Flow of Molten Slag through a Coke Packed Bed

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The productivity and performance of the ironmaking blast furnace is significantly affected by the flow behaviour in the lower zone. The flow of reducing gas and liquids (iron and slag) through coke particles is often characterised as flow through a packed bed. To improve understanding of the flow in the lower zone of the blast furnace, an investigation has been carried out, where the primary aim was to obtain a physical description of the high temperature flow phenomena of liquid slag through a coke packed bed, based on characterisation of laboratory scale packed bed systems.

A synthetic slag in the CaO–SiO₂–MgO–Al₂O₃ system was fed at a controlled rate to pass through a coke packed bed heated to 1,500–1,600 °C. The mass of slag passing through the bed was logged. The bed was packed using synthetic coke to minimise the experimental uncertainty associated with the heterogeneity of industrial coke. Slag supply-drain curves, liquid holdup and residence time have been characterised. The effects of bed packing density, temperature and mineral content of the coke were tested.

Increasing the packing density or decreasing the temperature of the packed coke bed was found to increase the total liquid holdup and residence time of the slag. Increasing coke mineral content from 4.4% to 12% resulted in a decrease in the total holdup and the residence time. Mathematical models from the literature based on cold packed beds were used to predict the liquid holdup for the experiments, but were found to not adequately describe the results.

KEY WORDS: blast furnace; slag; liquid flow; liquid holdup; packed bed; coke.

1. Introduction

The iron blast furnace is the principal iron production unit in steelmaking. It is a counter-current reduction furnace that requires high gas permeability to achieve the high iron production rates that characterise modern blast furnace ironmaking. A key area of the furnace that affects permeability is the lower zone. This is the area below the cohesive zone bounded by the raceway and the hearth. The permeability of the lower zone is significantly affected by the liquid (iron and slag) flows and their holdup/retention in the furnace. An improved understanding of the liquid holdup and flow phenomena in the lower zone offers possibilities for enhanced productivity, efficiency and product quality.

The lower zone of a blast furnace is often approximated to flow through a coke packed bed. Consequently, studies of liquid flows in a packed bed and the channels developed between packed coke particles should offer insights into blast furnace performance and operation. In a coke packed bed, the voids (pores) formed between coke particles are generally interconnected through pore necks. A pore neck can be identified as a local minima of the hydraulic radius between particles. Pore interconnectivity and neck size have a significant bearing on the path of a liquid flowing through the bed (illustrated in Fig. 1) and possible sites of liquid blockage. Pore size, shape and interconnectivity depend on the particle size distribution, particle shape and packing density.

The flow of fluids through packed beds has been studied and described by formulae that treat the bed as a single unit. Examples of approaches to flow though a packed bed include Darcy’s law for liquid flow and Ergun’s formula for gas flow. These approaches are phenomenologically based and give a good description of the overall flow across a bed but do not consider the localised variations in packing structure, the flow phenomena at the pore level or the effect of chemical reactions between liquids and bed materials.
Early studies of the flow of liquids through packed beds based on room temperature experiments have attempted to relate the static and dynamic liquid holdup to bed voidage, and the viscosity and surface tension of the liquid, in addition to the interfacial properties between liquid and bed material. Static holdup \( (H_s) \) is defined as the amount of liquid that remains within the bed’s voids after stopping the flow and generally depends on bed and liquid properties. Dynamic holdup \( (H_d) \), also known as operating holdup, is defined as the liquid that flows out of the bed after stopping the liquid supply. The dimensionless numbers that describe the parameters of liquid flow through a packed bed include the modified Reynolds number \( (Re_m) \), the modified Capillary number \( (Cp_m) \), the modified Galileo number \( (Ga_m) \) and the Interfacial force number \( (Ne) \), as defined in Eqs. (1)–(4).

