Briquetting of fine-grained residues from iron and steel production using organic and inorganic binders

Laura Lohmeier, Christoph Thaler, Christopher Harris, Ralf Wollenberg, Hans-Werner Schröder*

Laura Lohmeier, Dr.-Ing. Ralf Wollenberg, Dr.-Ing. Hans-Werner Schröder
TU Bergakademie Freiberg (TUBAF), Institute of Thermal-, Environmental- and Resources' Process Engineering (ITUN), Leipziger Straße 28, 09599 Freiberg, Germany
E-mail: Laura.Lohmeier@tun.tu-freiberg.de

Dr. mont. Christoph Thaler, Dr. Christopher Harris
voestalpine Stahl GmbH, voestalpine-Straße 3, 4020 Linz, Austria

Keywords: Midrex® process, residues, briquetting, binders, disintegration

The Midrex® process produces metallurgical residues in the form of dust, sludge and fines. As these have a high iron content, the aim of this study is to recycle the residues and use them as an educt in the Midrex® process, thus closing the material cycle and increasing raw material efficiency. Briquetting of these materials with binder is one possibility to prepare them for the use as an educt in the Midrex® process. Experiments were carried out to test the suitability of the organic binders starch and cellulose for briquetting. Furthermore, tests with the inorganic bentonite were included for comparison. Briquettes are generally characterized by high strength. However, compared to iron oxide pellets, they have a low porosity and thus a higher apparent density and consequently a worse reducibility. The use of organic binders should improve the reducibility. The iron oxides are in close contact with the C-carrier of the organic binder so that a solid-solid phase direct reduction can take place. Furthermore, the solid carbon reacts to CO and thus increases the presence of reducing gas in the enlarged pores of the briquettes. And should therefore increase the degree of reduction.

1. Introduction

During the production of hot briquetted iron (HBI) from raw iron oxide pellets in the Midrex® process a wide variety of residues are generated. These residues are sludges, screened fines and dust and are generally rich in iron oxides [1-2]. Hence, they have the potential of being recycled back to the Midrex® shaft after having been agglomerated. For agglomerates to be considered suitable as feed material for the Midrex® direct reduction shaft, they should have sufficient cold strength, thermal stability and reducibility of iron containing species [3]. The cold bond pressure agglomeration process offers a possible method for achieving this. For cold bond pressure agglomeration, a binder is necessary [4]. Therefore, this paper presents results of the experiments carried out to study the effect of various parameters on the quality of briquettes prepared using the Midrex® residues and different binders. The binders were organic (starch, cellulose) or inorganic (bentonite). The experiments were based on a statistical experimental design and analysis strategy

This article has been accepted for publication and undergone full peer review but has not been through the copyediting, typesetting, pagination and proofreading process, which may lead to differences between this version and the Version of Record. Please cite this article as doi: 10.1002/srin.202000238.

(DOE-Design of experiments) and take a look at the influence of water and binder content of the mixture and the used binder on briquette strength. As briquettes are characterized by high density and low porosity, they have a worse reducibility compared to iron oxide pellets, for example. The use of organic binders is intended to improve reducibility. The iron oxides are in close contact with the C-carrier of the organic binder so that a solid-solid phase direct reduction can take place. Furthermore, the solid carbon reacts to CO and thus increases the presence of reducing gas in the enlarged pores of the briquettes. All in all, this increases the degree of reduction.

2. Midrex® process and residues

The dominating technology for the production of direct reduced iron as a pre-product for crude steel production is the Midrex® process. 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 direct reduced iron (DRI). Hot reducing gas (H_2 and CO) is fed countercurrently to the shaft and passes upward and reduces the iron oxide pellets (these consist mainly of hematite) to the metallic phase. Since DRI pellets have the potential to reoxidize and generate heat, the hot DRI is briquetted to make a much more stable product called Hot Briquetted Iron (HBI) [1]. During the various stages of HBI production a number of iron bearing residues are generated. In order to reduce material losses to the off-gas and minimize flow problems in the shaft furnace, the fed iron oxide pellets are screened at 5 - 6 mm, resulting in net fines losses of up to 10 wt%. Moreover, the hot briquetted iron is screened (losses 1 - 3 wt%) and there are fines losses from the reduction shaft to the off gas (1 - 1.5 wt%). The off gas goes to a scrubber and the scrubber residue (sludge) is dried [2]. Since these residues cannot be recycled back, they need to be agglomerated. The aim is to collect the fines, dusts and sludge generated in the Midrex® process, briquette it and charged the briquettes directly back to the shaft together with the iron oxide pellets. The main benefit is to substitute raw material and avoid disposal of the residues.

3. Quality requirements for feed materials used in Midrex® process

Briquettes to be considered suitable as feed material for the Midrex® process should have sufficient strength for handling, transportation and storage (physical properties), sufficient thermal stability to avoid premature disintegration, complete reducibility of iron oxides to metallic iron (metallurgical properties), and the content of gangue should be as low as possible (chemical properties). The only major chemical change to pellets in the direct reduction process is the removal of oxygen, because there is no melting or refining. As a result, impurities and gangue in the DRI-briquettes are present. Therefore, the iron content of the feed materials should be as high as possible (preferred 67 wt%) and the gangue content as low as possible. Especially acid gangue constituents like silica and alumina should amount to max. 2 wt%. The total amount of gangue in feed material generally

should not exceed 3 – 4 wt%, since gangue will require additional electric power in the electric arc furnace (EAF) and increase refractory wear [3]. Physical characteristics are defined for iron oxide pellets for use as Midrex® feedstock. A preferred Midrex® feedstock will be of consistent size to allow homogeneous feeding and sufficient mechanical strength to prevent degradation and fines generation during handling and transport. About 95 wt% of iron oxide pellets should be in the size range of 9 - 16 mm, while the fraction < 3 mm should be minimized. The pellets should have a tumble strength of 90 - 95 % > 6.73 mm. Cold compressive strength for pellets should be 250 kg or greater [3]. The requirements of pellets cannot be transferred directly to briquettes, nevertheless adequate mechanical strength of the briquettes is necessary [4]. Furthermore, the complete reduction of the metallic oxides in the briquettes to metallic iron in the Midrex® shaft is important.

4. Briquetting with a binder

Cold bond briquetting may offer a method for recycling the residues. Fines, dusts and sludge can be briquetted using a binder to a form that is suitable for charging into the Midrex® shaft. To get briquettes with sufficient mechanical strength and thermal stability a binder is needed. With the selection of a binder the mechanical and metallurgical properties of the briquettes can be influenced. A distinction between organic and inorganic binder materials is possible [5]. Carbonaceous organic materials can be included in the briquettes to improve the reduction kinetics (reduction rate and reducibility) owing to the presence of a larger number of reaction sites simultaneously and due to shorter diffusion paths compared to inorganic binders.

