High-Carbon Fly-Ash Binders for Iron Ore Pellets

S. K. Kawatra, T. C. Eisele, and D. Banerjee*
Department of Metallurgical and Materials Engineering
Michigan Technological University,
1400 Townsend Drive
Houghton, Michigan, 49931-1295
Telephone: (906) 487-2064

*Illinois Clean Coal Institute
Carterville, IL 62918-0008

ABSTRACT

In the United States, approximately 62 million tons of iron ore pellets are produced every year. The current practice in pellet production is to utilize bentonite (a clay mineral) as a binder for these pellets, with 637,000 tons of bentonite being used in this application every year. This paper reports the first successful use of high-carbon fly-ashes as a replacement for bentonite in iron-ore pellet production. The carbon content of the fly-ash was found to have no detrimental effects on the quality of the pellets produced, and the carbon contained in the pellets will tend to reduce the fuel needed for pellet sintering. The advantages and disadvantages of fly-ash as a binder are discussed.

1. INTRODUCTION

This project investigated the use of Illinois fly-ashes as binders for the iron-ore pellets produced in the Lake Superior iron district, with potential future application to binding other inorganic particulates. The U. S. Geological Survey estimated that 62 million metric tons of iron-ore pellets were produced in the US in 1995, mainly in the Lake Superior district, and that approximately 637,000 metric tons of binder were consumed by this market. With an average price of $58/ton for conventional bentonite binders in 1995, the value of fly-ash based binders can be significant.

Fly-ashes have never been used in this type of application before, with most of the fly-ashes which are currently utilized being sold as cement admixtures. Other applications for some fly-ashes have been developed, but these are generally either not able to utilize more than a small portion of the fly-ash, or have even more stringent quality requirements than cement admixtures. Since iron-ore binders have different requirements than do cement admixtures, this new application will provide a means for utilizing those ashes that are currently unmarketable.

It is particularly important to find applications that can tolerate, or even benefit from, high unburned carbon contents in the fly ash. Carbon is a particularly harmful impurity in fly-ashes used as cement admixtures, because the carbon has a very high surface area and tends to adsorb chemicals such as air-entraining agents that are routinely added to cement.

Since chemical additives are not added to iron ore pellets, this new application for fly-ash was expected to be capable of utilizing otherwise useless high-carbon ashes. The ability to tolerate high carbon contents will become progressively more important for users of Illinois coals in the future, because the adoption of Low-NOx combustors is leading to higher levels of unburned carbon in the fly-ash.

The primary questions in using high-carbon fly-ashes as binders for iron-ore pellets were as follows:

- Does the presence of carbon interfere with the formation of pellets, and can the high-carbon fly-ashes produce pellets of sufficient strength to meet specifications?
- Does combustion of the carbon in the pellets during sintering have any harmful effects on the finished pellet properties?

2. FLY-ASH CLASSIFICATIONS AND PROPERTIES

ASTM classifies marketable fly ashes as either Class F or Class C, which have the following characteristics:\n
- Class F fly ash: Normally produced from burning anthracite or bituminous coal. It will react with calcium hydroxide and water to form cementitious compounds (known as a pozzolanic reaction), but it does not show cementing behavior if water is added without lime or other alkalis.
- Class C fly ash: Normally produced from lignite or sub-bituminous coal. In addition to Pozzolanic properties, it also shows cementitious properties because of a high free lime content. Some Class C fly ashes contain more than 10% CaO as free lime.

For both types of fly ash, ASTM has set a maximum limit of 6% Loss-on-Ignition (LOI). For fly ashes the LOI value corresponds closely to the content of unburned carbon. LOI values higher than 6% are considered unacceptable for use as cement admixtures. The content of SO3 must also be less than 5%, and moisture content must be less than 3%. For Class F fly-ashes the sum of the SiO2, Al2O3, and Fe2O3 analyses must be greater than 70%. For Class C fly-ashes, SiO2+Al2O3+Fe2O3 must be greater than 50%. If the fly-ash fails to meet any of these requirements, then it is neither a Class F nor a Class C fly-ash.

