1. Introduction

Basic oxygen practice in general, uses burnt lime as a coolant as well as a flux. However, lime charging often creates problems in operation due to its high melting point (~2700°C), poor dissolution property, fines generation, hygroscopic nature. Some plants also employ a limited quantity of iron ore as coolant, along with burnt lime. Several investigators have tried to utilize iron oxide in basic oxygen steel making in the form of pellets, briquettes or sinter, with a view to increase total iron oxide content in slag and waste utilization. Guangqing Zuo has reported that softening point of BOF slag becomes lower with high FeO and Fe₂O₃ content. Majority of the above investigators found faster refining, improved metallic yield, lower scrap consumption, reduced oxygen consumption etc.

Some of them found high to medium slag foaming and slopping, which could be and be controlled through modified lance practice. None of the above investigators had tried using fluxed agglomerates of iron ore. Use of composite agglomerate of iron oxide and lime in BOF process is likely to offer the following two benefits:

- Easy formation of primary slag, facilitating onset of refining reaction and hence reduction of tap to tap time.
- Better utilization of wastes like LD sludge, lime fines etc.

Choi and Kim used lime pellets, coated with iron oxides (a mixture of mill scale, yellow soil and silica) to enhance slag formation in the BOF. A low-melting slag was formed by iron oxides and a part of burnt lime at the initial stage of blowing. This low melting slag acted as a seed for further slagging of the lime and thus improved the refining efficiency and decreased the frequency of slopping. A plant trial with the above pellets at Pohang Works, in a 100-ton converter showed multiple benefits such as lower lime consumption, enhancement of metallic yield, increase in slag basicity and improvement in dephosphorization, compared to the conventional process. However, the difficulties encountered in coating of lime pellets restricted its commercialization.

The authors developed a binderless process for the preparation of Fluxed Lime-Iron oxide Pellets (FLIP) at room temperature in which hydrated lime is reacted with CO₂ gas to develop the cold strength. The operating mechanism for this room temperature strengthening is presented in brief as follows:

When the lime (CaO), present in the base mixture, comes in contact with water, added before pelletization, reacts to form calcium hydroxide:

\[ \text{CaO (s)} + \text{H}_2\text{O (l)} = \text{Ca(OH)}_2 \]

On pelletization, this calcium hydroxide provides primary bonding amongst the particles and imparts strength to the green pellets. During subsequent treatment of the air-dried green pellets by a stream of CO₂, the Ca(OH)₂ undergoes the following reaction:
\[ \text{Ca(OH)}_2 (s) + \text{CO}_2 (g) = \text{CaCO}_3 (s) + \text{H}_2\text{O} (l/g) \]  

(2)

Due to the above reaction, a hard CaCO\(_3\) phase is formed in the pellet. During transformation of Ca(OH)\(_2\) to CaCO\(_3\), a considerable volume expansion occurs.\(^{16,17}\) The expanded volume is accommodated by the pores of the pellets and hence increased the compactness of the pellet and accordingly, increase in strength.

The FLIP thus produced has been intended for use in BOF towards partial replacement of burnt lime. Accordingly, this paper focuses on the high temperature characteristics of the developed pellets to find its suitability in BOF.

2. Experimental

The details of preparation of the pellets and their strengthening have already been presented elsewhere.\(^{16}\) However, for the sake of completeness, a brief account of the same is presented as below.

Iron ore fines collected from Barbil, India was dried and classified into two broad size ranges \(-72\) and \(-30+72,\) ASTM mesh. The details of size distribution in the above two ranges are given in Tables 1 and 2 respectively. Commercial grade lime fines of \(-30\) mesh was used in the current study. The chemical analyses of both iron ore fines and lime fines are presented in Table 3.

| Size range, ASTM mesh | Wt % |
|-----------------------|------|
| \(-200\)              | 80   |
| \(-170+200\)          | 4.2  |
| \(-150+170\)          | 1.6  |
| \(-100+150\)          | 2.35 |
| \(-72+100\)           | 11.85|

| Size range, ASTM mesh | Wt % |
|-----------------------|------|
| \(-60+72\)            | 20.23|
| \(-52+60\)            | 52.35|
| \(-30+52\)            | 27.42|

