Characterization of iron ore pellets with dextrin added organic binders under different time and temperature conditions

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Abstract: In the present work, Dextrin is used as a binder for iron ore pelletization, as it’s free from Silica and Alumina. Green pellets were prepared by mixing of iron ore particles (-75 µm) with varying binder percentage (1 wt. % bentonite, 0.5 and 1 wt. % of dextrin) separately. The green pellets were first dried in air for 24 h and then in an electric oven at 383 K for 4 h. The dried pellets were fired at varying temperatures (1173, 1273, 1373, and 1473 K) and indurated for 1 and 2 h. The samples were characterized for physical (pellet size) and mechanical (compressive strengths, porosity and hardness) properties. Good quality pellets were prepared with organic binder, and which increases the compressive strength of dry and fired pellets. Strength results are matching with the bentonite binder pellets and it is well above the industrially acceptable limit (250 kg/pellet). Porosity of the fired pellets decreases with increasing temperature and induration time. The hardness of the pellet varies from surface to core of the pellet. Moreover, the influence of wear mechanism (based on collisions) on pellets and its characteristics of generated particle size distribution (PSD) have been investigated. It was noticed that the material loss during wear test decreases with increasing strength of the pellet. From PSD analysis, the coarse particles were revealed that the collisions are dominating during wear.

1. Introduction

Iron-ore is a common element in the earth crust (5-6%) and it is most used metal, accounting for 93% of global metallic and alloy products [1]. During mining process, huge amount of fines is generated which cannot be used directly in the furnaces, these fines are need to be recycled and re-utilized. The extraction of Fe content from low grade iron ores are not suitable for iron making process due to its production cost and they are crushed in to fines, these fines can be enhanced through mineral-agglomeration processes [2]. C.V Firth 1944. Published the first systematic research works on the pelletization of magnetite and hematite iron ores. He showed that they have roughly similar pelletization characteristics [3]. In Popular pelletization process moisture, flux and binders are added to the iron ore powder and make pellets subsequently thermally treated to attain the required strength (ISO). The cold strength of the pellet was investigated using a compressive strength and drop number [4]. The use of bentonite is favorable concerning physical, mechanical and metallurgical properties. However, because of its acid constituents (SiO2 and Al2O3), it is considered as a chemical impurity, especially when concentrate with high SiO2 content. These have adverse effects on the process. De Souza, Kater and Steeghs (1984) reported that with the addition of 1 % bentonite, containing 85 % SiO2+Al2O3 decreases the iron content (0.6-0.7 wt %) in the pellets [5]. The increase in silica content can lead to increases in the steel production cost as reported by Chizhikova et al., 2003, Schmitt, 2005. Different researchers have tried to find alternative to bentonite to overcome problems as mentioned above. In recent times they have focused on organic binders due to its outstanding binding properties and free from acid constituents. Mostly, Carboxymethyl cellulose (CMC), Peridur C-10 and
Peridur CX3, organic polymers, lime and Pridor, domestic CMC, Na lignosulphonate (NLS), starch and glue.

