2. Briquetting of Residues without DRI Pellets

Briquette quality data and process parameters are shown in Table 8 for hot briquetting tests without pellets. The selected mixture composition is linked to different accruing scenarios in Midrex facilities. The bigger HBI screened fines particles act as supportive grains in the formation of a strong briquette compound. Therefore, this component should not be left out.

The compressive strength of the briquettes is very good again. Unfortunately, this is not the case for the abrasion resistance. Only the M4 mixture meets the briquette quality parameter that is needed for the transportation requirement. The safe transportation over a longer distance is generally questionable due to the low apparent densities ranging between 4.5 and 4.9 g cm$^{-3}$. The risk of self-ignition should, though, be lower due to the lower content of pure metallic Fe in the briquette mixture. It seems reasonable to consider the densification efficiency for the three mixtures that had been tested. Mixture 4 achieves the highest densification efficiency with 3.21 (kN m)$^{-1}$, followed by mixture 2 (2.93 (kN m)$^{-1}$) and mixture 3 (2.91 (kN m)$^{-1}$). Ranking is clearly related to the content of HBI components in the mixture. The more HBI is in the mixture, the better the densification efficiency. This test result once again shows the advantage of
including as many HBI components as possible in the mixture to assure the briquette quality and briquetting efficiency. Compared with the tests with pellets, the values for the compression ratio and densification energy are lower, whereas the densification ratio and densification efficiency are higher. This can be explained by the much lower bulk density of the residue mixtures compared with the DRI pellets.

Summing up, it can be said that the M2 to M4 mixture briquettes can be stored safely and handled on the grounds of the Midrex facility so that they can be recycled in the shaft furnace for direct reduction. These briquettes should have sufficient thermal stability because their water content is zero, and they contain virtually no volatile matter that could trigger thermal disintegration. To fully assess the suitability of the residues briquettes for recycling, it is necessary to test the reducibility.

7.3. Transferability of the Test Results to Roller Presses

In principle, the results of pressing experiments are transferable from the hydraulic piston press scale to the roller press scale as far as the qualitative trends of the quality parameters of the briquettes are concerned. This is acceptable because material densification is realized by both types of pressing machines mainly through the compression force associated with the formation of similar binding forces within the particle compound. This implies that optimum parameter ranges apply to both press types, for example, when it comes to the grain size and the grain size distribution, the pressing temperature, and the material moisture. Otherwise, it would not make any sense to conduct briquetting tests with a hydraulic piston press to prepare industrial briquetting.

When it comes to the pressing force which is needed for densification, however, transferability is not immediately given because the pressing forces are defined differently: the pressing force related to the piston area (hydraulic piston press) and the pressing force related to the active width of the mold segments (roller press). It is, though, also necessary to take into account the one-axial pressing force (hydraulic piston press) as opposed to the three-axial compression force combined with the shear stress (roller press). A practical way out of this dilemma is to compare briquettes with the same apparent density from both types of presses to define an equivalent pressing force for roller presses. This is possible because the apparent density can be seen as a kind of “universal” briquette quality parameter that is linked to all briquette strength parameters and also to some chemical briquette properties such as reactivity, the oxidation/reduction behavior, or hygroscopicity.

The apparent density provides information about the state of the particle aggregation in the briquette and, thus, about the type and the efficiency of the binding forces. But a comparison of the apparent density makes only sense if the material and the machine parameters of both types of presses are constant. This correlation is shown in Figure 8. The parameter values in Figure 8 relate to the briquetting tests which had been conducted earlier with lignite. When it comes to the hot briquetting of the DRI pellets which were used in the trials, we can give the following relevant data for a comparison with the roller press and the hydraulic piston press (at an apparent density of the briquettes ranging between 5.05 and 5.08 g cm⁻³):

For the roller press: 1 m roll diameter, 0.3 m s⁻¹ roll speed, 14 cm segment width, specific pressing force of the roller press between 149 and 156 kN cm⁻², pressing temperature 700–800 °C, briquette shape: full pillow (12 cm × 6 cm × 4 cm).

For the hydraulic piston press: Preset pressure 250 MPa at a constant piston speed, 3 s residence time at maximum pressure, temperature 800 °C, briquette shape: cylindrical (Ø 50 mm, height 14–18 cm).

This experimental data provides sufficient proof that a roller press with a roll diameter of 1 m can achieve a specific compression force that is suitable to form briquettes with the same quality parameters as produced by a hydraulic piston press and, thus, assure a sufficient production rate at the same time. Nevertheless, to validate the results of the study, trials should be conducted with a roller press as a next step.

8. Conclusion

Hot briquetting tests with mixtures of sponge iron pellets and residues of the Midrex direct reduction process were conducted
to ascertain the feasibility of recycling residues by including them directly in HBI. The inclusion of such residues as oxide fines, dried sludge, HBI screened fines, HBI classifier dust, process classifier dust, and remet screened fines in HBI is possible when applying a high pressure of 350 MPa and a high briquetting temperature of 800 °C. Up to a content of 20 wt% residue mixture, HBI meets the quality standards and requirements for the safe transportation to steel plants if the mixture contains sufficient HBI screened fines and HBI classifier dust, respectively. In other words, when the HBI briquettes have a compressive strength of >300 MPa, an abrasion resistance of >80%, and, most importantly, an apparent density of >5 g cm⁻³. Successful briquetting is not restricted to hydraulic piston presses. The process parameters (necessary pressure and temperature) can also be realized with roller briquetting machines.

Hot briquetting at 350 MPa and 800 °C of Midrex residues without sponge iron pellets is an option for recycling them in the Midrex shaft furnace. These residue briquettes do not qualify for safe transportation over a long distance, but it should suffice for transportation on the grounds of the direct reduction plant. The hot residue mixture briquettes contain no water or volatile matter, so that no premature disintegration in the shaft furnace should occur. However, their reduction properties still need to be ascertained.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

direct reduced iron, hot briquetted iron, hot briquetting, Midrex process, residues

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