INTRODUCTION

The standard blast furnace pellet grade produced by LKAB in Malmberget and supplied to Nordic customers (MPBO) is manufactured from high grade magnetite concentrate with minor additions, mainly olivine. Outstanding operating results have been proven over the past twenty-five years, but nevertheless work is on-going to further optimize pellet properties to future demands. In this investigation four modifications of MPBO were tested in standard laboratory tests and in the LKAB experimental blast furnace (EBF). Some of the modifications have also been tested in full scale operation.

From laboratory testing it is known that also small changes in pellet composition can have some effect on pellet properties such as reduction strength and reducibility, and this can be observed for the MPBO modifications in this study. In full scale testing, and also in EBF tests operating results are quite similar for all MPBO versions. Process stability is good and no difference in the need of reductants can be observed. From full scale testing it is difficult to deduct whether or not there are differences in the amounts of screened-off fines or dust in the top gas.

To further investigate if the differences in pellet properties observed in lab testing can be linked to any variation in material strength or degradation on samples from the LKAB experimental blast furnace a relatively large number of probe samples were retrieved during operation. Previously no thorough investigation was performed to evaluate the reproducibility of test results on probe samples. Therefore an effort is made here to analyze how results vary between pellet grades compared to the variations that can be observed between sub-samples of each type, originating from different positions along a furnace diameter and sampled at different times and under varying process conditions.
MATERIAL

The LKAB olivine pellet (MPBO) supplied to Nordic customers and produced in Malmberget is used in this investigation. The effect of small additions of quartzite and limestone was tested. Contents of SiO$_2$, CaO and MgO were varied within a range of 0.2% respectively. For the EBF-tests, four pellet grades with adjusted composition were produced in a pilot scale pelletizing plant in Malmberget.

Previous lab scale trials indicate that a small quartzite addition might improve the mechanical strength (according to ISO4700 and ISO3271) and that it improves reduction properties, for example resulting in lower pressure drop in the reduction under load test (ISO7992). Limestone was also added, primarily to compensate for the extra SiO$_2$ added to the blast furnace slag. The CaO has a negative effect on reduction strength but helps improve reducibility. To maintain a high iron content, the olivine addition was decreased, resulting in a somewhat reduced MgO-content. From laboratory testing we know that this has a favorable effect in the low temperature disintegration test (ISO13930).

Target values for the chemical analysis of the four pellet grades produced for the EBF-test and results from standard pellet tests are presented in Table I. MP164 is the reference pellet. To MP161 and MP162 quartzite and limestone are added and the olivine addition is decreased. MP163 is produced without any extra limestone to make up for the quartzite addition.

Table I Chemical composition and mechanical and metallurgical test results

<table>
<thead>
<tr>
<th>MP161</th>
<th>MP162</th>
<th>MP163</th>
<th>MP164</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical composition</td>
<td>SiO$_2$</td>
<td>%</td>
<td>1.85</td>
</tr>
<tr>
<td></td>
<td>MgO</td>
<td>%</td>
<td>1.2</td>
</tr>
<tr>
<td></td>
<td>CaO</td>
<td>%</td>
<td>0.5</td>
</tr>
<tr>
<td>ISO4700 Crushing strength</td>
<td>daN</td>
<td></td>
<td>223</td>
</tr>
<tr>
<td>ISO3271 Tumble strength</td>
<td>&gt;6.3mm</td>
<td>%</td>
<td>93,0</td>
</tr>
<tr>
<td></td>
<td>&lt;0.5mm</td>
<td>%</td>
<td>6,0</td>
</tr>
<tr>
<td>ISO13930 Low temperature reduction disintegration</td>
<td>&gt;6.3mm</td>
<td>%</td>
<td>34</td>
</tr>
<tr>
<td></td>
<td>&gt;3.15mm</td>
<td>%</td>
<td>61</td>
</tr>
<tr>
<td></td>
<td>&lt;0.5mm</td>
<td>%</td>
<td>23</td>
</tr>
<tr>
<td>ISO4695 Reducibility</td>
<td>$R_{40}$</td>
<td>%/min</td>
<td>0.49</td>
</tr>
<tr>
<td>ISO7992 Reduction properties</td>
<td>$R_{40}$</td>
<td>%/min</td>
<td>1.08</td>
</tr>
<tr>
<td></td>
<td>$p_{max}$</td>
<td>mmWG</td>
<td>13</td>
</tr>
<tr>
<td>ISO4698 Free-swelling</td>
<td>%</td>
<td></td>
<td>16</td>
</tr>
</tbody>
</table>

The actual chemical composition was very close to the target. Most mechanical and metallurgical test results vary according to expectations. In most tests, MP163 reaches the best results, or at least it has the best strength, while it also has somewhat lower reducibility than the other grades. Low temperature disintegration (ISO13930) is high for all pellet types.