When burned in conventional pulverized-coal burners, western low-sulfur coals generally produce Class C fly-ashes, while Eastern coals typically produce Class F fly-ashes. However, it has become common to blend low-sulfur and high-sulfur coals, and to use advanced burners such as fluid-bed combustors, to reduce sulfur emissions. Low-NOx combustors, which tend to produce ashes with
high levels of unburned carbon, are also becoming more common. These combustion methods often lead to coals which are neither Class C nor Class F. For example, in a fluid-bed combustor ash that was previously studied, SiO₂+Al₂O₃+Fe₂O₃ was only 31.26%, and it contained 46.19% CaO and 12.53% SO₃. This large deviation from the specifications for Class C fly-ash was due to the addition of lime to the fluid-bed combustor as a sulfur absorbent, which is normal practice for this type of combustor.

The binding action of fly-ash is due to “pozzolanic reactions” between the ash particles, lime, and water. These reactions occur because the fly-ash contains a great deal of aluminosilicate glass, which is much more reactive than crystalline materials of similar composition. When combined with water and lime, the particles are in contact with a solution that has a pH higher than 12.7 and a high concentration of Ca²⁺ ions. The SiO₂ tetrahedra that make up the glassy particles react with OH⁻ ions in solution, eventually passing into solution as H₂SiO₄⁻ ions. These react with the Ca²⁺ ions to form precipitates of calcium silicate hydrates. Aluminum oxides react similarly, although they diffuse more rapidly than the silicate ions, and require a higher concentration of calcium ion to precipitate. As a result, the calcium silicates tend to form on the particle surface, while the calcium aluminates and aluminosilicates tend to re-precipitate at some distance from the particle. It is the formation of these precipitates that is responsible for the binding properties of fly-ash undergoing pozzolanic reactions.

If fly-ashes were simple homogeneous materials, the reaction between high-purity aluminosilicate fly-ash would be as follows (using cement chemistry notation where C = CaO; A = Al₂O₃; S = SiO₂; H = H₂O; S = SO₃; z is a variable):

$$\text{AS}_2 + 3\text{CH} + z\text{H} \rightarrow \text{C-S-H}_{z.5} + \text{C}_2\text{ASH}_8$$

The products produced by this particular reaction are an amorphous calcium silicate hydrate gel (C-S-H) and gehlenite hydrate (C₂ASH₈). However, fly-ashes are not simple homogeneous materials, and so this is by no means the only reaction occurring in actual fly-ashes, for two reasons: (1) It does not account for the presence of sulfur trioxide or iron oxide, and (2) It assumes complete reaction, and no side reactions or intermediate phases. With real fly-ashes, a variety of other phases also form, with the most important phases including calcium aluminum hydrate (C₆A₁₉), ettringite (C₃A·3CS·H₁₉), and calcium monosulfatehemihydrate hydrate (C₃A·CS·H₁₂), which also act as binders upon formation. The rates of these reactions depend strongly on many factors, including composition, intrinsic fly-ash characteristics, temperature, and the presence of chemicals that act as hardening accelerators or retarders.

Class C fly ashes will react directly with water to form cementitious materials, because the Class C ashes contain calcium oxides that can initiate the pozzolanic reactions. However, Class F fly ashes, and other low-calcium ashes such as those used in this study, will only form a cement if lime is added.

3. EXPERIMENTAL PROCEDURE

Materials

The material being pelletized in these experiments was magnetite concentrate (10% moisture, ground to 80% passing 25 μm), provided by a mine in the Lake Superior iron district. The magnetite was divided into samples of approximately 3 kg using a riffle splitter, and the individual samples were sealed into plastic bags to prevent moisture losses.

Fly-ash used in these experiments was from both conventional and low-NOₓ pulverized coal burners operated by an Illinois utility. The ashes were collected as a dry powder from the electrostatic precipitators. These ashes had the compositions shown in Table 1.