In the literature, there are mainly studies on the production of self-reducing or composite pellets with organic substances [6 - 15]. However, the addition of carbon usually lowers the strength of agglomerates. Therefore, briquettes normally contain less than 10 wt% carbon [15-16]. Another disadvantage of organic binders is the volatility of the organic substance during thermal treatment (begins at around 300 °C). The most important binder for production of iron oxide pellets is inorganic bentonite [17]. Bentonite is a silicate with a low melting temperature. The silicate components melt, pull particles together and promote sintering of iron oxide grains. Upon cooling additional solid bonds are added (recrystallization processes). Thus, a high thermal stability of the pellets is achieved [18]. However, the disadvantage is, that bentonite increases the silica content in the HBI (gangue) [19].

Concerning briquettes, produced with organic or inorganic binders, it can be assumed that they have a worse reducibility than iron oxide pellets due to their larger size and lower porosity. On the other hand, the briquettes from Midex® residues partly contain already reduced material. It is expected

that by using organic binders the reducibility can be improved compared to briquettes with inorganic binders.

5. Materials and methods

5.1. Materials

The materials for briquetting were the Fe-carrier (a mixture of residues from Midrex® process), binder and water. The mixture of Fe-carriers includes screened oxide fines, HBI screened fines, HBI classifier dust, dried sludge, process dust and remet fines. **Table 1** shows the composition of the Fe-carrier used for the tests. The results of the analysis for chemical composition of the different residues is shown in **Table 2** and the water content in **Table 3**. The residues have already been pre-reduced to varying degrees. The HBI screened fines were characterized by the highest content of metallic iron (Fe_{met} = 74.9 wt%) and total iron (Fe_{tot} = 88.7 wt%). **Table 4** shows the cumulative particle size distribution $Q_3(d)$, where d is the grain diameter, the median particle size d_{50} and the mean particle size d_m of the different residues. Two organic and one inorganic binder were used for the briquettes. These were pre swelled wheat starch, cellulose glue (dried, pre-swollen and ground cellulose) and a bentonite clay.

5.2. Feed preparation and briquetting

The materials, at first the Fe-carrier components, then the binder and at the end water, were fed into an Eirich-Mixer „R02“ with a volume of 2 liter and mixed for 10 min. The amount of water needed was calculated based on the water content of the Fe-carriers and the binder. After mixing, the water content of the mixture was analyzed and, if needed, more water was added. The temperature of the material in the mixer was 35 °C up to 40 °C. The mixture was then preheated prior to briquetting to 60 °C. The preheated material was pressed with a hydraulic stamp press (Raster Zeulenroda, PYXE 250 F) into cylindrical briquettes, each having a diameter of 5 cm and a height of 2 cm and a briquette mass of approx. 150 g. A pressure of 140 MPa as well as a pressing time of 3 seconds and a pressing temperature of 60 °C were determined and used for the experiments. The briquettes were hardened under ambient conditions for one day.

5.3. Briquette testing procedure

In order to characterize the briquette quality the following parameters were determined:

The compressive strength σ_P (based on former TGL 9491) of the briquettes was 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 was an indicator of the compressive strength σ_P. The compressive strength of five briquettes was determined and averaged. The standard deviation for the compressive strength was on average less than 1 MPa. A significant difference between the various binders concerning variability of compressive strength cannot be determined. The abrasion resistance was determined according to DIN 51717 by the defined stress which was imposed on the briquettes inside a drum. The drum has a diameter of 50 cm and a length of 50 cm. Apart from this the drum has four blades of 8 cm inside displaced by 90°. The residue on the 30 mm sieve (m_R *(d > 30 mm)*) after a sample of five briquettes had tumbled for 100 rotations inside the drum, in relation to the total mass of the weighed briquettes (m_{tot}), was an indicator of their abrasion resistance *R30(100)*.

$$R30(100) = \frac{m_R(d>30\ mm)}{m_{tot}} \cdot 100\% \quad (1)$$

The shatter strength S_{20} was tested based on ISO 616:1995. A test portion of five briquettes is dropped from 2 meters to a base plate 4 times. After the drops the mass of briquettes retained on a test sieve with 20 mm (m_R *(d > 20 mm)*) in relation to the total mass of the weighed briquettes (m_{tot}) is determined.

$$S_{20} = \frac{m_R(d>20\ mm)}{m_{tot}} \cdot 100\% \quad (2)$$

The apparent density ρ_{app} of the briquettes was determined by measuring the briquette (height and diameter) with caliper gauge and additionally weighing it. The apparent density is the ratio of the mass of the briquette to the volume. The low-temperature disintegration test was performed based on ISO 4696. The briquettes are isothermally reduced in a fixed bed, at 550 °C, using a reducing gas consisting of 20.5 vol% H_2, 13.5 vol% CO, 10 vol% CO_2 and 44 vol% N_2 (adapted for conditions in Midrex® shaft), for 30 min. The reduced sample is then tumbled in a specific tumble drum for 900 revolutions and then sieved. Furthermore, a reduction test was carried out for suitable briquettes in accordance with ISO 4695. The briquettes are first heated in an N_2 atmosphere, then reducing gas consisting of 42.5 vol% H_2, 31.5 vol% CO, 15.5 vol% CO_2 and 10.5 vol% N_2 (also adapted for conditions in Midrex® shaft) is introduced at 800 °C to 850 °C and the briquettes are isothermally reduced. During the reduction process, the decrease in mass is continuously recorded.

5.4. Design of experiments

As a first step, the influence of water content and binder content on the cold strength of the briquettes was investigated for different binders. A sufficient cold strength of the briquettes is an essential requirement for the use in the Midrex® process. In most cases the compressive strength, apparent density and the abrasion or shatter resistance go not on the same trend line so that a global parameter optimum has to be found which meets all strength requirements. This was done by means of statistical design of experiments with the software Statgraphics 18®. In the second step, briquettes with suitable strength values were then tested for low-temperature disintegration. If the briquettes passed this test, a reduction test could be carried out.