A.I. Arol et al. (1989) comparatively studied bentonite and certain organic binders namely Carboxymethyl cellulose (CMC), Peridur C-10 and Peridur CX3 with magnetite concentrates from Divrii, Turkey. They observed that the organic binders have achieved the wet and dry (673 K) pellet properties. However, the fired compressive strength of pellets produced with organic binders at 1473 K for 120 minutes was found insufficient as these organic binders do not contribute to the compressive strength of fired pellets [6]. Osman Sivrikaya et al. (2012) have focused on the effect of organic binders, (calcined colemanite (Ca₂B₆O₁₁. 5(H₂O)), DPEP06-0007 Polymer and Superfloc A150-LMW) individually and along with combinations on wet, dry, preheated and fired (thermally-treated) pellet strengths. It was concluded that the organic binders alone have satisfy the wet and dry pellet properties, but preheated and fired pellet have resulted in insufficient strengths. Combination of binders (Technical CMC 0.10%+Calcined colemanite 0.66%, DPEP06-0007 Polymer 0.10% + Calcined colemanite 0.66% and Superfloc A150-LMW 0.10% + Calcined colemanite 0.66%) have both sufficient wet and dry pellet qualities and improved preheated and fired pellet strengths [7]. Guanzhou Qiu et al. (2004) have studied the effect of organic polymer (Funa) as binders for agglomeration. It behaves like surfactants and colloids. Organic polymer sharply diminishes the contact angle of the iron ore from 46° to 0° and expands on the surface of the concentrate, this improves the hydrophilic ability due to this, they becomes more negative with addition of Funa indicates that the iron ore particles will not adhere together through electrostatic attraction when Funa is used as binder[8]. Ammasi et.al. (2015) used Na lignosulphonate (NLS) and copper smelting slag (Cu-SS) as a binder with varying compositions. The Pellets prepared with NLS as a binder, it shows a good wet and dry compressive strength and increasing with increasing NLS percentage. Even at higher temperatures the compressive strength has become constant even with an increasing NLS percentage. When The Pellets made with 1.0 wt. of Cu-SS along with 0.5 wt. % NLS, it was observed that the compressive strength was increased with increasing temperature and required compressive strength was attained at 1523 K [9]. J. Kamalabadi et al. (2013) have studied the effect of lime and Pridor binder and compared with the bentonite binder in dried conditions. They were observed that the dry strength of the samples prepared through bentonite binder was higher than that of the samples prepared by the lime binder, strength of the pellet has increased with small amount of addition of peridor binder [10].

Dextrin is an organic binder that has been successfully used in various applications such as a thickening and binding agent in pharmaceutical products and paper coatings. In the present study dextrin is used to study the effect on agglomeration of iron ore at various firing temperatures and induration times. This present study was focused on the effect of firing temperature and induration time on dust generation during mechanical wear of iron ore pellets. The influence of the dextrin binder and its composition on the physical, mechanical and wear rate of the pellets has been investigated and compared with the standard binder bentonite. Furthermore, the particle size distributions in the dust generated during the collision of pellets were investigated. Establish a relationship among the critical diameters of dust particles, and flow behavior of off-gases from the blast furnace.

2. Materials and methods

2.1. Raw Materials

2.1.1. Iron ore concentrate. In the present study, Hematite Iron ore concentrate was collected from Orissa Mining Corporation (OMC). The as-received ore was dried at 378 K overnight. The ore was divided into representative samples of 1.5 kg by sampling methods and sealed in plastic bags, and its nominal chemical composition has been outlined in Table 1. From the chemical composition, Hematite concentrate has a relatively high amount of Fe₂O₃ and aluminum, and silicon oxides are major gauge constituents. Particle size measurements of the iron ore al fines were performed by using
the Vibratory Laboratory Sieve Shaker (Fritsch, Germany) ‘‘Analysette3’’. The required particle sizes that is -75 µm and it was separated from raw materials.

Table 1. Nominal Chemical composition and loss on ignition of raw materials

| Iron Ore Source          | Chemical Composition ( weight percent on dry basis) | Loss on Ignition |
|--------------------------|----------------------------------------------------|------------------|
| Orissa Mining Corporation (OMC) | Fe (Total) 62.63  Fe₂O₃ 89.56  Al₂O₃ 4.09  SiO₂ 3.80  TiO₂ 0.11  MnO 0.01 | 2.43             |
| Bentonite binder         | - 3.96  58.36  21.14 | 6.75             |

2.1.2. Binder. Two types of binders used for this study: bentonite and dextrin. Tests conducted with (0.5 and 1 w.t % of dextrin and I w.t % bentonite) and without binder added to the iron ore and pellets were prepared.

2.1.3. Preparation of Iron Ore Pellets. The green pellets were prepared by mixing of dry concentrate of iron ore particles, required amount of moisture (Water ≈10%) and pre-defined binder percentage, for 5-10 min. Then the mixture is added to the disc pelletizer, and it operated at a speed of 25 rpm to make pellets. The pellets were pulling out from the pelletizer and screened to required size ratio which is between 9-16 mm diameters. The pellets were exposed to the atmosphere for 24 h and then dried in an electric oven at 383 K for four hours. The dried pellets fired in the muffle furnace from room temperature to predefined temperatures ranging from 1173 to 1473 K and indurated at varying time periods (1 and 2 h) to attain the workable strength (ISO 4700 standards), pellets to allow for furnace cooling. Then pellets were allowed to characterization.