Modified Reynolds Number, \( Re_m = \frac{\rho_l u d_p \varphi}{(1 - \varepsilon) \mu} \) .......................................... (1)

Modified Galileo Number, \( Ga_m = \frac{\rho_l g (d_p \varphi)^3}{(1 - \varepsilon)^3 \mu^2} \) .......................................... (2)

Interfacial Force Number, \( Ne = 1 + \cos \theta \) ......................... (3)

Modified Capillary Number, \( Cp_m = \frac{\rho_l g (d_p \varphi)^3}{(1 - \varepsilon)^3 \sigma (1 + \cos \theta)} \) .......................................... (4)

where, \( \rho_l \): liquid density, kg/m\(^3\), \( u \): liquid superficial velocity, m/s, \( d_p \): particle diameter, m, \( \varphi \): particle sphericity, \( \varepsilon \): bed voidage, g: gravitational acceleration, m/s\(^2\), \( \mu \): liquid viscosity, Pa·s, \( \sigma \): liquid surface tension, N/m, and \( \theta \): liquid contact angle with bed material. The conduit diameter for the flow condition was taken as the particle diameter.

Using a dimensionless number approach, Fukutake \textit{et al.}\(^2\) and Sugiyama \textit{et al.}\(^10\) have developed empirical models to predict liquid holdup. The Fukutake \textit{et al.}\(^3\) predictions are given by Eqs. (5) and (6), and the Sugiyama \textit{et al.}\(^10\) predictions by Eqs. (7) and (8), for \( H_s \) and \( H_d \) respectively.

\[
H_s = \frac{1}{20.5 + 0.263 \cdot Cp_m} \quad \text{(5)}
\]

\[
H_d = 6.05Re_m^{0.648}Ga_m^{-0.485} \left( Cp_m (1 + \cos \theta) \right)^{0.097} N_e^{0.648} \quad \text{(6)}
\]

\[
H_s = 0.0194 \frac{Ga_m}{Cp_m^{0.0254}} \quad \text{(7)}
\]

\[
H_d = 8.122Re_m^{0.581}Cp_m^{0.298} \frac{Ga_m}{Ga_m^{0.42}} \quad \text{(8)}
\]

There have been few high temperature experimental studies that simulated the blast furnace lower zone.\(^11\)–\(^13\) In general, it was found that the liquid holdup calculated using the room-temperature based models did not agree with data from the high temperature experimental studies.\(^14\)

In a packed bed, bed voidage is the ratio of the pore (or void) volume to the bulk volume of the bed. Alternatively, bed packing density \( (\rho_b) \) can be used to describe a packed bed where \( \rho_b \) is defined as the volume fraction of the bed that is occupied by the packing material.\(^3\) The relation between both terms is given in Eq. (9).

\[
\varepsilon = 1 - \rho_b \quad \text{(9)}
\]

The objective of this study was to obtain a physical description of the high temperature flow phenomena of liquid slag through a packed bed of coke based on the characterisation of laboratory scale packed bed systems. This has been done by quantifying the effect of key variables such as temperature, packing density and coke ash content on liquid holdup and liquid residence time in packed beds of coke. Supply drain curves were generated to obtain the liquid holdup data and to give insight into liquid flow behaviour through the bed. Inspection and analysis of the slag-coke interface after flow was carried out by means of SEM and EDS analysis to understand any reactions that occurred.

In order to overcome issues relating to the heterogeneous nature of metallurgical coke obfuscating findings of this study a synthetic coke was used. The synthetic coke or coke analogue has a simplified carbon structure, controlled porosity, mineralogy and mineral phase dispersion. It has been shown in previous studies to mimic coke dissolution behaviour in liquid iron\(^15\)–\(^19\) and mineral reactivity effects in coke combustion.\(^17\)

2. Experimental

An experimental setup has been developed to measure the flow of molten slag through a coke packed bed. The setup was designed to investigate aspects of the flow under conditions that simulate the lower zone of a blast furnace. A schematic of the setup is given in Fig. 2.

In this experimental setup, a packed bed of coke analogue particles was heated to a temperature in the range of 1 500–1 600°C in a resistance furnace in a high purity (99.999%) argon atmosphere at a flow rate of 0.5 L/min. Based on an internal cross sectional area of the furnace tube of 0.00385 m\(^2\), this flow rate represents a gas velocity of 0.0021 m/s. The argon was passed through ascarite, drierite and then activated carbon to remove impurities.