Table 3. Chemical analysis of iron ore fines and lime, wt%. | Materials | Fe\(_{2}\)O\(_3\) | SiO\(_2\) | Al\(_2\)O\(_3\) | CaO | MgO | LOI |
|-----------------|----------------|----------------|-------------|-----|-----|-----|
| Iron ore fines  | 66.46          | 3.05           | 1.34        | -   | -   | 0.5 |
| Lime            | -              | 0.73           | 1.12        | 95.1| 0.8 | 2.0 |

Table 4. Composition of different kinds of pellets made.

| Kinds of pellets | Lime | Iron ore fines |
|-----------------|------|----------------|
|                 | \(-30+72\) size | \(-72\) size | Total |
| A               | 20   | 40             | 40    | 80   |
| B               | 25   | 37.5           | 37.5  | 75   |
| C               | 30   | 35             | 35    | 70   |
| D               | 30   | 70             | 0     | 70   |
| E               | 30   | 0              | 70    | 70   |
| F               | 40   | 30             | 30    | 60   |

Iron ore and lime fines were mixed together in the weight ratio of 80 : 20 to 60 : 40. Then the mixture was pelletized\(^{16}\) in a pelletizer to \(5–16\) mm size green pellets and produced pellets were left in natural air for 1–3 d for drying. Different pellets prepared are shown in Table 4. The partially dry pellet (1.5–2% moist) was treated with commercially pure CO\(_2\) gas. Pellet with \(25–30\)% lime and 15 min treatment exhibited maximum strength (~30 kg/pellet).

In order to assess the suitability of these pellets for use in BOF, the change in pellet properties were studied under slow and drastic heating conditions. The slow heating study helped in quantitative estimation of phases in the pellets as well as their softening points.

When the cold pellets are charged in to a BOF, they will undergo severe thermal shock due to sudden exposure to high temperature. This may result in crumbling of the pellets, leading to generation of fines and these fines would get carried away along with the furnace gas to cause extra load on the gas cleaning system. The drastic heating scheme was designed to investigate the property changes under thermal shock. However, prior to subjecting the pellets to thermal treatment, the phases present in the pellets (also after the thermal treatment) were identified by XRD. A TG-DTA facility was also utilized to determine the dissociation temperature of phases in the hardened pellets. The conditions and procedure adopted for the above tests are presented below.

2.1. XRD Study

An XRD facility, fitted with Siemens D500 X-ray diffractometer using Cu-K\(_{\alpha}\)/Co-K\(_{\alpha}\) radiation was utilized for phase identification of untreated, treated and heated pellets. Powder of \(-100\) mesh size was used for this purpose. The scanning speed was maintained at 2°/min. Peaks of Fe\(_2\)O\(_3\), CaCO\(_3\), Ca(OH)\(_2\), CaO, CaO–Fe-oxides, CaO–Silica–Fe-oxides etc. were identified by JCPDS files.

2.2. TG-DTA Study

Dissociation temperatures of the major phases, namely, Ca(OH)\(_2\) and CaCO\(_3\) were identified by TG-DTA study in a Netzsch make (model: STA-409) equipment using 0.081 g of sample at the heating rate of 10°C/min in air atmosphere and continued up to 1 450°C of sample temperature. Burnt kaolinite was used as the reference standard.

2.3. Slow Heating Study in Muffle Furnace

From TG-DTA study, the dissociation temperatures of the two major phases in pellet, namely, Ca(OH)\(_2\) and CaCO\(_3\) were found to be 480°C and 830°C respectively, which will be discussed later. These temperatures were used as a guide line for fixing the operating temperatures for conducting experiments under slow heating study. And from these experiments the quantitative estimation of two major phases in CO\(_2\) treated pellets were done.

Accordingly, the pre-weighed pellet samples of around 10 g were taken in a silica crucible. The samples were heated at 110°C, 500°C and 975°C in a muffle type resistance furnace in air atmosphere and were soaked for two hours at each temperature. The corresponding weight loss after soaking at each of the above temperatures was measured. The first weight loss (at 110°C) represents the mois-
ture content of pellet and the weight loss at the next two temperatures represents the loss due to dissociation of Ca(OH)$_2$ and CaCO$_3$ respectively. The procedure of calculation will be presented in the subsequent section.