2.2. Pellet quality testing
Pellet quality were determined by testing the pellets at different stages of the process. Tests were conducted for the pellets: 1) drop test for dry pellets, 2) compressive strength and porosity for dry and heated pellets, and 3) Hardness for heated pellets 4) wear rate 5) Particle size distribution analysis.

2.2.1. Drop number of dry pellets. 10 dried pellets were utilized to focus the dry pellet drop number by dropping a solitary fired pellet over and over from a stature of 46 cm (18 inch) onto a steel plate. The drop test for a solitary fired pellet was preceded until a split happened on the pellet and the last esteem recorded. A pellet should withstand at least 4 drops [11].

2.2.2. Compressive Strength. Compressive strengths of individual pellets depend on the chemical composition and physical properties of the concentrate, additives used in the pelletizing method, particle size, firing technique, and temperature. A dried pellet is crushed and the maximum load is recorded. It measures the ability of dried pellets to survive handling during the firing process. It should be a mean value at least 2.24 kg/pellet. A fired pellet is crushed and maximum load is recorded. It must satisfy the ISO 4700 standards [12].

2.2.3 Porosity. Total porosity of balls is expressed as a portion of pore volume out of the total volume of the input raw material, the apparent porosity values of iron pellets were determined in accordance Archimedes principle. In the porosity measurements kerosene was used as a medium. It is calculated accordance with the following formula.
Apparent porosity \(= \frac{W - D}{W - (S - s)} \) \quad \ldots \ldots \ (1)

Where, ‘\(D\)’ is the weight of dried piece;
‘\(W\)’ is the weight of oil saturated piece;
‘\(S\)’ is the weight of the piece + thread while immerse in oil, and
‘\(s\)’ is the weight of thread only while immerse in oil

Lower porosity leads to a better particle–particle contact but obstruct the diffusion of oxygen throughout the pellet during reduction process. The porosity of typically fired pellets is in the range of 18 - 32% [13].

2.2.4. Hardness. The hardness of the fired iron ore pellets was determined by using a Vickers hardness tester to measure the microhardness (HV) according to the ISO6507-1 standard. The hardness measurement performed on a cross section of a polished surface by moving from the surface to focal point of the pellets [14].

2.2.5. Mechanical Wear of Pellets. In order to evaluate the mechanical wear and fines generation rate from fired pellets, the method of Muhammad Nabeel (2016) was directly followed. Predefined pellets (60 g) were placed in a metallic jar and rotated with a 300 rpm up to 30 minutes. Effect of wear rate was calculated in two methods as shown in Fig. 1. In method 1 the generated fines were kept in the jar and weight of the pellets was measured at a regular time interval of 5 minutes, so that the mass of the jar was constant throughout the experiment. In method 2, take out formed fines from the pellets at regular intervals. The fines generation was calculated from the mass loss of the pellets after rotation and wear rate was calculated equation (2) [15] accordingly.

\[
\text{Wear rate} \left(\%\right) = \frac{W_{t} - W_{t+\Delta t}}{W_{t}} \times 100 \quad \ldots\ldots (2)
\]

\(W_{t}\) = weight of the pellet before rotation
\(W_{t+\Delta t}\) = weight of the pellet after rotation

2.2.6. Particle Size Distribution. Particles generated from the wear tests have collected from the jar and the Particle Size Distribution of the fines was measured by using scanning electron microscope (SEM) at different magnifications.
3. Results and Discussion
A set of experiment was performed on pellets to investigate the quality of pellets which are made-up of organic binders, concerning drop number, porosity and compressive strength of dry pellet and, porosity and compressive strength of fired pellet.