Approximately 300 g of granulated slag was added to the top of the bed via a screw feeder at a feed rate of 3.3 g/min.
The slag melted and passed through the bed. The slag dripping from the bed was collected in a graphite collecting crucible placed on a micro-balance of 0.001 g accuracy. The primary output from each packed bed experiment was to obtain a supply-drain curve for the molten slag through the bed. The slag feeding rate was pre-set and the drained slag weight captured and logged with time via the micro-balance. A typical supply-drain curve of an experimental run is given in Fig. 3 for a packing density of 50% and bed temperature of 1500°C. From these two curves, \( H_s \), \( H_d \), \( H_t \), flow start time, \( t_s \), flow end time \( t_e \) and the average residence time, \( \text{ART} \), could be calculated as per Eqs. (10)–(13).

\[
H_s = \left( \frac{\text{total supply weight} - \text{total drain weight}}{\text{slag density} \times \text{total bed volume}} \right) \times 100
\]

\[............................. (10)\]

\[
H_d = \left( \frac{\text{mass of liquid drained after supply stop}}{\text{slag density} \times \text{total bed volume}} \right) \times 100
\]

\[............................. (11)\]

\[
H_t = H_s + H_d
\]

\[............................. (12)\]

\[
\text{ART} = \text{average of } (t_e - t_v)
\]

\[............................. (13)\]

The packed bed cell shown in Fig. 4 was designed to be equivalent to that found in a reference industrial coke.\(^{19}\) The mineral content of 12 mass% was selected to be equivalent to that found in a reference industrial coke if the silica component has been lost due to reaction, as was found in a previous study.\(^{19}\) Coke analogue particles used to pack the bed were obtained by crushing and sizing the prepared coke analogue into particles of between 8 and 10 mm. The particle density (\( \rho_p \)) of the coke analogue was measured in accordance with Australian Standard AS 1774.5-2001.\(^{20}\)

### 2.2. Bed Preparation

The packed bed cell shown in Fig. 4 was designed to be placed in the hot zone of the heating furnace. A high purity graphite crucible was used to accommodate the coke particles. Twelve drain holes were cut into the crucible bottom to allow the liquid slag to drip from the bed. The diameter of the drain holes was 7 mm. This value was established experimentally\(^{21}\) and chosen to ensure it did not impede slag flow. The height of the packed bed was 70 mm. This value represents the maximum bed height that gave a temperature consistency within the packed bed of ±11°C.

Packing density can effectively be controlled by changing the range of particle sizes and the bed compaction.\(^5\) In the

![Fig. 3. A typical supply-drain curve plotted on normalized axes. \( H_s \): static holdup, \( H_d \): dynamic holdup, \( H_t \): total holdup, \( v \): liquid volume at any time, \( t_v \): time of supply of liquid volume, \( t_s \): time of drain of liquid amount, \( t_e \): time of drain start, \( \text{ART} \): average residence time.](Image 45x107 to 286x253)

![Fig. 4. A schematic of the coke packed bed used.](Image 357x107 to 388x135)

<table>
<thead>
<tr>
<th>Table 1. Packing materials used and their mineral components.</th>
</tr>
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<tbody>
<tr>
<td>Coke analogue used to pack the beds</td>
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<tr>
<td>--------------------------------------</td>
</tr>
<tr>
<td>Coke analogue - CA6 (4.4%)</td>
</tr>
<tr>
<td>Coke analogue - CA6 (12%)</td>
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</tbody>
</table>

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current study the particle size was fixed between 8.0 – 10.0 mm to avoid blocking the slag flow, which meant that only bed compaction could be used to obtain the range of packing density tested. Loosely pouring the coke particles into the crucible and not disturbing them resulted in a $\rho_B$ of 50%. Higher values of $\rho_B$ were obtained by bed compaction. A laboratory vibrating table (Cleveland Vibrator Company - model VJ-1515) was utilised for this purpose. In general, approximately 100 g of packing material was poured into the graphite crucible and vibrated for different times at a fixed frequency of 60 Hz. Table 2 lists the vibrating time used for each packing density obtained. As the vibration resulted in an increase in the compaction of the bed, more particles were added when needed to keep the height of the bed fixed at approximately 70 mm. The bed packing density was established via Eq. (14).