EXPERIMENTAL EQUIPMENT

The LKAB experimental blast furnace in Luleå (EBF) was built and commissioned in late 1997, primarily for product development of iron ore pellets. With the objective to investigate different pellet types and numerous other aspects of blast furnace production eighteen campaigns have now been completed. Figure 2 gives a short description of the furnace. The EBF is further presented in other papers\textsuperscript{1,2}.

Shaft probes are installed at three different levels on the EBF. Two of the probes go horizontally through the furnace, one in the upper shaft, and one in the lower shaft. The third is an inclined probe, installed at bosh level. The three probes can be used for sampling of materials or for measuring temperatures during operation and retrieving furnace gas for analysis. The material probe has an inner diameter of 70mm and reaches across the furnace diameter. It is emptied into a cylindrical container to create sub-samples. The probes are water cooled and purged with nitrogen which ensures quite rapid cooling of the material inside. Ideally, completely filled probes would give five sub-samples from the upper probe and six from the lower. The material probes are usually not completely filled. The normal amount of sub-samples generated from each probe, are three to four from the upper probe and four to five from the lower.
Number of tuyeres 3
Hearth diameter 1.4m
Height of heart 1.7m
Hearth volume 2.8m³
Working height 5.9m
Working volume 9.0m³
Bosh height 1.0m
Bosh angle 79°
Diameter at belly 1.7m
Diameter at stock line 1.0m
Shaft angle 86°

The upper shaft probe is situated 1.1m below stock line
The lower shaft probe is situated 2.5m below stock line

To determine reduction strength, samples (typically 300g) are tumbled in a rather narrow tube. We call it “I-tumbling”. Test equipment and test procedure are described in Figure 2. The equipment is frequently used in LKAB for testing material strength on reduced material from probe samples from the EBF. It is also routinely used to check the strength on samples reduced by the ISO4695 reducibility test.

I-tumbling is carried out in a vertical rotating steel tube that rotates 600 times at a revolution of 20 rpm. The test will in this paper be referred to as ITH-test.

TEST WORK

Each pellet grade was tested in the EBF for three or four days. What is considered to be “normal operating parameters” were used. Typically the probes have been operated about once or twice every day previously, to either retrieve material samples or to measure temperature and make gas analysis. In this investigation material samples were taken up to four times a day with the upper probe and once a day with the lower.

After dividing the material from a probing into sub samples the material was sieved (on 0.5mm, 3.3mm and 6.3mm sieves) and each fraction weighed. The >6.3mm material was sorted, by manually picking out pieces of coke and additives into separate sub-samples. Chemical analysis were made on most samples and pellets (>6.3mm) from the upper probe were tested for reduction strength in the ITH-test.

RESULTS AND DISCUSSION

EBF operation

Some key parameters for the EBF test are listed in Table II. During the trial the blast parameters, including PCI, were held constant. In order to evaluate any differences in the amounts of reductants (and also to simulate real blast furnace operation) an effort was made to find “the minimum coke requirement” for each pellet grade.
Therefore there are a few occasions when we go too far and the furnace gets a little cool and extra coke is then added. Table II shows very similar results for all four pellet grades when the averages are compared.

All the pellet grades are considered to perform well and no differences in process stability were observed based on gas utilization, pressure drop over the furnace, burden descent rate and hot metal quality. The coke consumption is almost identical for all four periods during the tests. More PCI was injected in the MP164 period, due to problems in the injection system and not because we needed extra coal. The excess coal resulted in higher hot metal temperature, carbon and silicon. No other differences in pig iron and slag composition were observed between the periods. Alkali outtake was within normal variations and considered good with all pellet grades but slightly higher during the MP163 period. The amount of generated flue dust was relatively constant for all materials, except for MP162 when less dust left the furnace.