3.1. Dry pellet drop-number (DPDN)
The DPDN of the iron ore pellets was obtained by using different types of binders, and the results were compared with pellets made using bentonite as binder and pellets made without any binder. Reference bentonite binder was found to be 4.82 as shown in Figure 2, which are slightly over the limit so they are sufficient. However, DPDN values for pellets made without binder was 2.8 and with dextrin (0.5 and 1 Wt. %) addition were determined to be 3.6 and 4.7, without binder and 0.5 Wt. % of dextrin is a little lower than the required value.

![Figure 2. Effect of binders on Dry Pellet Drop number](image)

3.2. Dry pellet compressive strengths (DPCS)
The DPCS of the iron ore pellets was determined by using different types of binders. Bentonite samples were considered as the reference point for strength calculation. The DPCS of the iron ore pellets obtained by bentonite binder was 4.6 kg/pellet which are over the limit, so they are sufficient. However, DPCS values for pellets made without binder and with dextrin addition were determined to be 1.34, 3.9 and 4.3kg/pellet respectively as shown in Figure 3. The pellets made without binder is a lower than the required value. Therefore, they can be considered insufficient, and the production of pellets without binder is not suitable. The DPCS produced with organic binders were found to be much greater than required value 2.24 kg/pellet dextrin was suitable for the pelletization process. On the other hand, the DPCSs of pellets by organic binders were found to be lower than those obtained by bentonite, but they are still greater than the minimum required limit.
3.3. Porosity of Dried pellets

Porosities of pellets produced with different binders were determined after drying. The porosity of the iron ore pellets of bentonite binder has shown 32.8 % which are lower when compared to ISO standards. However, porosity values for pellets made with no binder and with dextrin addition were determined to be 35.6, 33.5 and 33.2 % in all the conditions they have satisfied the required value as shown in figure 4. From the results it is confirm that these pellets can be used for pelletization. Therefore, they can be considered sufficient to the production of pellets without binder. The porosity of the dried pellets produced with organic binder was found close to the bentonite samples.

3.4. Characterization of fired pellets

In thermal heat treatment process, pre-heating strength is the most important factor because the pellets are moving from traveling grate to the rotary kiln. If the pre-heating strength is insufficient, fragments and dust will form from the pellets due to this both pellet quality and operation efficiency will be affected adversely. The properties of the pellets are depending upon the thermochemical conditions.
like firing temperature and induration time. Due to the thermo-chemical reaction, recrystallization and slag formation takes place. Slag phase formed around the pellet and it moves towards the center of the pellet. The slag phase is densely sintered and it contributes to the pellet strengthening.

3.5. Effect of binder, firing temperature and induration time on compressive strength

The compressive test of pellets having different binders was determined, which are produced with without binder, 1.0 % bentonite, 0.5 and 1 % of dextrin addition. The compressive strength of the pellets has increased with increasing firing temperature and induration time. It was observed that the pellets were heated at 1173 K for 1 hr. is 31.32, 98.32, 86.3 and 94.3 kg/ pellet respectively. Further, it was increased to 35.6, 102.36, 98.36 and 110.5 kg/pellet with increasing the induration time to 2 hr. A similar trend was observed at 1273, 1373 and 1473 K as shown in Figure 5, it is due to increasing induration time, the binder melts and fuse the particles, increase the compressive strengths of fired pellets. Pellets fired At 1473 K compressive strengths of without binder, 1% bentonite and 0.5 and 1 % of dextrin were, 145.8, 3.2.32, 238.5 and 275.4 Kg/pellet respectively, these values are increased with increasing induration time up to 2 h 154.23, 350.5, 280.32 and 295.6 kg/pellet, which were the highest values of all results. Reference binder (1% bentonite) gives the best results than the organic binder but at 1% dextrin at 1473 K for 1 and 2 h induration time, and 0.5 % of dextrin at 1473 K for 2 h induration time was satisfy the industrial requirement.