$$\rho_B = \left( \frac{\text{packing material weight}}{\text{bed volume}} \right) \rho_p$$

### 3. Results and Discussion

Each high temperature flow experiment resulted in a supply-drain curve, showing the mass of slag supplied to and drained from the coke packed bed. A typical supply-drain curve was given in Fig. 3. The supply curve was a straight line whose slope is the feeding rate. In general, the drain curve started after a period of time allowing for the slag to melt and travel through the bed. Then it initially continued in large steps, alternating between periods of flow and no flow. These steps became smaller with time.

The base case experiment was repeated three times to assess experimental repeatability. A standard deviation of 0.9%, 1.1%, and 3.4% was achieved for $H_s$, $H_d$, and ART respectively.

The $H_s$ and $H_d$ obtained were generally consistent with those found in other high temperature experimental studies. However, they were generally higher than the room temperature experimental values. The $H_s$, $H_d$, and $H_t$ determined from the supply-drain curves and their comparison with the results of high temperature studies and room temperature studies from the literature are given in Table 3.

### 3.1. Effect of Temperature

The effect of temperature on the measured liquid holdup and ART for a fixed bed packing density and coke analogue ash content is given in Fig. 5. From Fig. 5, it can be observed that increasing bed temperature resulted in a decrease in the $H_s$, $H_d$, and $H_t$ and in the ART.

In order to better understand the change in the measured $H_s$ and $H_d$ with temperature, the effect of temperature on key slag properties in the empirical models for $H_s$ (Eqs. (5) and (7)) and $H_d$ (Eqs. (6) and (8)) was examined. The viscosity and surface tension of the slag were calculated using the Riboud model\(^{25}\) and NPL slag model\(^{26}\) respectively at each temperature and are given in Table 4. The viscosity and the surface tension of the slag were found to decrease with increasing temperature.

The decrease in the viscosity and surface tension and the consequent increase in slag fluidity with increasing temperature are consistent with the decrease in the liquid holdup and ART reported in Fig. 5. The empirical models for $H_s$ (Eqs. (5) and (7)) and $H_d$ (Eqs. (6) and (8)) predict that lower $H_s$ and $H_d$ will occur as slag surface tension and viscosity decrease.

### Table 2. Vibrating time used for each packing density.

<table>
<thead>
<tr>
<th>Bed packing density $\rho_B$, %</th>
<th>Vibration time, min</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>0</td>
</tr>
<tr>
<td>55</td>
<td>2</td>
</tr>
<tr>
<td>60</td>
<td>12</td>
</tr>
<tr>
<td>65</td>
<td>30</td>
</tr>
</tbody>
</table>

### Table 3. A comparison of measured $H_s$, $H_d$, and $H_t$ for all experimental runs with published high temperature data\(^{7,11}\) and room temperature data\(^{2,22,23}\).

<table>
<thead>
<tr>
<th>Holdup</th>
<th>Current study</th>
<th>High temperature studies</th>
<th>Room temperature studies</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H_s$, %</td>
<td>5.6</td>
<td>8.3</td>
<td>6.8</td>
</tr>
<tr>
<td>$H_d$, %</td>
<td>1.8</td>
<td>10.2</td>
<td>6.5</td>
</tr>
<tr>
<td>$H_t$, %</td>
<td>8.8</td>
<td>17.4</td>
<td>13.3</td>
</tr>
</tbody>
</table>

### Table 4. Slag viscosity (calculated by Riboud model\(^{25}\)) and surface tension (calculated by NPL slag model\(^{26}\)) as a function of temperature.

