Static Holdup of Liquid Slag in Carbonaceous Beds

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The static holdup of liquid slag in carbonaceous beds at high temperatures was studied using lab-scale experiments. The effects of particle size, type of carbonaceous materials, and slag composition on the static holdup of liquid slag were investigated. The particle diameter was found to be the most important factor determining the static holdup. From the experimental results, the empirical dimensionless correlation for the static holdup of liquid slag (H_s) in the carbonaceous beds was derived in terms of the modified capillary number (C_{pm}).

$$H_s = 32.9 \times C_{pm}^{-0.562}$$

KEY WORDS: capillary force; char; coke; carbonaceous bed; ironmaking; slag holdup.

1. Introduction

In the view of the global warming and depletion of natural resources, much effort has been directed toward environmentally-kindly iron-making process. For instance, in order to increase the energy efficiency of the blast furnace (BF), recently, the size of the BF has been increased. One of the largest BFs owned by POSCO at its Pohang steel works has a volume of 5 600 m³. On the other hand, new iron-making processes such as FINEX, COREX, FAST-MET, ITmk3, *etc.* have been developed to use lower grades of raw materials. High production efficiencies can be achieved in these processes when the counter flow of gas and molten slag and metal in a carbonaceous bed is controlled effectively. Excessive slag holdup in the carbonaceous bed may prevent continuous gas flow, yielding uneven temperature distribution in the carbonaceous bed. In addition, while the use of non-coking coal with and without cokes has been studied for many years, it is uncertain whether non-coking coals show different holdup properties in comparison to cokes.

Liquid holdup has been studied extensively for many years by many researchers.^{1–17)} Shulman *et al.*²⁾ suggested that the liquid holdup is composed of static and operating (dynamic) hold-up. Because of the difficulties encountered in high temperature experiments, many cold model studies have been widely carried out.^{1–12)} In the cold model studies, aqueous solutions and alumina or waxed spheres were generally used instead of liquid slags and carbonaceous materials, respectively. However, in the cold model studies, the

density of the liquid is smaller than the solid particles, whereas the density of the liquid slag in the BF operations is larger than the solid carbonaceous materials. In addition, the carbonaceous materials used in the BF operations generally show non-wetting behavior by liquid slag, whereas the solid particles used in the cold model studies show wetting behavior by aqueous solutions. Therefore, the cold model study is considered insufficient to predict the slag holdup behavior in the carbonaceous beds at high temperatures. Several researchers investigated the liquid slag holdup in a coke bed at high temperatures.^{13–17)} However, the experimental results obtained at high temperatures could not always be explained by the equations suggested by the cold model studies. Therefore, in order to understand the holdup of liquid slag in carbonaceous beds at high temperatures, high temperature experiments and relevant models are required.

In this study, lab-scale slag holdup experiments were carried out at high temperatures. Two kinds of carbonaceous beds (cokes and chars) were used. Based on the experimental results, a new slag holdup equation is suggested.

2. Theory

When the supply of liquid through a packed bed is the same as the drain, *i.e.*, when the liquid flow through a packed bed is in a steady state, the volume fraction of the liquid in the packed bed is called liquid holdup. Liquid holdup (H_t) is composed of static (H_s) and dynamic (H_d) holdups.³⁾ Static holdup is the volume fraction of the liquid in the packed bed, which remains in the bed when the supply of liquid is stopped. Dynamic holdup is the volume fraction of the liquid in the bed, which drains out from the bed when

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the supply of liquid is stopped.

$$H_t = H_s + H_d \quad (1)$$

Kawabata *et al.* reported that when there is no gas injection from the bottom, the static holdup, which is much larger than the dynamic holdup, is a dominant factor determining the total holdup.^{8,9)} Moreover, when the static and dynamic holdups are investigated simultaneously, the reliability of the obtained results becomes lower.¹⁴⁻¹⁶⁾ Therefore, in this study, only the static holdup is considered.

Static holdup is determined by the balance between the gravitational force and the solid-liquid wetting force. A dimensionless number is defined as the modified capillary number (C_{pm})³⁾ as per Eq. (2).

$$C_{pm} = \frac{\rho_L \cdot g \cdot \phi^2 \cdot D_p^2}{\sigma_L \cdot (1 + \cos \theta) \cdot (1 - \varepsilon)^2} \quad (2)$$

In the above equation, ρ_L , g , ϕ , D_p , σ_L , θ , and ε denote the density of liquid, gravitational acceleration, shape factor, particle size, surface tension of the liquid, contact angle between the liquid and a solid particle, and void fraction, respectively.