### Table II  EBF Process parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Plan</th>
<th>MP161</th>
<th>MP162</th>
<th>MP163</th>
<th>MP164</th>
</tr>
</thead>
<tbody>
<tr>
<td>Production rate</td>
<td>tHM/h</td>
<td>1.55</td>
<td>1.56</td>
<td>1.56</td>
<td>1.57</td>
<td>1.53</td>
</tr>
<tr>
<td>Blast volume</td>
<td>Nm³/h</td>
<td>1715</td>
<td>1713</td>
<td>1715</td>
<td>1716</td>
<td>1706</td>
</tr>
<tr>
<td>Blast temperature</td>
<td>°C</td>
<td>1225</td>
<td>1224</td>
<td>1224</td>
<td>1224</td>
<td>1224</td>
</tr>
<tr>
<td>PCI</td>
<td>kg/tHM</td>
<td>130</td>
<td>133</td>
<td>134</td>
<td>133</td>
<td>138</td>
</tr>
<tr>
<td>Coke rate</td>
<td>kg/tHM</td>
<td>400-410</td>
<td>402</td>
<td>403</td>
<td>404</td>
<td>403</td>
</tr>
<tr>
<td>Dust (dry in dust bin)</td>
<td>%</td>
<td>4.2</td>
<td>3.3</td>
<td>4.1</td>
<td>4.1</td>
<td></td>
</tr>
<tr>
<td>Alkali output</td>
<td>%</td>
<td>75</td>
<td>89</td>
<td>100</td>
<td>81</td>
<td></td>
</tr>
<tr>
<td>HM temp</td>
<td>°C</td>
<td>1430</td>
<td>1434</td>
<td>1432</td>
<td>1430</td>
<td>1441</td>
</tr>
<tr>
<td>HM C</td>
<td>%</td>
<td>4.1</td>
<td>4.10</td>
<td>4.09</td>
<td>4.07</td>
<td>4.14</td>
</tr>
<tr>
<td>HM Si</td>
<td>%</td>
<td>1.5</td>
<td>1.6</td>
<td>1.65</td>
<td>1.52</td>
<td>1.77</td>
</tr>
<tr>
<td>HM S</td>
<td>%</td>
<td>&lt;0.06</td>
<td>0.051</td>
<td>0.051</td>
<td>0.064</td>
<td>0.041</td>
</tr>
</tbody>
</table>

**Probe samples**

Typically, enough material for four sub-samples was retrieved with the upper probe and four or five by the lower. Usually the first sub-sample contains more coke than the others and the first two sub-samples also contain less fines. In Figure 3 and 4 average distributions of pellets, coke, slag formers and amount of fines (a mixed material) are shown, based on all sub-samples from all the samplings in this investigation. Only three samplings by the lower probe gave five sub-samples.

![Figure 3 Sub-sample 1-4 upper shaft probe](image)

![Figure 4 Sub-sample 1-5 lower shaft probe](image)

Reduction degree for pellets varies between 10 and 25%. Pellets from sub-sample 1 have the highest reduction degree and pellets from sub-sample 4 the lowest, as seen in Figure 3. There is no difference in average reduction degree between the pellet grades. It should be noted that the amount of fines is relatively limited. The fraction <3.3mm is only a few percent of the total sample (1-3 % in the upper probe and 2-6 % in the lower probe). No significant differences in amount of fines (fraction below 3.3mm) were detected between pellet grades, which could be correlated to the small decrease in amount of dust for MP162 shown in Table II. Chemical composition of the fine material is similar for all pellet grades.
As the first sub-sample (or second for the lower probe) contains the largest amount of coke and as more coke is charged in the centre we assume that these samples originate from the middle part of the furnace. Apparently we don’t manage to get out much material from the opposite side of the furnace. Figure 5 show where the origin of each sub-sample is believed to be.

![Figure 5 Upper and lower shaft probe](image)

**Reduction strength of probe samples compared to laboratory testing**

Results from ITH-testing of probe samples and of samples reduced according ISO4695 are presented in Figure 6. The average results for sub-samples 1, 2 and 3 from the upper probe have been calculated. Sub-sample 4 was omitted as it sometimes gets contaminated by material stuck in the furnace wall, where the probe is entering. Between 25 and 40 sub-samples were tested and included for each of the four pellet grades. Laboratory data are based on a single test run. There is a significant difference in disintegration between laboratory reduced material and furnace material, probably much due to the difference in degree of reduction. It is interesting to note though, that the ranking between the four materials is similar, with MP163 having the best strength after reduction, and MP162 the lowest. Results from other laboratory tests of reduction strength show a similar relation, as indicated by Table I.

![Figure 6 Probe samples compared to laboratory reduced pellet](image)

It thus seems possible to correlate differences in average strength of a large number of sub-samples to similar results in laboratory testing but results are not statistically verified. There is a large variation between samples with a standard deviation (1 sigma) of 3-5 for the fraction <0.5mm and 10-12 for the >6.3mm fraction. Results from the ITH-test varies both between sub-samples from the same probing, as can be expected when they origin from different radial positions in the furnace, and between different probings for a given pellet grade, possibly due to variations in process conditions.