The values margin of the variables was: Fe-carrier 89 to 94 wt%, binder 3 to 8 wt% and water 3 to 8 wt% (the amount of water includes also the water in the binder and Fe-carrier). The total for controllable mixture components was 100 wt%. For the tests a Simplex-Centroid-Design was chosen that runs at all primary blends, binary blends and the tertiary blend (7-run simplex centroid design) and additional runs are located along axial lines running from the pure blends through the centroid (3 additional checkblends). The 10 design points are shown in **Figure 1**. The tests were carried out for three different binders. Bentonite as inorganic binder as well as cellulose and starch as organic binder. With the design of experiments an optimum mixture composition can be determined for each binder.

6. Results and discussion

6.1. Mechanical strength of briquettes

Table 5 shows the tests carried out and the strengths achieved for the binder starch (A1 to A10), the binder cellulose (B1 to B10) and the binder bentonite (C1 to C14). The compressive strength of the briquettes when using the various binders is shown in **Figure 2**, **Figure 3** and **Figure 4**. With all binders, high compressive strengths of over 30 MPa can be achieved with a suitable mixture composition, whereby the influence of the mixture composition is greatest with cellulose as a binder. Compressive strengths of up to 42.1 MPa (C4) can be achieved for bentonite and up to 43.2 MPa (A2) for starch. The compressive strength of the briquettes can be described with sufficient accuracy of R^2 = 70.7 % by a quadratic model for the binder bentonite and by a quadratic model for the binder starch (R^2 = 90.89 %). An overview of the model equations for describing the mechanical properties of the briquettes is shown in **Table 6**. The compressive strengths achieved with the binder cellulose tend to be lower than with starch and bentonite. Nevertheless, compressive strengths of up to 34.7 MPa (B1) are achieved. The compressive strength can also be described by a quadratic model with an R^2 of 93.87 %. Particularly high compressive strengths of greater than 35

MPa are achieved for the binder bentonite at a binder content of 3 to 5.5 wt% and a water content of 3.7 to 7 wt%. For the binder starch, the water content is decisive. For compressive strengths above 35 MPa, this should be between 3 and 4 wt%. For cellulose, the highest strengths (greater than 33 MPa) are achieved at a binder content of 3 to 5 wt% and a water content of 3 to 6 wt%, provided that the Fe carrier content is greater than 91 wt%.

Furthermore, sufficiently high abrasion resistance of more than 85 % could be achieved with all binders (with suitable water and binder content), shown in **Figure 5**, **Figure 6** and **Figure 7**. For bentonite, a very strong influence of the water and binder content on the abrasion resistance was found, as values for abrasion resistance between 0 and 91.5 % were determined. The determined maximum value is 91.5 % with 5.5 wt% water and 5.5 wt% binder (C13). High abrasion resistance of more than 85 % is achieved with a water content of the mixture between 5 and 8 wt%. The abrasion resistance for the binding agent bentonite can be determined with sufficient accuracy of R^2 = 83.2 % with a quadratic model. For the binder starch all determined abrasion resistance values are between 91.9 % and 98.5 %. The abrasion resistance for the binder starch can be described with a quadratic model with a high accuracy of R^2 = 93.6 %. Good abrasion resistance between 80.9 % and 92.1 % is also achieved with the binding agent cellulose, with the exception of test point B1 (abrasion resistance 66.4 %). For an abrasion resistance of more than 85 %, a water content between 5 wt% and 8 wt% is particularly important. The abrasion resistance can be described with an accuracy of R^2 = 92.9 % using a quadratic model.

With regard to shatter resistance, sufficient strengths of more than 85 % can also be achieved for all binders, shown in **Figure 8**, **Figure 9** and **Figure 10**. In this context, the adjustment of a suitable water and binder content for the briquettes with bentonite is again of particular importance. The shatter strength of the briquettes with bentonite lies between 15.5 % and 97.6 % for the investigated briquettes. A water content of approx. 5 wt% is decisive for sufficiently high shatter resistance. The shatter strength of the bentonite briquettes can be described with a quadratic model with sufficient accuracy of R^2 = 90.2 %. For the binder starch, all shatter strengths are between 99 % and 99.7 % and thus reach the maximum possible in practice. An influence of binder and water content is not visible (p-value > 5 %). With the binding agent cellulose, shatter strengths of a maximum of 97.8 % (B10) are achieved. As the p-value is also higher than 5 %, a description based on a model is not possible. A water content of the mixture of 5 to 6 wt% favors a high abrasion resistance.

Figure 11, **Figure 12** and **Figure 13** shows the apparent density of the briquettes. The apparent density of the briquettes can be represented with high accuracy (R^2 > 84 %) for all three binders by a quadratic model as a function of the mixture composition. The apparent density of the briquettes is in the range of 3.4 g cm^{-3} to 3.7 g cm^{-3} for the binding agent bentonite, in the range of 3.3 to 3.5 g

cm^{-3} for the binding agent starch and in the range of 3.1 to 3.3 g cm^{-3} for cellulose. Thus, higher apparent densities are achieved with the inorganic binder than with the two organic binders. But this does not correspond to the porosity of the briquettes due to the higher weight of the inorganic binder particles. When using the binder starch, the apparent density is mainly dependent on the binder content. For a low binder content of 3 wt% the highest apparent densities are achieved. When using bentonite as a binder, the apparent density is determined by the water content. For a high water content of more than 5 wt% the highest apparent densities are achieved. For cellulose as a binder the Fe-carrier content is decisive. A high content of Fe-carrier results in a high apparent density. As a result, the apparent density for the three binders investigated behaves differently. The bulk density of the briquetting mixtures of Fe-carrier, binder and water are similar for all mixtures (1.6 - 1.8 g cm^{-3}) and do not correlate with the apparent density of briquettes. Bentonite is a silicate with a low melting temperature. The particles are encased by the bentonite, the silicate components melt and thus contract the particles, increasing the apparent density of the briquettes in the Midrex® shaft [19]. The lower apparent density of briquettes containing starch and cellulose can be explained by the lower weight of the macromolecular binder particles in the first place. These binders promote plastic compaction as they act like a lubricant. It therefore can be assumed that they lead to less pore volume of the briquettes despite the fact of a lower apparent density.

All three investigated binders are characterized by a macromolecular structure, have the property of swelling and can absorb many times their own weight of water. The water absorption capacity and thus the swelling behavior can be characterized by means of the PWAT (Plate Water Absorption Test) value [5]. The exact value depends strongly on the composition of the binder and possible modifications. Basically, cellulose has particularly high PWAT values of up to 3000 %, while starch and bentonite reach values of approx. 1000 %. This behavior also influences briquette density [20].