<table>
<thead>
<tr>
<th>Temperature, °C</th>
<th>1500</th>
<th>1550</th>
<th>1600</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity, Pa.s</td>
<td>0.264</td>
<td>0.200</td>
<td>0.154</td>
</tr>
<tr>
<td>Surface tension, N/m</td>
<td>0.494</td>
<td>0.486</td>
<td>0.479</td>
</tr>
</tbody>
</table>

Fig. 5. Effect of temperature on liquid holdup (Left) and on ART (Right). Packing density: 55%, Packing material: coke analogue-CA6 (4.4%). (data at 1 500°C (base case) are the average of 3 repeats).
3.2. Effect of Bed Packing Density

The effect of bed packing density on the measured liquid holdup and ART is given Fig. 6. From Fig. 6, it can be seen that the $H_t$ and the ART increased with increasing bed packing density. The effect on the $H_s$ and $H_d$ varied. An increase in packing density resulted in an increase in $H_d$. The measured $H_d$ depends on the time taken by the liquid to flow through the bed after the supply is stopped. This time relates to the ART which in turn increased with packing density. Given the scatter in the experimental data $H_t$ is either independent of packing density or is a weak function of it decreasing with increasing packing density.

The effect of packing density on $H_t$ and $H_d$ was considered using the empirical models for $H_t$ (Eqs. (5) and (7)) and $H_d$ (Eqs. (6) and (8)).

For $H_d$, both Fukutake’s formula (Eq. (6)) and Sugiyama’s formula (Eq. (8)) predicted that $H_d$ increases with an increase in packing density. This is consistent with the current observations. For $H_t$, however, Fukutake’s formula (Eq. (5)) predicted that $H_t$ would increase with an increase in packing density, while Sugiyama’s formula (Eq. (7)) predicted an opposite effect as it showed that $H_t$ would decrease with an increase in packing density. The difference in the mathematical model response to packing density is difficult to analyse with fundamental flow characteristics, as both models are empirically formulated.

The variation between the prediction of both models and the current experimental results could be discussed considering a primary difference between the two cases. The above models were based on cold experimental conditions where packed beds were pre-irrigated with the liquid and where all the possible flow channels were saturated prior to performing the measurements. This is not the case in the current experiments where some flow channels in the bed might be inaccessible due to the blockade of bed pores with small pore necks. Thus possibly influencing how packing density affects the number of contact (or near contact) sites where static holdup tends to accumulate.

3.3. Effect of Ash Content in Coke

In this study, two levels of the mass content of ash were tested: 4.4% and 12%. The effect of the ash content in the coke analogue on the measured liquid holdup and ART is given in Fig. 7. The data reported were an average of 3 runs for 4.4 mass% of ash and one run for 12 mass% of ash. It can generally be observed that an increase in the content of ash resulted in a decrease in $H_t$, that was mostly due to the decrease in $H_d$ ($H_s$ did not show significant change with the ash content) as well as a decrease in ART.

To investigate the effects of slag-coke interaction, attempts were made to assess changes in the interface chemistry by carrying out SEM-EDS analyses of the slag-coke interface. A typical microstructure of a slag-coke analogue-CA6 (4.4%) interface is given in Fig. 8. EDS elemental maps for aluminium, silicon, calcium and magnesium are also included in Fig. 8.

There has been slag penetration into open pores at the coke analogue surface and there would appear to be Si enrichment of the slag at and near the coke-slag interface. This silicon enrichment was found in all the coke-slag systems investigated. In addition, similar silicon enrichment was found and discussed in detail in a related study on slag
flow through coke channels.\textsuperscript{21)} Further examination of the interface using EDS spot analysis revealed not only a Si-rich phase near the slag-coke interface, but that directly coincident with the interface the SiO\textsubscript{2} equivalent concentrations could be >90 mass% and generally greater than 70%. It should be noted that the coke analogues reported in this paper had no SiO\textsubscript{2} in the ash analogue and therefore could not be a source of this enrichment.