**Reduction strength of probe samples correlated to process conditions**

An effort was made to try and correlate process conditions during probe sampling to ITH-test results, using time-plots and multivariate data analysis (using Simca-P+ 11 software). Process data from the furnace are logged during operation. In Figure 7, 10, 13 and 14 results from the ITH-tests are plotted together with some key process parameters for each period (i.e. pellet grade). It is indicated in the time-plot when each experimental period starts and stops. The stock line level at charging is plotted as any periods of process disturbances and uneven burden descent are clearly visible. Gas utilization is shown as is potassium content of the slag and hot metal silicon, to indicate variations in heat level. Average, minimum and maximum amount of >6.3mm material from ITH-testing of the sub-samples are presented for each probing. From these plots the rather large variations in results between samples is evident. It can also be observed that the test result in the ITH-test often show better reduction strength on samples retrieved when the process is running a little cool (low silicon content in the hot metal). Similar observations where made from evaluations of differences in pellet degradation caused by changes in PCI®.
Figure 8, 11 and 16 are generated in the Simca software, by multivariate analysis (mva) on a set of process data from the hour prior to each probing. The dataset was selected to represent what process conditions each sample has been subjected to. It contains gas temperature measurements and gas analyses. It has been used to identify whether or not any of the probings have been done under process conditions that deviate from the normal and to identify changes in the process. Generally the fit of the models are not so great, as often experienced in this type of analysis on process data. All results should therefore be considered as indications. In case the process data form groups within a test period, average results from the ITH-test have been compared. The results are plotted in Figure 9, 12 and 17.

**MP161**

Figure 7 Process parameters and ITH-test results expressed as fraction > 6.3mm for MP161

![Figure 7 Process parameters and ITH-test results expressed as fraction > 6.3mm for MP161](image)

Figure 8 shows that the process conditions during the first period (MP161) form two groups and from Figure 9 it is seen that the differences in process conditions also effects the ITH-test results. Group 2 consists of the last four probings, performed on October 1st. Also Figure 7 shows that they are different from the rest. The material in group 2 has better reduction strength and the heat level of the process is low, with low hot metal silicon, low gas utilization and uneven burden descent. From the Simca analysis the main differences between the groups are temperatures generally and top gas composition (CO and CO$_2$). The degree of reduction was a little higher for group 2, 22% on average compared to 19% for group 1.
MP162

According to Figure 10 the process was stable during this period except for a shorter period when the silicon in hot metal decreased, which indicate a colder furnace, but no probe sampling was made at that time. The probe samples from the middle part of the period had a comparatively large deviation between the sub samples.

![Figure 10 Process parameters and ITH-test results expressed as fraction > 6.3mm for MP162](image)

In Figure 11 results from the mva analysis in Simca is presented. From process parameters it is possible to distinguish three groups but, they are not correlated by the time for sampling. Instead process parameters have shifted during this period. The difference is basically related to heat level and Figure 10 shows that the variation is very small. There is no significant difference in the ITH-test results between the groups.

![Figure 11 Process data MP162](image)

MP163

Figure 13 shows a rather stable process with few disturbances during the period. No groups could be identified by the mva that was attempted, at least not from the two main components of the model. According to the time-plot the first three samples show greater degradation than the others and the last sample on October 17 shows lower degradation and decreasing silicon content in the hot metal. It is possible to visualize this by mva results by choosing lower order components. For this period a large number of model components were needed and each explains a rather small fraction of the variations.

![Figure 12 ITH-test results for group 1-3](image)
According to Figure 14 this is a period of stable operation. Two groups are formed in the Simca-analysis on process data as seen in Figure 15. The main difference between the two groups (four first samples make up group one and the five remaining is included in group two) is that the gas distribution changes. Wall temperatures shift to higher values on the side where the probe is situated in the later part of the period. ITH-test results indicate higher strength for samples from the earlier period, when group averages are compared. Reduction degrees are similar for both groups. Figure 14 suggests that the reason for the difference in ITH-test results might be the one sample taken on October 10th. By evaluating mva results further it is possible to identify that what differs between this sample and the rest is a higher heat level of the process, although this is not so clear from Figure 14. As the first four samples and the last four has similar reduction strength it can also be concluded that the shift in gas distribution has little influence on the results.
CONCLUSIONS

- All tested pellet grades preformed well in the EBF test. The process conditions were stable. Small adjustments in coke rate were made in order to optimize and control the heat level of the blast furnace. The material sampling program went well.
- The rather small modifications in composition of pellets, resulting in differences in pellet strength in laboratory testing are also noticeable as differences in reduction strength of material sampled from the experimental blast furnace.
- The small variations in pellet strength between the pellet grades did not result in differences in blast furnace performance.
- It has not been possible to link the small variations in pellet strength to differences in the amounts of dust in the top gas.
- The heat level in the blast furnace process influences the reduction strength on probe samples. Therefore it is important to keep this in mind when evaluating and comparing samples. It should be possible to set up criteria for what process conditions need to be met.
- No other process variations (changes in gas distribution) experienced in this investigation had significant impact on reduction strength.

REFERENCES

3. S Wold, “Chemometrics; what do we mean with it and what do we want from it?” Chemometrics and Intelligent Laboratory systems 30 (1995), SSDI 0169-7439(95)00042-9, pp 109-115