2.4. Softening Point Determination

The softening point of pellets, determined by TG-DTA study was cross-checked by monitoring the shape change of the pellets during slow heating until melting. For this study, a pre weighed representative sample of the CO$_2$-treated pellets (4–6 g) was taken in quartz crucible (flat-shaped) and was slowly heated (5°C/min) in a high temperature muffle furnace in the ambient from room temperature up to 1 150°C, and held for 2 h for homogenization of the furnace temperature. Beyond this, for each 5°C rise in temperature, a soaking of 20 min duration was given and the physical state of the pellets was monitored. When the sample started loosing its shape, creating a little amount of molten mass, the temperature was noted and considered as the softening point of the pellet. Heating was continued for approximately 15°C above the softening point to ensure melting. The representative samples before softening, after softening and after melting were collected for phase analysis by XRD. The samples were also analyzed under optical microscope.

2.5. Thermal Shock Resistance Study

A pot type resistance furnace, was heated in the temperature range of 1 200–1 300°C in air atmosphere and was soaked for 30 min at the desired temperature for thermal stabilization. The test pellet sample encased in a small stainless steel wire basket was rapidly introduced in the hot zone of the furnace through an opening at the top of the furnace cover, with the help of a stainless steel wire. The sample, after retaining at the hot zone for 10–60 s was rapidly withdrawn from the furnace and air cooled. The heat treated sample was then subjected to the Cold Crushing Strength (CCS) and drop strength tests. CCS was measured in Honsfield’s Materials Testing Machine, Model: H 10K-S. The drop strength was measured by dropping an individual pellet on a mild steel plate from a height of 0.45 m. The number of drops required to break the pellet is a measure of its drop strength, the DSN. In keeping with the charging scope.

3. Results and Discussion

During pelletization, the lime fines (95.1% CaO), used in preparation of the green pellets, reacted with the externally added moisture to produce Ca(OH)$_2$. Figure 1(a), the XRD pattern of the pellet C, before CO$_2$ treatment indicates that almost whole of the CaO got converted to calcium hydroxide. This Ca(OH)$_2$, further transformed to CaCO$_3$ during subsequent treatment of the green pellets with CO$_2$. This is evident from the XRD pattern of the treated pellet presented in Fig. 1(b). Since, these pellets will be used at elevated temperature of BOF, the effect of heating on pellets up to high temperature is needed to be discussed which is as follows.

3.1. Slow Heating Study

3.1.1. Decomposition of Phases on Heating

When sample is heated, free moisture gets removed at temperatures above 100°C. On subsequent heating at elevated temperature, the samples undergo further loss in weight due to the release of chemically bonded moisture through Reactions (3) as follows.

\[
\text{Ca(OH)}_2 = \text{CaO} + \text{H}_2\text{O} \quad \text{(3)}
\]

Finally, CaCO$_3$ in pellet breaks to form CaO and CO$_2$ gas as per Reaction (4).

\[
\text{CaCO}_3 = \text{CaO} + \text{CO}_2 \quad \text{(4)}
\]

It has been reported that dissociation of Ca(OH)$_2$ in air starts at 270°C and reaches maximum at around 400°C. Dissociation is virtually complete by 500°C. CaCO$_3$ dissociate at 894°C temperature and 1 atm pressure. However, the actual dissociation temperature of these two phases in this particular study have been determined from TG-DTA study. Curves obtained from TG-DTA study for pellets A, C and F for 15 min treatments are shown in Figs. 2–4. It may be noted from these curves that mainly two weight loss peaks appear at around 460–500°C and 820–840°C. Since the moisture of the test sample was low (~0.5%), no significant weight loss peak could be observed at around 100–200°C temperature. The weight loss in the temperature range of 460–500°C indicates evaporation of moisture, produced due to breakage of Ca(OH)$_2$. The DTA lines, showing prominent endothermic peaks at around 480°C and 830°C for all three treated pellets in Figs. 2–4, represent the heat absorption due to the dissociation of Ca(OH)$_2$ and calcination reaction of CaCO$_3$, respectively.