For the briquettes with the binding agent cellulose, the apparent density correlates with the compressive strength of the briquettes. A high apparent density and a high compressive strength is achieved for low water contents (< 5 wt%) (the briquettes behave brittle), while high shatter and abrasion resistance is achieved for water contents greater than 5 wt%. Due to the higher water content the briquettes have a pressure plastic behavior. For the briquettes with bentonite and starch, the correlation between apparent density and compressive strength is not observed. In terms of strength, a high apparent density is desirable. However, a high apparent density of briquettes also results in lower porosity of briquettes, which can be a disadvantage especially for the reducibility of briquettes [21].

It is shown that when considering the abrasion and shatter resistance, the water content of the briquette mixture is the decisive factor. With increasing water content, higher strength values tend to be achieved, irrespective of the binder. During the shatter and abrasion test, the briquettes are subjected to dynamic and multi-axial mechanical stress. Therefore, a certain plastic deformability of the briquettes is useful to withstand this stress. In this case the addition of water ensures a certain plastic behavior of the briquettes. The pressure stress, on the other hand, is a quasi static load. The lower the water content, the more brittle the briquettes are, which is ultimately reflected in the higher compressive strength. For high water contents, on the other hand, the lubricating effect of the water comes into play, so that the briquettes are deformed under load. This is mainly visible for the binders starch and cellulose. With bentonite, compressive strengths of higher than 30 MPa are achieved in almost the entire area under investigation.

In terms of cold strength, the inorganic binder bentonite can be used to achieve a higher compressive strength, while the briquettes with the organic binders show better abrasion and shatter resistance. In general, however, briquettes with sufficient strength for transport and handling can be produced with all three binders investigated.

The advantage of briquettes with organic binders concerning reduction efficiency can be taken for granted. Nevertheless, this quality parameter has to be tested. The same applies to the thermal stability of the briquettes which is also an important requirement for the briquettes to be recycled into the Midrex® shaft. As already mentioned, the silicate components of bentonite favor sintering, as a result of which a high thermal stability of the briquettes can be expected [19]. Organic binders decompose at about 300 °C and form solid coke bridges that may also contribute to thermal stability.

The strengths of the Midrex® residue briquettes with the different binders tested can be described with sufficient accuracy (R^2 mostly > 90 %) using quadratic models. This is a quality criterion for the tests carried out and indicates a stable briquetting process and a low influence of disturbance variables, such as mixture inhomogeneities. **Table 7** gives a summary overview of the necessary composition of the mixtures to achieve sufficient strength. Additional briquettes were produced with each of the three binders for a suitable mixture composition to assess metallurgical properties and thermal stability.

6.2. Metallurgical properties of briquettes

In addition to cold strength, the thermal stability of the briquettes under the corresponding gas atmosphere and the reducibility is also of essential importance. The results of the low-temperature disintegration tests are shown in **Table 8**. It is obvious that the briquettes with bentonite are also suitable for use at elevated temperatures and corresponding gas atmospheres. The proportion of

briquette fragments larger than d = 6.3 mm after loading with reducing gas at temperatures of 550 °C and the subsequent abrasion stress is 91.8 wt%. On the other hand, the briquettes with organic binders cannot withstand these stresses. For the use of cellulose as a binding agent, the proportion greater than 6.3 mm is 17.3 wt%, and only 3.3 wt% for starch. This means that the briquettes with starch and cellulose would probably disintegrate when fed into the reduction shaft. This can be explained by the fact that the organic binders decompose at temperatures of approx. 300 °C and solid coke bridges form only at temperatures higher than 800°C. Although, part of the carbon passes to the gaseous phase through the reaction with reducing gas. As partial melt adhesion between the iron oxide particles does not yet occur at these temperatures, the briquettes disintegrate. The disintegration of the briquettes is not necessarily to be seen as negative, but the high dust content (d < 0.5 mm) of 59.7 wt% for cellulose and 68.6% for starch is problematic. This means that the gas flow through the reduction shaft could be disturbed. On the other hand, the briquettes have to break up into fragments to some extent in order to ensure trouble-free combined hot briquetting with the directly reduced iron oxide pellets and not to negatively influence the strength properties of the hot briquetted iron. Furthermore, the reducibility of smaller briquette pieces is better than that of a whole briquette.

For the briquettes with bentonite a reduction test was still being carried out due to the positive results of the low-temperature disintegration test. This shows that the reduction process is very slow due to the high density of the briquettes and their size. Only after 300 minutes a reduction degree of 80 % was achieved. As already predicted, briquettes with bentonite are thermally stable, but are not suitable for use in the Midrex® reduction shaft with regard to reducibility.

7. Conclusion

The Midrex® direct reduction process produces a number of fine-grained residual materials such as dust, sludge and fines. As these residues have a high iron content, the aim of these investigations was to briquette the residues in order to use them as feedstock in the Midrex® direct reduction process. The suitability of organic and inorganic binders has been investigated with regard to the cold strength and metallurgical properties of the briquettes. In the first part, tests were carried out on the basis of a simplex mixture experimental design. The tests showed that briquettes with sufficient cold strength could be produced with all three investigated binder cellulose, starch and bentonite. However, it is important to use a suitable water and binder content depending on the binder. The low-temperature disintegration tests have shown that the briquettes with the inorganic binder bentonite withstand the loads when fed into the reduction shaft, whereas the briquettes with the organic binder cellulose and starch disintegrate. However, it is also shown that the briquettes

with the inorganic binder bentonite have poor reducibility and are therefore also not suitable for use in the Midrex® direct reduction process. Consequently, the procedure of briquetting with organic binder needs further optimization for sufficient thermal stability. Furthermore, the expected effect of the better reducibility of the briquettes with organic binder still has to be proven. One chance beside others to make this possible could be the joint use of bentonite and starch or cellulose as a binder.

Acknowledgements

The authors gratefully acknowledge the funding support of K1-MET GmbH, metallurgical competence center. The research programme of the K1-MET competence center is supported by COMET (Competence Center for Excellent Technologies), the Austrian programme for competence centers. COMET is funded by the Federal Ministry for Climate Action, Environment, Energy, Mobility, Innovation and Technology, the Federal Ministry for Digital and Economic Affairs, the Federal States of Upper Austria, Tyrol and Styria as well as the Styrian Business Promotion Agency (SFG). Beside the public funding from COMET, this research project is partially financed by the scientific partner TU Bergakademie Freiberg (TUBAF), Institute of Thermal-, Environmental- and Resources' Process Engineering (ITUN) and the industrial partner voestalpine Stahl GmbH Linz.

Received: ((will be filled in by the editorial staff))
Revised: ((will be filled in by the editorial staff))
Published online: ((will be filled in by the editorial staff))

References

[1] T. Battle, U. Srivastava, J. Kopfle, R. Hunter, J. McClelland, in *Treatise on Process Metallurgy*, Vol. 3 (Eds: S. Seetharaman), Elsevier, Amsterdam, Netherlands **2014**, Ch. 1.2.