![Figure 5](image)

**Figure 5.** Effect of firing temperature and induration time on Compressive strength of pellets with different binders for (1) 1 hr. and (2) 2hrs

3.6. Porosity of fired pellets

Porosity of pellets made with different binders was determined to determine the effect of different firing temperatures and induration times. With increasing temperature, the porosity decreases for all pellets as shown in Figure 6. The porosity differences between pellets contain dextrin and bentonite was only about 2-3%. The pellets produced without binder and fired at 1173 K have the highest porosity about 31.8 and 31.03 % at 1 and 2hrs induration time respectively, it was observed for pellets produced with 1% bentonite, 0.5 and 1% Dextrin are 27.5, 28.3 and 27.8 % at 1hr and 26.3 28.3 and 27.4 % 2 h induration time respectively at the same temperature. The porosity is decreased with increasing temperature up to 1473 K they are 25.2, 20.3, 21.9 and 21.8 % at 1 h and 24.3 18.5, 21.5 and 21.3 %at 2 h induration time for without binder, 1% bentonite and 0.5 and 1% dextrin respectively. In literature, typical porosities for dry and fired pellets from high-grade iron oxide are mentioned in the range 31-36%, whilst a porosity of 22-30% for fired pellets is associated with good reducibility. The porosities for pellets produced with different binder addition were found in the industrial pellet porosity percentage interval.
Figure 6. Effect of firing temperature and induration time on Compressive strength of pellets with different binders for (1) 1 hr. and (2) 2hrs

3.7. Hardness of fired

The hardness of the fired pellets made with binders (different) and without binder, were determined, and studied the effect of firing temperature and time on hardness. The increasing temperature and induration time shown a pronounced effect on hardness of the pellets. However, it was observed that the hardness values are decreasing from surface to the center/core of the pellet in all conditions. The hardness of fired pellets made with organic binders has shown improvement when compared to without binder and less than the bentonite binder. Figure 7 shows the effect of 1 h induration time on the hardness of the surface at 1173 K is 128, 225, 185 and 218 HV increased to 251, 340, 315 and 332 HV with increasing temperature up to 1473 K for without binder, 1% bentonite and 0.5 and 1% Dextrin respectively and the same trend was observed for 2 h induration time as shown in Figure 8. However, it was found that the hardness values of the surface of all pellets are 3 to 4 times higher than the values observed in the center of the pellet. It is due to the presence of a hematite and slag content on the surface of the sintered pellets.
Figure 7. Effect of firing temperature and 1 hr induration time on hardness of pellets with different binders 1) Without Binder 2) 1% Bentonite 3) 0.5% Dextrin and 4) 1% Dextrin
3.8. Mechanical Wear of Pellets

To evaluate the effect of strength on mechanical wear and dust generation were investigated by using collision test. The results are shown in Figure 9 it can be seen that there was a significant difference between fines generation rates for pellets with good strengths. From the compression test results, 1% of bentonite (302 and 350 Kg/pellet) and 1% dextrin (275.6 and 295 Kg/pellet) at 1473 K for 1 and 2 h induration time, and 0.5 % of dextrin (280 kg/ pellet) at 1473 K for 2 h induration time were satisfying the industrial requirement and rest of compositions were not attaining the required strength so, the pellets were not suitable for DRI process. Wear test was conducted on the above-mentioned compositions. It observed that the fines generated from the pellets decreased with increasing strength which in turn lead to less material loss from the pellets. In method 1, the weight of the pellets maintains a constant throughout the experiment, it can be observed that the wear rate decreases (~2 times) with an increased rotational time of the test experiments as shown in Figure 9 (1). In method 2, removing of formed fines from the jar at regular intervals, the wear rate was increased with increasing the rotational time as shown in Figure 9 (2). The difference in the wear rate between the two methods was affected by the cushioning effect of dust, that means the generated dust in the jar was reduce the wear rate and it reported by several authors. The increase of the wear rate values during the wear tests can have revealed by that a hardness gradient exists in all pellets. It is clear that the harder materials have a higher have a higher resistance to wear.
3.9. Size Distributions of Particulates Generated by wear test
Dust particles were collected from the wear test to study the particle size distribution by using the SEM. 1 gram of each dust sample was taken from the jar to determine the effect of strength on powder fineness of each pellet. From the investigation, the particles were divided into three categories, i.e. fine particles (0–10 \( \mu \)m), medium size particles (10–20 \( \mu \)m) and coarse particles (>20 \( \mu \)m). The size distributions of the dust particle are shown in Figure. From the SEM analysis, high amount of coarse particles was present in the method 2 for all type of pellets as shown in Figure10, which is significantly larger weight fraction than the method 1. Evidence of Coarse particles, collisions is a dominating mechanism during the wear.