It has been mentioned earlier that in order to quantitatively estimate the major phases, the pellets were heated and soaked in muffle furnace at 500°C and at 975°C for 2 h. The XRD pattern of pellet C heated at the above two temperatures are presented in Figs. 5(a) and 5(b) respectively. On comparing Fig. 5(a) with XRD pattern of the same sam-
prior to heating (Fig. 1(b)), it is evident that on heating whole of the unreacted Ca(OH)$_2$ got converted to CaO and the lime is primarily present in the form of CaCO$_3$ along with equivalent amount of CaO. Figure 5(b) does not show any CaCO$_3$ peak indicating that all lime is present either in the form of CaO or in combined form with other oxides like Fe$_2$O$_3$, SiO$_2$, etc., i.e. at 975°C, all CaCO$_3$ breaks to CaO as per Reaction (4). It has been observed that upon heating at 975°C, there is drastic deterioration in strength of the pellets. This is due to dissociation of CaCO$_3$ to CaO. The CaO thus produced presumably reacted with iron oxides at this temperature. The reaction between CaO and Fe$_2$O$_3$ is thermodynamically feasible even at room tempera-

ture and above as per Reaction (5)

\[
\text{CaO(s) + Fe}_2\text{O}_3(s) = \text{CaFe}_2\text{O}_4(s); \Delta G^\circ = -29700 - 4.8T J^20
\]

The formation of CaFe$_2$O$_4$ at 975°C was found (Fig. 5(b)), but the sample appears to contain a substantial amount of free CaO and Fe$_2$O$_3$. This is possibly due to the very slow kinetics at solid state. The silica bearing phase like (CaFe)SiO$_3$ also forms when heated at 975°C.

It may be noted that all the moisture, either free or combined form, evaporates by 500°C. The weight loss undergone by the sample at temperatures above 500°C till 975°C is due to evolution of CO$_2$ (Reaction (4)). Thus quantitative estimation of CaO in the form of CaCO$_3$ in pellet was done from the cumulative weight loss above 500 to 975°C and the balanced was considered to be in form of Ca(OH)$_2$. Appendix presents the calculation procedure adopted in quantitative estimation of phases and the calculated percentage of CaCO$_3$ and Ca(OH)$_2$ in different pellets are presented in Table 5.

Percentage of unreacted Ca(OH)$_2$ and CaCO$_3$ produced in pellet C are plotted against CO$_2$ treatment time in Fig. 6.
It has been found that 87% of transformation of Ca(OH)₂ was complete within 15 min of treatment and no significant change took place even with additional 7 min treatment. Therefore, it appears that after 15 min of CO₂ treatment, the reaction became sluggish. Percentage of CaCO₃ and unreacted Ca(OH)₂ in the three pellets, namely A, C and F, treated for 15 min in CO₂ were also calculated from the respective TG curves and are compared in Table 6 with the corresponding results obtained through weight loss study. In general a reasonably good agreement can be noted between the results obtained through two methods.

In this slow heating study, outer layer and inner core of treated pellets were manually separated and collected samples were subjected to weight loss studies, similar to what has been described in Sec. 2.3. It may be observed from the results listed in Table 7 that there is a substantial difference in Ca(OH)₂ contents at the outer layer and inner core of the pellet C for 5 min treatment. The difference is less significant for 15 min treatment. The CaCO₃ estimated by weight loss route was again cross checked by the carbon analyses of outer layer, inner core and bulk of pellets using TCD detectors (LECO, CS-444) and are presented in Table 8. The percentage of CaCO₃ obtained through both the methods appears to be in good agreement. Moreover, XRD analyses of the outer surface and inner core of the 5 min treated pellet(16) showed more residual hydroxide at core than outer surface. Therefore, these observations support the presumption that Reaction (2) proceeds from surface towards the centre of the pellets.

3.1.2. Softening Point Determination

The melting points of Fe₂O₃–CaO compounds are available from the binary phase diagram of Fe₂O₃–CaO system and is presented in Fig. 7.21 In the present study, both iron ore and lime fines contained some (~5%) gangue, and, hence, the system is not strictly a binary one. Therefore, towards realistic determination of softening/melting points of pellets, experimental investigation was done through (1) estimating the temperature for initiation of pellet’s shape change upon heating and (2) TG-DTA technique. In the shape change study, the temperature for initiation of the shape change of a pellet under the given heating schedule was noted. The state of pellet C (15 min CO₂ treated) at 1 210°C and 1 215°C are shown in Figs. 8(a) and 8(b), respectively. It is evident from the figures that pellet apparently retains its original shape till 1 210°C. Therefore, these observations support the presumption that Reaction (2) proceeds from surface towards the centre of the pellets.