[2] C. M. Ravenscroft, *Direct from Midex* **2011**, 3rd and 4th Quarter.

[3] L. Lorraine, *Direct from Midex* **2019**, 4th Quarter.

[4] S. H. Freitas Seabra da Rocha, *Dr. Thesis*, Technische Hochschule Aachen, **2009**.

[5] T. C. Eisele, S. K. Kawatra, *Miner. Process. Extr. Metall. Rev.* **2003**, 24, 1.

[6] C. Takano, M. B. Mourao, *Miner. Process. Extr. Metall. Rev.* **2003**, 24, 3 & 4.

[7] M. Sunde, *Dr. Thesis*, Norwegian University of Science and Technology, **2012**.

[8] D. Zhu, G. Qiu, T. Jiang, J. Xu, *J. Cent. South Univ. Technol.* **2000**, 7, 2.

[9] R. Sah, *Dr.-Thesis*, Maharaja Sayajirao University of Baroda, **2008**.

[10] G. Qiu, T. Jiang, Z. Huang, D. Zhu, X. Fan, *ISIJ Int.* **2003**, 43, 1.

[11] B. B. Agrawal, K. K. Prasad, S. B. Sarkar, H. S. Ray, *Ironmaking Steelmaking* **2001**, 28, 1.

[12] R. Sah, S. K. Dutta, *Miner. Process. Extr. Metall. Rev.* **2010**, 31, 2.

[13] H. W. Gudenau, B. Lukat, K. Stoesser, *Steel Res. Int.* **1998**, 69, 10 & 11.

[14] M. A. Somerville, *J. Sustain. Metall.* **2016**, 2, 3.

[15] M. Singh, *Dr. Thesis*, Lulea University of Technology, **2003**.

[16] J. A. Halt, S. C. Roache, S. K. Kawatra, S. Komar, *Miner. Process. Extr. Metall. Rev.* **2014**, 36, 3.

[17] H. Liu, Bing. Xie, Y. Qin, *J. Chem.* **2017**.

[18] O. Sivrikaya, A. I. Arol, *Powder Technol.* **2011**, 210, 1.

[19] J. A. Halt, S. K. Kawatra, *Miner. Metall. Process.* **2014**, 31, 2.

[20] L. A. Haas, J. A. Aldinger, R. K. Zahl, Bur. *Mines Rep. Invest.* **1989**, RI 9230.

[21] W. Pietsch, *Agglomeration in Industry. Occurrence and applications*, Wiley-VCH, Weinheim, **2008**.

The table of contents entry should be 50–60 words long and should be written in the present tense and impersonal style (i.e., avoid we). The text should be different from the abstract text.

The Midrex® direct reduction process produces a number of ferrous, fine-grained residues. To avoid disposal, a way of briquetting the residues with binder and returning them to the Midrex® direct reduction process is demonstrated. Laboratory-scale briquetting experiments with inorganic and organic binders are presented and their suitability is compared with regard to the mechanical and metallurgical properties of the briquettes.

Keyword briquetting

Christoph Thaler, Christopher Harris, Ralf Wollenberg, Hans-Werner Schröder, Laura Lohmeier*

Briquetting of fine-grained residues from iron and steel production using organic and inorganic binders

ToC figure

Table 1. Composition of the Fe-carrier mixture

Component	Oxide fines	Dried sludge	Process dust	HBI (screened fines)	HBI (classifier dust)	Remet fines
Content (wt%)	30	40	5	15	5	5

Table 2. Chemical composition of the residues

Component	Fe_{tot} wt%	Fe_2O_3 wt%	FeO wt%	Fe_{met} wt%	C wt%	SiO_2 wt%	CaO wt%	Al_2O_3 wt%	MgO wt%	K_2O wt%	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 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

Table 3. Water content of the residues

Component	Oxide fines	Dried sludge	Process dust	HBI (screened fines)	HBI (classifier dust)	Remet fines	Bentonite	Cellulose	Starch
Amount of water in wt%	1.0	1.3	8.5	0.41	10.8	5.4	16.2	5.83	7.86

Table 4. Grain size of the residues

	unit	Oxide fines	Dried sludge	Process dust	HBI (screened fines)	HBI (classifier dust)	Remet fines
$Q_3(10\ mm)$	wt%	100.0	100.0	100.0	100.0	100.0	100.0
$Q_3(6.3\ mm)$	wt%	99.6	100.0	99.5	100.0	98.6	99.6
$Q_3(4\ mm)$	wt%	97.4	99.9	99.3	99.8	98.0	98.4
$Q_3(2\ mm)$	wt%	86.1	99.0	92.5	48.1	85.5	71.7
$Q_3(1\ mm)$	wt%	71.4	97.2	55.2	28.1	21.5	55.1
$Q_3(0.63\ mm)$	wt%	63.5	96.1	16.3	19.5	18.6	14.7
$Q_3(0.5\ mm)$	wt%	60.9	95.1	3.0	16.8	0.9	14.5
$Q_3(0.315\ mm)$	wt%	60.8	94.5	1.2	16.7	0.2	13.8
$Q_3(0.125\ mm)$	wt%	44.4	74.1	0.1	5.3	0.1	0.1
$Q_3(0.09\ mm)$	wt%	34.7	61.9	0.0	4.1	0.0	0.1
$Q_3(0.063\ mm)$	wt%	24.8	43.8	0.0	2.9	0.0	0.1
d_m	mm	0.764	0.140	1.122	1.865	1.416	1.401
d_{50}	mm	0.190	0.072	0.951	2.074	1.445	0.953