In a previous study by the current authors using the same materials under similar experimental conditions\textsuperscript{21)} this silicon enrichment was analysed using a thermodynamic approach to determine whether it was caused by silicon rich phases precipitating from the slag during cooling or by a reaction between the slag and coke. It was found that while there were possible phases precipitating from the slag on cooling that could represent the near interface Si enrichment, no Si phases were predicted that had concentrations at the levels of those found at the interface. Thus, it is likely that the Si-rich phase at the interface is due to a reaction between the slag and coke. If slag is reacting with the coke, the most probable reaction is between SiO\textsubscript{2} in the slag and carbon in the coke to form SiC as given in Eq. (15).\textsuperscript{24)} It is known that SiO\textsubscript{2} in slag can form SiC in reaction with coke at the experimental temperatures.\textsuperscript{24)}

\[
\text{SiO}_2(\text{slag}) + 3\text{C}_n(\text{coke}) = \text{SiC}_n + 2\text{CO}_2(\text{g}) \quad \ldots \ldots \quad (15)
\]

While it can be expected that the formation of SiC will change the flow characteristics of the slag through the packed bed, the mass% of the ash fraction within the coke may also be having an effect. The greater mass% of ash in the coke increases the probability of contact between the slag and coke ash, as the coke ash being at least partially oxide based will more readily be accommodated (react) with the slag. This would have the effect of increasing the wettability of the slag-coke analogue system. This potential ash effect is consistent with the findings of Kang et al.\textsuperscript{27)} and Sun et al.\textsuperscript{28)} that showed increased reactivity of slag and coke at higher ash in coke levels. This ash effect on reactivity with slag may also be a factor in this study and could explain the lower H\textsubscript{d} and ART at the higher ash contents.

3.4. Comparison with Room Temperature Based Mathematical Models

The H\textsubscript{s} and H\textsubscript{d} measured under the experimental conditions were compared with those calculated from the room temperature based mathematical models (Eqs. (5)–(8)) (see Fig. 9).

The H\textsubscript{s} and H\textsubscript{d} values determined from this study were generally higher than the values predicted by the cold models. From Fig. 9, it can be seen that there were no obvious correlations between the experimental results and the model predictions. This might indicate that the holdup models based on room temperature experiments do not adequately describe the flow in the current hot experiments. This finding is consistent with Husslage’s study\textsuperscript{7,14)} when comparing the holdup results of their high temperature experiments to the predictions of these models. Specifically, Husslage et al.\textsuperscript{14)} stated that the experimentally measured static holdup for slag and metal at 1 400–1 600°C was much higher than that calculated using the Fukutake and Sugiyama models, which agrees with the results shown in Fig. 9. Husslage\textsuperscript{7)} attributed this discrepancy to the lack of bed irrigation dur-
ing hot experiments.

It may be that the inability of the cold models to describe the hot experiments is due to the fact that the models were derived under different conditions (pre-saturated uniform packing with all pore necks large enough to allow liquid flow) and do not account for the distributed nature of bed properties such as pore size, particle size and sphericity. In addition, the cold models do not account for any changes in flow as a result of slag-coke reactivity in the bed, as described in section 3.3.

While it would seem from the Fukutake\(^2\) and Sugiyama\(^10\) models that it is not possible to predict the liquid holdup of the packed bed experiments, they did offer some predictability of the effect of changes in single variables such as packing density and temperature on trends of liquid holdup and ART.

4. Conclusion

The flow of liquid slag through a laboratory scale coke packed bed was studied over a temperature range of 1 500°C to 1 600°C to characterise the liquid flow through, the liquid holdup of, and the liquid residence time in, the packed bed. The effect of bed packing density and coke ash content on packed bed flow was also assessed.

The \(H_s\), \(H_d\) and the ART of the slag flowing through a packed bed were found to be dependent on bed temperature, bed packing density and ash content in the packing coke. An increase in temperature decreased \(H_s\), \(H_d\), \(H_t\) and ART, which could be explained by the decrease in slag surface tension with increasing temperature. A similar argument can be made for a decrease in viscosity with temperature for \(H_s\), \(H_t\) and ART.

An increase in packing density increased \(H_s\), \(H_d\) and ART. An increase in ash content in the coke decreased \(H_s\), \(H_d\) and ART. Analysis of the slag-coke interface showed evidence of a reaction occurring between the slag and coke. The room-temperature-based empirical models available in the literature for liquid holdup were applied to the packed bed experimental conditions. While the models offered some predictability of the effect of changes in single variables such as packing density and temperature on trends of liquid holdup and ART, overall the experimental results did not match the predictions of the cold models.

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