The fly-ashes used were low in calcium, and varied in their carbon contents depending on the type of combustor. The conventional burners produced an ash containing 6.90% carbon, while the Low-NOₓ burners produced an ash with 11.11% carbon. These fly-ashes were ASTM Class F ashes, which do not have binding properties when used alone. They were therefore combined with calcium hydroxide, which would chemically react with the fly-ashes in the presence of water to give them binding properties. Calcium hydroxide ("slaked lime") was used instead of calcium oxide ("quicklime") because calcium oxide expands as it reacts with water, which was found to cause the pellets to crack or turn to powder upon drying or firing.

In related studies, the authors found that salts such as calcium chloride and magnesium sulfate can act as accelerators for the reaction of fly-ash with water and calcium hydroxide, speeding the binding process. No accelerators were used in the experiments described here. However, since the ashes were collected dry, they contained water-soluble salts that could act as accelerators to some extent.

In addition to the two raw ashes collected from the plant, a portion of the "medium-carbon" material was treated by froth flotation, to reduce its Loss-on-Ignition (LOI) value to a very low level. This also resulted in the removal of water-soluble salts, and so this fly-ash contained a lower content of potential accelerators than the raw, unprocessed ashes. Ashes with intermediate LOI values were prepared by mixing ashes with different carbon contents.
Table 1: Chemical Analyses of the Illinois fly-ashes being used, with the analysis of a typical western bentonite included for comparison purposes. The “Loss on Ignition” value is the weight loss upon heating the sample to 950°C for an extended period, and is a measure of unburned carbon content in the fly-ash. A “Decarbonized” fly ash was also produced from the “Medium-Carbon” ash, by removing the carbon using a froth flotation process.

<table>
<thead>
<tr>
<th></th>
<th>Class F Medium-Carbon</th>
<th>Class F High-Carbon</th>
<th>Western Bentonite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon Dioxide, SiO₂</td>
<td>50.07</td>
<td>48.00</td>
<td>39.62</td>
</tr>
<tr>
<td>Aluminum Oxide, Al₂O₃</td>
<td>24.41</td>
<td>23.87</td>
<td>23.16</td>
</tr>
<tr>
<td>Iron Oxide, Fe₂O₃</td>
<td>9.51</td>
<td>9.34</td>
<td>5.49</td>
</tr>
<tr>
<td>SiO₂ + Al₂O₃ + Fe₂O₃</td>
<td>83.99</td>
<td>81.21</td>
<td>68.27</td>
</tr>
<tr>
<td>Calcium Oxide, CaO</td>
<td>2.72</td>
<td>2.63</td>
<td>9.63</td>
</tr>
<tr>
<td>Magnesium Oxide, MgO</td>
<td>0.85</td>
<td>0.84</td>
<td>2.11</td>
</tr>
<tr>
<td>Sodium Oxide, Na₂O</td>
<td>0.61</td>
<td>0.55</td>
<td>1.06</td>
</tr>
<tr>
<td>Potassium Oxide, K₂O</td>
<td>2.68</td>
<td>2.61</td>
<td>0.39</td>
</tr>
<tr>
<td>Titanium Dioxide, TiO₂</td>
<td>1.22</td>
<td>1.25</td>
<td>1.25</td>
</tr>
<tr>
<td>Manganese Dioxide, MnO₂</td>
<td>0.05</td>
<td>0.05</td>
<td>0.01</td>
</tr>
<tr>
<td>Phosphorus Pentoxide, P₂O₅</td>
<td>0.09</td>
<td>0.12</td>
<td>2.20</td>
</tr>
<tr>
<td>Strontium Oxide, SrO</td>
<td>0.15</td>
<td>0.14</td>
<td>0.39</td>
</tr>
<tr>
<td>Barium Oxide, BaO</td>
<td>0.16</td>
<td>0.16</td>
<td>0.53</td>
</tr>
<tr>
<td>Sulfur Trioxide, SO₃</td>
<td>0.61</td>
<td>0.54</td>
<td>5.93</td>
</tr>
<tr>
<td>Loss on Ignition</td>
<td>6.90</td>
<td>11.11</td>
<td>13.24</td>
</tr>
</tbody>
</table>

Table 2: Descriptions of the tests used for evaluating pellet quality. The “wet-knock” and “wet-crush” values are primarily functions of the pellet moisture content and how the pellets are made, and did not vary in any systematic way in these tests. The “dry-crush” values are a function of the binder, and were the measurements of greatest interest in this study.