Table 5. Results of the briquetting tests for mechanical strength

Number	Binder	Fe-carrier wt%	Binder wt%	Water wt%	σ_P MPa	*R30(100)* %	S_{20} %	ρ_{app} g cm^{-3}
A1	Starch	94.00	3.00	3.00	36.9	91.9	99.0	3.33
A2	Starch	92.33	3.83	3.83	43.2	96.5	99.6	3.42
A3	Starch	91.50	3.00	5.50	30.2	97.0	99.6	3.29
A4	Starch	89.83	6.33	3.83	30.6	97.5	99.7	3.49
A5	Starch	89.00	5.50	5.50	23.3	97.1	99.6	3.38
A6	Starch	90.67	4.67	4.67	23.9	98.5	99.7	3.47
A7	Starch	89.00	3.00	8.00	6.5	98.2	99.6	3.36
A8	Starch	91.50	5.50	3.00	37.5	95.8	99.2	3.46
A9	Starch	89.00	8.00	3.00	41.8	97.2	99.6	3.42
A10	Starch	89.83	3.83	6.33	17.7	98.4	99.7	3.45
A11[a)]	Starch	93.40	3.00	3.60	42.1	95.5	98.8	3.52
B1	Cellulose	94.00	3.00	3.00	34.7	66.4	89.6	3.31
B2	Cellulose	91.50	3.00	5.50	33.2	86.2	97.6	3.25
B3	Cellulose	89.00	5.50	5.50	32.3	92.1	97.7	3.17
B4	Cellulose	89.83	6.33	3.83	28.9	87.4	83.2	3.18
B5	Cellulose	92.33	3.83	3.83	34.0	82.4	87.0	3.29
B6	Cellulose	90.67	4.67	4.67	31.6	83.3	75.9	3.23
B7	Cellulose	91.50	5.50	3.00	31.2	80.9	85.5	3.22
B8	Cellulose	89.00	3.00	8.00	15.6	90.4	87.8	3.07
B9	Cellulose	89.00	8.00	3.00	29.2	85.4	94.2	3.12
B10	Cellulose	89.83	3.83	6.33	30.6	90.9	97.8	3.21
B11[a)]	Cellulose	92.40	3.00	4.60	41.4	91.9	98.1	3.36
C1	Bentonite	94.00	3.00	3.00	34.0	0.0	15.5	3.51
C2	Bentonite	90.00	7.00	3.00	31.7	59.4	39.8	3.43
C3	Bentonite	90.00	3.00	7.00	35.8	85.7	73.0	3.69
C4	Bentonite	91.33	4.33	4.33	42.1	85.8	84.2	3.64
C5	Bentonite	92.00	5.00	3.00	33.6	22.3	32.9	3.33
C6	Bentonite	92.00	3.00	5.00	39.9	86.1	78.2	3.70
C7	Bentonite	90.00	5.00	5.00	34.8	86.0	84.3	3.63
C8	Bentonite	92.67	3.67	3.67	35.7	5.2	28.3	3.54
C9	Bentonite	90.67	5.67	3.67	34.2	68.0	49.3	3.49
C10	Bentonite	90.67	3.67	5.67	41.2	88.5	88.3	3.66
C11	Bentonite	89.00	8.00	3.00	34.0	25.6	26.4	3.49
C12	Bentonite	89.00	3.00	8.00	24.4	86.5	66.5	3.67

C13	Bentonite	89.00	5.50	5.50	36.3	91.5	97.6	3.71
C14	Bentonite	90.67	4.67	4.67	32.9	83.9	88.9	3.54
C15[a)]	Bentonite	90.00	5.00	5.00	41.5	87.1	92.9	3.68

[a)]Briquettes used for tests on metallurgical properties

Table 6. Overview of the model equations to describe the mechanical properties of the briquettes (Fe is the amount of Fe carrier, W of water and B of binder, analyzed for $0.89 < Fe < 0.94$; $0.03 < W < 0.08$; $0.03 < B < 0.08$ and $Fe + W + B = 1$)

Binder	Compressive strength σ_P in MPa	Abrasion resistance *R30(100)* in %	Shatter strength S_{20} in %	Apparent density ρ_{app} in g cm^{-3}
Bentonite	=33.9·Fe+33.6·B+25.6·W +8.1·Fe·B+44.4·Fe·W+20. 3·B·W (R^2=70.7%)	=11.6·Fe+36.9·B+82.5·W +92.9·Fe·B+187.5·Fe·W+ 130.5·B·W (R^2=83.2%)	=11.4·Fe+29.0·B+63.7·W +48.4·Fe·B+164.7·Fe·W+ 202.5·B·W (R^2=90.2%)	=3.49·Fe+3.51·B+3.69·W +0.78·Fe·B+0.42·Fe·W+0. 35·B·W (R^2=84.9)
Starch	=39.6·Fe+41.2·B+6.3·W+ 12.6·Fe·B+29.6·Fe·W+14. 4·B·W (R^2=90.9%)	=91.9·Fe+97.0·B+98.1·W +7.8·Fe·B+10.7·Fe·W (R^2=93.6%)	p-value > 0.05 (description with model not useful)	=3.47·Fe+3.29·B+3.46·W +0.01·Fe·B+0.09·Fe·W+0. 08·B·W (R^2=97.8%)
Cellulose	=34.9·Fe+28.6·B+16.5·W +8.6·Fe·B+30.2·Fe·W+35. 4·B·W (R^2=93.9%)	=67.4·Fe+85.7·B+90.5·W +14.3·Fe·B+34.4·Fe·W+9. 4·B·W (R^2=92.9%)	p-value > 0.05 (description with model not useful)	=3.31·Fe+3.12·B+3.08·W +0.01·Fe·B+0.30·Fe·W+0. 33·B·W (R^2=98.1%)

Table 7. Summary of necessary water, binder and Fe-carrier contents to achieve sufficient mechanical strength (analyzed for 89 wt% < Fe < 94 wt%; 3 wt% < W < 8 wt%; 3 wt% < B < 8 wt%)

	Compressive strength σ_P > 35 MPa	Abrasion resistance *R30(100) > 85 %*	Shatter strength S_{20} > 85 %	Apparent density ρ_{app}
Bentonite	Binder 3 – 5.5 wt% Water 3.7 – 7 wt%	Water 5 – 8 wt%	Water 5 wt%	ρ_{app} > 3.6 g cm^{-3} for Water > 5 wt%
Starch	Water 3 – 4 wt%	sufficient strength in the entire area examined	sufficient strength in the entire area examined	ρ_{app} > 3.4 g cm^{-3} for Binder < 5 wt%
Cellulose	Binder 3 – 5 wt% Water 3 – 6 wt% Fe-carrier > 91 wt%	Water 5 – 8 wt%	Water 5 – 6 wt%	ρ_{app} > 3.2 g cm^{-3} for Fe > 91 wt%

Table 8. Results of the sieve analysis after the low-temperature disintegration tests

Binder	d > 6.3 mm	d < 3.2 mm	d < 2.8 mm	d < 0.5 mm
Bentonite	91.8 wt%	8.2 wt%	8.1 wt%	7.5 wt%
Starch	3.3 wt%	91.6 wt%	89.7 wt%	68.6 wt%
Cellulose	17.3 wt%	78.4 wt%	76.7 wt%	59.7 wt%