![Fig. 7. Fe₂O₃–CaO binary phase diagram,21](image)
obtained from the shape change study have been reported in Table 9. It may be noted from the table that the softening points of the pellets are in the range of 1215–1280°C. It is further evident from the table that the softening temperature of the pellet decreases with increase in the lime content from 20 to 30%, which is not as per expectation. However, with further increase of lime beyond 30%, the softening point increases. This is in accordance with the binary phase diagram (Fig. 7). The softening points of CO2 treated and untreated pellet C were found to be the same (1215°C). This may be explained by the fact that on heating at elevated temperature, the moisture in untreated pellet, or the moisture and carbon dioxide in treated pellet get removed and the resulting composition of both the pellets attain the base composition containing CaO and Fe2O3 (plus gangue).

The softening temperatures determined by the shape change study were cross-checked by TG-DTA technique and the results have already been presented in Figs. 2–4. The softening points (major endothermic peaks) are marked with an arrow in the curves and the numerical values are indicated in the respective figures as well as listed in Table 9.

It is to be noted that both shape change and TG-DTA studies yielded the same softening temperature (1280°C) for pellets F. The softening temperatures of pellets with 20% and 30% lime (pellets A and C) were found to be the same (1180°C) in TG-DTA study and is 2–4% lower than the values obtained in the shape change study. Notably, the heating scheme, amount and shape of samples and crucible used were not identical in the two approaches. Therefore, this difference in the observed softening temperatures can possibly be attributed to the difference in experimental conditions. This difference may be considered as insignificant in respect to high temperature (1600–1700°C) of BOF, the intended place of its use.

In general, the observed softening points are in good agreement with the Fe2O3–CaO binary phase diagram (Fig. 7). It is worth mentioning that these FLIP possess a lower softening temperature than either pure Fe2O3 (dissociates into Fe3O4 that melts at 1597°C) or pure CaO (melting point, 2700°C), making them suitable for the early formation of low melting slag in the BOF.

### Table 9. Softening points of iron ore pellets containing varying lime.

| Pellets | Treatment time (min) | % lime in base mixture | Softening temperature, °C |
|---------|----------------------|------------------------|----------------------------|
|         |                      |                        | Shape change | TG-DTA (Major melting peak) |
| A-treate | 15 | 20 | 1235 | 1180 |
| C-treate | 15 | 30 | 1215 | 1180 |
| F-treate | 15 | 40 | 1280 | 1280 |
| C-untreate | 0  | 30 | 1215 | 1180 |

3.1.3. Phase Changes of Pellets Near Softening

The XRD analysis of powder sample of pellet C (Fig. 9(a)), soaked at the near softening temperature shows CaFeO3 as the major phase and with relatively minor amount of Fe2O3, Fe3O4 and silicates. The Figs. 9(b) and 9(c) present the phase analyses of the same sample at the start of softening and after complete melting respectively. After complete melting, the amount of Fe2O3 was found to be less than those before and at the start of melting. A considerable amount of Fe2O3 was found in all the three cases. This is presumably due to the transformation of Fe2O3 to Fe3O4 when it is heated in air atmosphere at high temperature.22)
The pellet samples at different heating stages were studied under optical microscope. Microstructures are shown in Figs. 10(a) and 10(b). It has been observed that pellets, before heating, contained higher amount of free iron oxides. On heating to near softening temperature, iron oxides reacted with lime and the amount of Fe$_2$O$_3$ in free form, drastically reduced.

3.2. Drastic Heating Study

The thermal degradation property of pellets at high temperatures is very important, as the pellets are intended to be used in BOF, operating at ~1 700°C. As the physical properties of CO$_2$ treated (15 min) pellets, B and C were comparable, the same pellets were subjected to the thermal shock resistance test as per procedure described in Sec. 2.5. The compressive (CCS) and drop strengths of these pellets, subsequent to the thermal shock for different retention times are listed in Table 10. The cold crushing strengths of these two types of pellets for different retention time at 1 300°C have also been graphically presented in Fig. 11.