<table>
<thead>
<tr>
<th>Test</th>
<th>Procedure</th>
<th>Use of Data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wet-knock</td>
<td>A single freshly-made (undried) pellet is dropped repeatedly from a height of 18 inches onto a steel plate. The number of drops required for fracture is recorded. This is repeated for 20 pellets, and the results averaged.</td>
<td>Measures the ability of the wet pellet to remain intact during handling.</td>
</tr>
<tr>
<td>Wet-crush</td>
<td>A single freshly-made (undried) pellet is compressed using an Instron compression test machine. The load required to fracture the pellet is recorded. This is repeated for 20 pellets, and the results averaged.</td>
<td>Measures the ability of the wet pellets to retain their shape during handling.</td>
</tr>
<tr>
<td>Dry-crush</td>
<td>Pellets are dried at elevated temperature (&gt;105°C) for at least 1 hour (normal industrial practice is to dry for approximately 24 hours to ensure complete drying). Single pellets are then compressed using an Instron compression test machine. The load required to fracture the pellet is recorded. This is repeated for 20 pellets, and the results averaged.</td>
<td>Measures the ability of dried pellets to survive handling during the firing process. Should be at least 5 pounds force per pellet. This is the most critical measurement.</td>
</tr>
</tbody>
</table>

These tests used a fly-ash dosage of 0.66% of the magnetite weight, and a Ca(OH)₂ dosage of 1.28% of the magnetite weight. No supplemental accelerators were added, so that inherent differences between the various fly-ashes could be more clearly seen. Since the ashes were from combustion of the same coal, their primary chemical difference was in their unburned carbon content.

The fly-ashes and fly-ash mixtures used for these experiments were as follows:

- Decarbonized, LOI = 0.94% (2 tests)
- 50% Decarbonized/50% medium carbon, LOI = 3.92
- Medium carbon, LOI = 6.90%
• 50% medium carbon/50% high carbon, LOI = 9.00%
• High Carbon, LOI = 11.11% (2 tests)

Pelletization Procedure

The procedure used for forming the pellets was developed by industry to evaluate various types of pellet binders, and was designed to closely reproduce the conditions that exist in the full-scale plant.

First, the fly-ash and calcium hydroxide (reagent grade, 98% purity) were measured out, and mixed for 5 minutes using a high-energy Spex mixer. The Spex mixer consisted of a cylindrical vessel with an inside diameter of 1.625 inches (4.13 cm), and a height of 2.5 inches (6.35 cm). This was shaken with an oscillatory motion at 14.4 cycles/second, with an amplitude of 2 inches (5.1 cm). After preparing the fly-ash/calcium hydroxide binder, the magnetite was placed in a kneader-mixer, which was used to combine the magnetite with binder. The mixture was then mixed for 5 minutes. After mixing, the magnetite/binder mixture was allowed to stand for a period of 1 hour to allow binding reactions to begin. The mixed material was then delumped through an 8 mesh (2.36 mm) screen before pelletizing.

Pelletization was carried out using a pelletizing drum with the dimensions shown in Figure 1, rotating at 25 rpm.