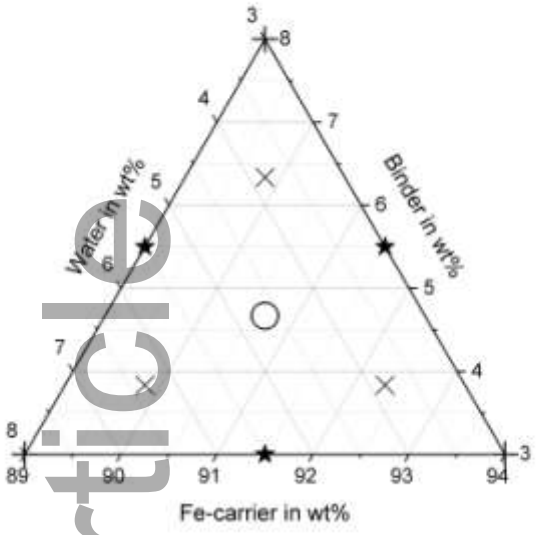

Figure 1. Design of Experiments: Simplex-Centroid-Design with all primary blends (marked with +), binary blends (*) and the tertiary blend (center point, O) and additional runs (x)

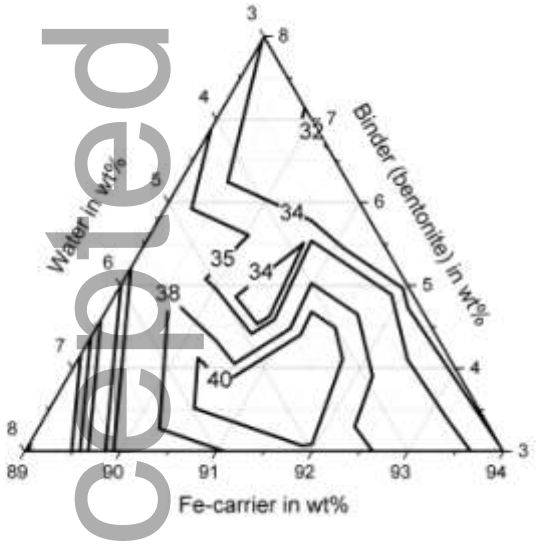

Figure 2. Compression strength σ_P in MPa of the briquettes with bentonite

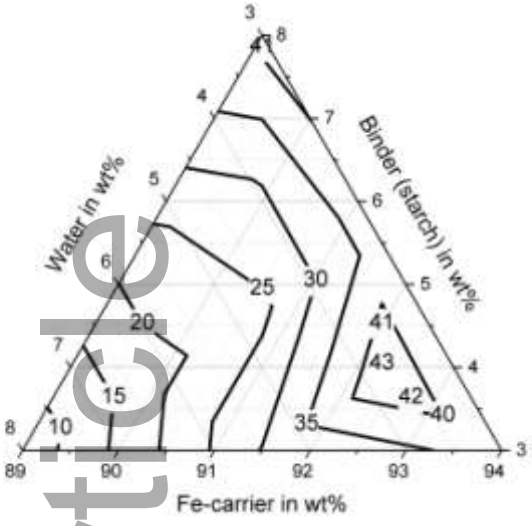

Figure 3. Compression strength σ_P in MPa of the briquettes with starch

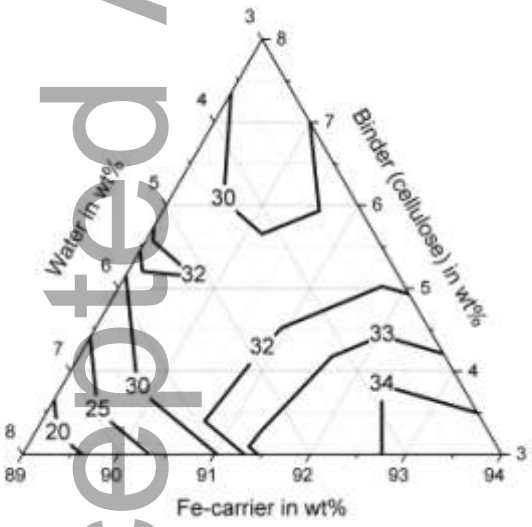

Figure 4. Compression strength σ_P in MPa of the briquettes with cellulose

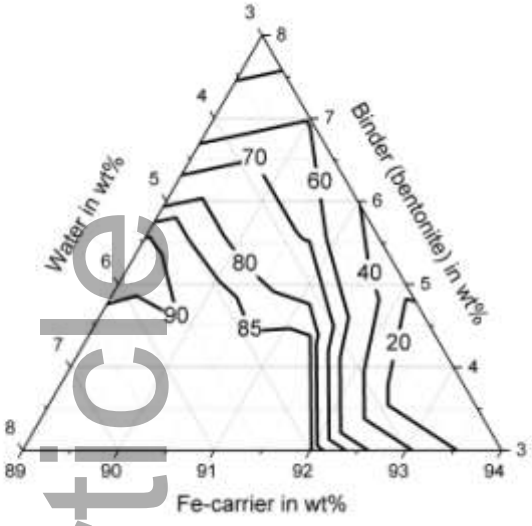

Figure 5. Abrasion resistance *R30(100)* in % of the briquettes with bentonite

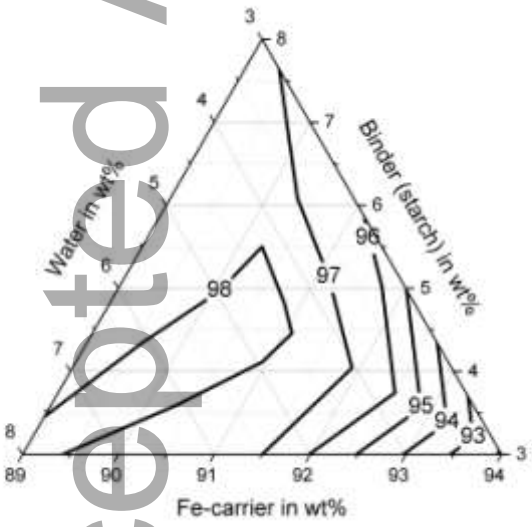

Figure 6. Abrasion resistance *R30(100)* in % of the briquettes with starch