No breaking of pellets could be observed on drastic heating which apparently indicates a good thermal shock resistance of pellet. However, it may be noted from Table 10 that both CCS and drop strengths of the pellets deteriorated after the thermal shock. From Fig. 11 it may be observed that for both the pellets, CCS decreases rapidly to 4 kg in the first 30 s, but marginally increases as the retention is continued up to 60 s. Similar observation could be made for drop strengths too. These characteristics can possibly be attributed to the fact that during the first 30 s of retention, CaCO$_3$ decomposes into CaO and CO$_2$, causing a decrease in strength of the pellets. With further retention, sintering of pellets results in slight increase in CCS and drop strength. On physical verification of the sample, retrieved after 60 s of thermal treatment at 1 300°C, it was observed that a fused layer was formed on the surface of pellets. No fusion was, however, observed for both the types of pellets when heated at 1 200°C even up to 60 s.

The pellet does not break or burst, however, strength deterioration of pellet happens at high temperature up to 4 kg, which takes a significant time. Once the pellets start joining the metal bath, the strength deterioration of the pellets is not likely to cause any fines generation and thus, no additional load is expected on the dust cleaning system when the pellets are used in the BOF. In addition, the indication of early fusion of pellets, particularly at high temperatures ($T>$1 300°C) should favor its usability in BOF in view of early formation of slag to begin early slag-metal refining reactions.

4. Conclusions

(1) Low softening/melting point of the CO$_2$ treated fluxed lime iron oxide pellets would assist easy formation of oxidizing slag and would promote refining when used in
BOF.

(2) The pellet exhibited reasonable thermal shock resistance at 1200–1300°C, ensuring low fines generation under BOF operating condition. A decrease in strength after high temperature exposure is not likely to restrict use of these pellets towards partial replacement of lump lime in BOF.

(3) Overall, this study demonstrates excellent possibility of the developed CO2 treated fluxed lime oxide pellets as an alternate feed material in BOF.

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Appendix

Calculation of CaCO3 Percentage

\[
\text{CaCO}_3 = \text{CaO} + \text{CO}_2 \ (g) \quad \text{(i)} 
\]

For Reaction (i) it is clear that 44 g weight loss is happened due to complete calcination of 100 g CaCO3.

Percentage weight loss at 500 to 975°C (only CaCO3) is 44 g/100 g CaCO3 × 100 = 44%. Therefore amount of CaCO3 in the treated pellets is 56/100 g = 56 g/100 g CaCO3.

Increase in weight for treatment of 100 g mixture after hydration is (34.5 − 28.5) g = 6 g.

Now during treatment Ca(OH)2 transforms to CaCO3.

Calculation of Ca(OH)2 Percentage

Initially during making of pellets it may be considered that whole amount of CaO transfer to Ca(OH)2.

\[
\text{CaO} + \text{H}_2\text{O} = \text{Ca(OH)}_2 \quad \text{..................................(ii)} 
\]

For 30% lime in base mixture of pellet, 100 g base mixture contains 30 g lime which is equivalent to (30/0.951) = 31.65 g CaO (as used lime contains 95.1% CaO).

28.5 g CaO converts to 74×28.5/56 = 37.66 g of hydroxide.

Increase in weight of 100 g mixture after hydration is (37.66−28.5) = 9.16 g.

Percentage of Ca(OH)2 in green pellets is 37.66/100×100 = 37.66/100 = 37.66%.

In Pellet C consider, y-grams of Ca(OH)2 has been transferred to CaCO3.

Therefore amount of CaCO3 = 100y/74 = 1.3514y g.

Increase in weight = (1.3514y−y) = 0.3514y g.

Percentage of CaCO3 in pellet C = 100×1.3514y/(100 + 0.3514y) = 35.89%. Or, y = 29.306 g.

Residual amount of Ca(OH)2 = (34.5−29.306) = 5.197 g.

Increase in weight for treatment = 0.3514×29.306 = 10.298 g.

Therefore %Ca(OH)2 = 5.197/(100 + 10.298) = 4.71%.

Total CaO in pellet (in form of hydroxide + Carbonate) is calculated by the respective multiplication factors.

\[
\text{Total } \text{CaO in pellets} = \%\text{Ca(OH)}_2 \times 56/74 + \%\text{CaCO}_3 \times 56/100 
\]

Other major compositions, %FeO, %SiO2 and %Al2O3 can also be calculated from the chemical analysis of the raw material in base mixture.