To form pellets, a small amount of the material was added to the rotating pelletizing drum to create pellet “seeds”. The seeds were moistened with water mist while adding additional material to enlarge them into pellets. The pellets were removed from the drum periodically, and screened to control the pellet diameter. This procedure was continued until 1-2 kg of finished pellets were produced with diameters between 1/2 inch (1.27 cm) and 7/16 inch (1.11 cm) for pellet testing. The time required for pelletization was 20 minutes. The finished pellets were then immediately dried at 105°C, and held at this temperature for 20 to 24 hours to ensure that they were completely dried.

The pellets were tested using the procedures given in Table 2. For each value reported, the mean and standard deviation were determined for 20 pellets. The error bars given on the graphs are 95% confidence intervals, calculated using the standard t-distribution.7

Once pellets were produced and dried, they were sintered at 1200°C, which is the sintering temperature used by the plant. The procedure was to fill a shallow crucible with a single layer of approximately 30 dried pellets. This crucible was then placed in a high-temperature electric furnace at room temperature. The furnace was heated from room temperature to the target temperature over a period of 15 minutes. This initial heating step was done to simulate the pre-heating stage used in industrial practice. It was found that when the pellets were placed directly into a hot furnace without preheating, the resulting pellets had unsintered “cores” and were markedly weaker than pellets made with the preheating step.

After preheating, the pellets were held at the target temperature for 10 minutes, and then immediately removed from the furnace to cool rapidly in the open. Once cooled, the pellets were tested to determine their compressive strengths. The results were calculated as an average over 25 pellets.

4. RESULTS AND DISCUSSION

In industrial practice, pellets are considered to be of satisfactory quality when their dried compressive strengths are greater than 5 pounds force (22.2 Newtons) per pellet, and their fired strengths are approximately 400 pounds force (1776 Newtons) per pellet. Using the fly-ash binders, it was shown to be possible to achieve these values.

By mixing the raw Class F ashes with each other and with the decarbonized ash, it was possible to vary the carbon content of the fly-ash over a wide range, up to a maximum of 11.11%. As the carbon content of the ash was increased, there was if anything a slight increase in
the strength of the pellets dried at 105°C, although any change was so small that it is not statistically significant, as shown in Figure 2. Upon firing at 1200°C, a small tendency for a change in strength with changing carbon contents was observed, which can be seen in Figure 3. It was originally feared that high carbon content in the fly-ash would cause pellets made with fly-ash to weaken or crack during sintering, due to combustion of carbon particles. However, the magnitude of the change observed was comparable to the random variations in fired pellet strength, and was therefore not statistically significant. This indicates that iron-ore pelletization can utilize fly-ashes with carbon contents as high as 11.11%, which are too high in carbon to be suitable for other applications.

The combustion of the carbon during pellet sintering provides heating value, and so the use of high-carbon ashes as binder will slightly reduce the fuel requirements for the sintering process. The dosage of fly-ash is similar to the dosage of bentonite which is currently used in industrial practice. Since fly-ash is readily available...
from power plants located close to iron ore pelletizing operations, it can be cheaply obtained at a low shipping cost. However, the fly-ash needs calcium oxide or hydroxide additions in order to act as a binder, and the need to add calcium compounds will increase the cost of this type of binder. The most likely application for fly-ash binders is therefore the production of fluxed pellets, which are pellets with lime or limestone added to act as flux in the blast furnace. The use of fluxed pellets is becoming more common, since they simplify the furnace operation and provide better contact between the pellets and the flux. Since the flux can provide the alkali needed to produce binding properties in fly-ash, this is an application that is well-suited for fly-ash binders.

5. CONCLUSIONS

Class F fly-ash can be used to produce iron-ore pellets with strengths comparable to the strengths obtained using conventional bentonite binder added at the same dosage. The benefit of this is that the fly-ash is produced at sites close to iron-ore producers, and so shipping costs will be much lower than that of the bentonite which must be shipped from considerable distances. The main limitation of the fly-ash is that it requires the addition of calcium hydroxide or calcium oxide before it can exhibit any binding properties. However, modern iron-ore pellets are often produced with several percent of calcium oxides added as flux, and so the calcium will already be present for activating the fly-ash binder.

6. Acknowledgments

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