4. Conclusions
In the present study, the addition of dextrin binder into pellet mixture was proposed to increase the low pellet compressive strength encountered with the use of organic binders in iron ore pelletizing. The performances of organic binders on physical-mechanical pellet qualities were comparatively tested against bentonite binder performance.
The DPDN produced without binder is a little lower than the required value. Therefore, they can be considered insufficient. The DPDN values of 1 Wt. % of bentonite and dextrin has shown marginally higher value than required value that is 4 and is suitable for pelletization.

The DPCS of pellets by organic binders were found to be lower than those obtained by bentonite pellet but they are still greater than the minimum required limit, due to the presence of Dextrin between the particle to particle.

It was observed that firing temperature, time, compositions were affecting the properties of iron ore pellets. The required compressive strength 250 Kg/Pellet was obtained at 1% of bentonite (302 and 350 Kg/pellet) and 1% dextrin (275.6 and 295 Kg/pellet) at 1473 K for 1 and 2hrs indentation time, and 0.5 % of dextrin (280kg/ pellet) at 1473 K for 2 h indentation time. From this we can conclude that with increasing the indentation time bonding between the particle increases as well as improves the strength.

The porosity of the fired pellets decreases with increasing temperature and indentation time. The porosity differences between pellets contain dextrin and bentonite were only about 2-3%.

The hardness of the pellet varies from surface to the center of the pellet. The surface has shown 3-4 times high hardness compared to the center of the pellet.

The strength of pellets under the current experimental conditions can influence the wear rate of pellets. The wear rate of method 1 decreases (~2 times) and increases in method 2 with an increased rotational time of the test experiments. The difference in the wear rate between the two methods was affected by the cushioning effect.

After wear test the generated particles were characterized and which are marginally coarser in case of method 2 than method 1 from the aforementioned we could say that wear collisions are dominating during wear, were as fine dust particles were observed in case method 1.

References

[1] Taylor S. R 1964 Geochim. Cosmochim. Acta, 28 1273
[2] Pal J, Ghorai S, Goswami M. C, Prakash, S, Venugopalan T 2014 ISIJ International, 54(3) 620
[3] Abouzeid A. Z, Saddik A. A. 1981 Powder Technol. 29 233
[4] Forsmo S. P. E, Apelqvist A. J, Björkman B. M. T, Samskog P. O 2006 Powder Technol. 169 147
[5] Srivastava U, Kawatra S.K, Eisele T.C 2013. Metallurgical and Materials Transactions B 44(4) 1000
[6] Haas L. A, Zahl. R. K, Aldinger J.A 1989 Rep. Investig. / United States. Bur. Mines 9230 21
[7] Sivrikaya O, Arol A.I, Eisele T, Kawatra S. K 2013 Miner. Process. Extr. Metall. Rev. 34 210
[8] Qiu G, Jiang T, Fa K, Zhu D, Wang D 2004 Powder Technol. 139(1) 1
[9] Ammari A, Pal J 2015 Ironmak. Steelmak. 43(3) 203
[10] Kamalabadi khorasani J, Saeidi A, Shafie A 2013 Int. J. ISSI, 10 34
[11] Zhucheng H, Yi L, Jiang T 2012 Powder Technol. 221 284
[12] Kisan M, Sangathan S, Nehru J, Pitroda S. G 2002 Iso 6507-1:1
[13] Amal T, Diana C, Véronique S, Philippe M, Véronique F 2012 Powder Technol. 230 86
[14] Kawatra S.K, Ripke S 2002 J Int. J. Miner. Process. 65 165
[15] Nabeel M, Karasev A , Jönsson 2016 P. G. ISIJ Int. 56 960