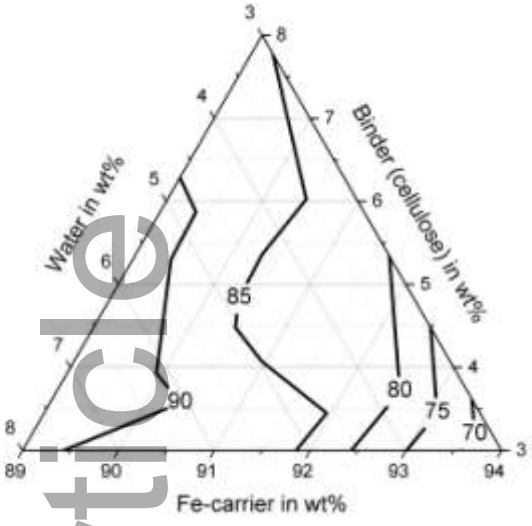

Figure 7. Abrasion resistance *R30(100)* in % of the briquettes with cellulose

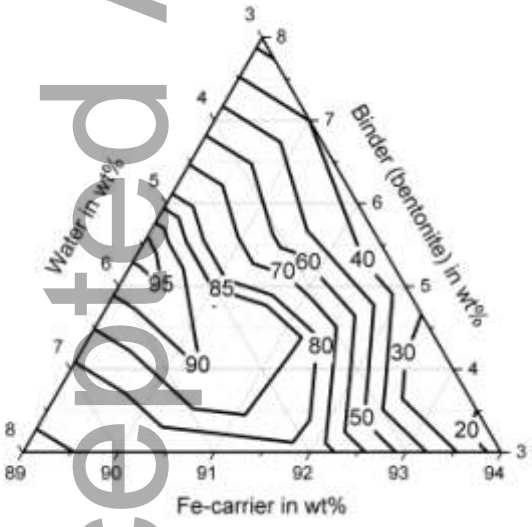

Figure 8. Shatter strength S_{20} in % of the briquettes with bentonite

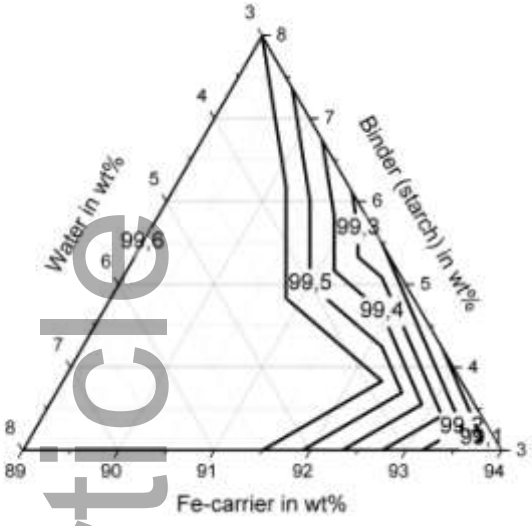

Figure 9. Shatter strength S_{20} in % of the briquettes with starch

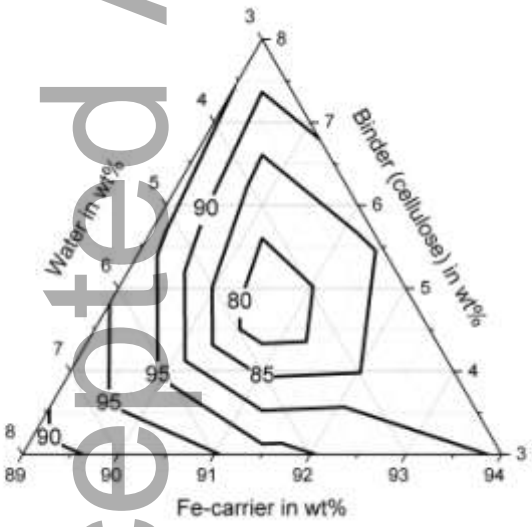

Figure 10. Shatter strength S_{20} in % of the briquettes with cellulose

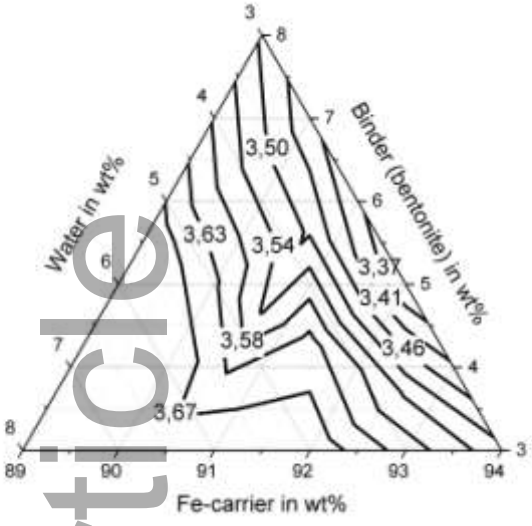

Figure 11. Apparent density ρ_{app} in g cm^{-3} of the briquettes with bentonite

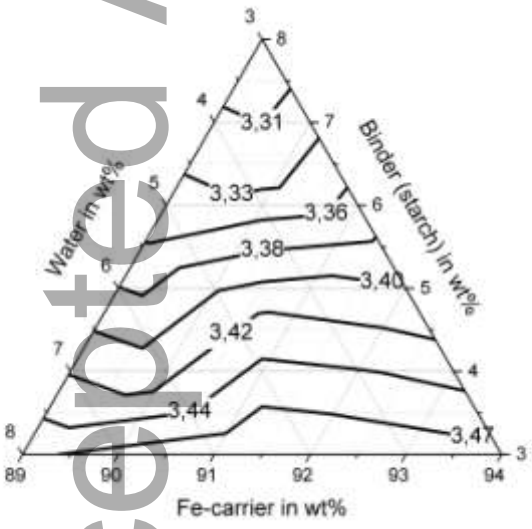

Figure 12. Apparent density ρ_{app} in g cm^{-3} of the briquettes with starch

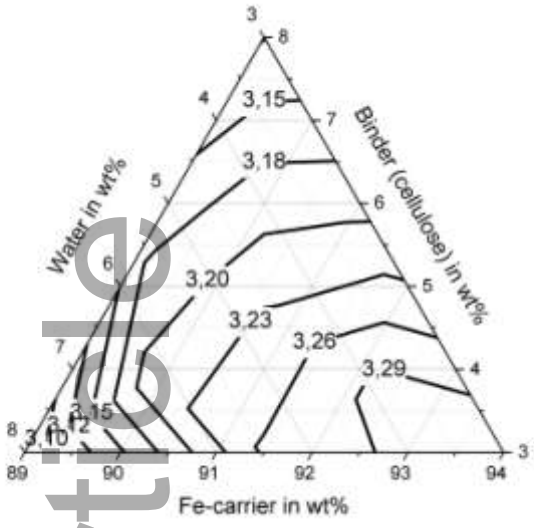

Figure 13. Apparent density ρ_{app} in g cm^{-3} of the briquettes with cellulose