It is generally accepted that the static holdup is described as a function of C_{pm} . **Table 1** summarizes the reported equations. All the equations can be expressed by two types of equations, Eqs. (3) and (4), as follows:

$$H_s (\%) = \frac{100}{a + b \cdot C_{pm}^n} \quad (3)$$

$$H_s (\%) = c \cdot C_{pm}^n \quad (4)$$

In the above equations, a , b , c , and n are empirical constants, which are determined from the best fit of the experimental data. Both equations suggest that the static holdup decreases with increasing C_{pm} .

In **Fig. 1**, the static holdup equations are plotted with respect to C_{pm} . For comparison, data reported by Niwa *et al.*¹³⁾ from BF sampling and data from the high temperature experiments carried out by Husslage *et al.*¹⁵⁾ are plotted together. The BF sampling data by Niwa *et al.*¹³⁾ showed reasonable agreements with the predicted values, while the high temperature experimental measurements by Husslage *et al.*¹⁵⁾ were much higher than the predicted values. Hence,

the current model determined by cold model studies is unsuitable to predict the slag holdup behavior in carbonaceous beds.

3. Experimental

Figure 2 shows a schematic illustration of the experimental apparatus. A graphite crucible (I.D = 55 mm, height = 75 mm) consisting of 33 holes (each of diameter = 5 mm) on the bottom was used. In the graphite crucible, coke or char particles were charged to form a carbonaceous bed. The compositions of coke and char are shown in **Table 2**. The coke particles were distributed in four groups (4–5, 8–9, 14–15, and 20–24 mm). The number of coke particles placed in each layer was 103, 28, 9, and 4, respectively. In the experiments, 16, 8, 5, and 4 layers of coke were charged

Table 1. Equations describing the static holdup as a function of the modified capillary number.

Researcher	Static holdup (%)	Ref.
Fukutake and Rajakumar	$100/(20.5 + 0.263 \cdot C_{pm})$	3)
Chew <i>et al.</i>	$100/(21 + 0.305 \cdot C_{pm})$	7)
Kawabata <i>et al.</i>	$100/(26 + 0.27 \cdot C_{pm}^{0.9})$	9)

Fig. 1. Static holdup as a function of the modified capillary number.

Fig. 2. Experimental setup used for evaluating the static holdup.

in the crucible. The height of the packed bed ranged from 25 mm to 75 mm. Two groups of char particles (8–9 and 14–15 mm) were used. The number of chars per layer was the same as that of the number of cokes per layer. Then, fine slag particles were filled in the space among the carbonaceous beds. The slag composition is shown in **Table 3**. The graphite crucible containing the carbonaceous materials and slag particles was placed in a mullite reaction tube (I.D = 92 mm, height = 900 mm). Under the graphite crucible, a slag receiver of the same size as that of the graphite crucible was placed. The sample assembly was heated in an Ar atmosphere to 1723 K at a heating rate of +8.3 K/min. The temperature was held at 1723 K for 90 min and the sample was cooled down to room temperature at a cooling rate of –8.3 K/min. When the slag was molten, it dropped into the receiver through the carbonaceous bed. After the experiment, the amounts of slag remaining in the carbonaceous bed and that drained into the slag receiver were measured. The experiments were repeated 4–5 times with the carbonaceous particles of the same size. In addition, the top surface and the cross section of the carbonaceous beds were investigated after the experiments. The shape factor of the

carbonaceous particles was estimated using a high resolution micro-CT (Syscan1172, Bruker Corporation). A typical cross-sectional image of a scanned particle is shown in **Fig. 3**.

4. Results and Discussion

Figure 4 shows the photos of the top-surface and the cross-section of the coke beds after experimentation with slag A. In the bed with coke particles of 4–5 mm in diameter, most of slag was placed on the top of the coke bed. Slag was absent in the slag receiver. We assumed that the liquid slag formed on the top surface soaked the liquid slag within the coke bed owing to capillary forces. In the bed with coke particles of 8–9 mm diameter, slag droplets were observed between the coke particles. In the bed with coke particles of 14–15 and 20–24 mm particles, slag was found on the surface of coke particles. Several researchers have pointed out that channeling of liquid flow may occur in a relatively large scale packed bed.^{8,9,11)} However, in the lab-scale high temperature experiments, the experimental system was not large enough to identify the channeling effect. Therefore, it is more realistic to consider the static holdup without taking into account the channeling effect.

In **Fig. 5**, the effect of the particle size of the cokes on the static holdup is shown with respect to C_{pm} . Since the two slags under examination showed no considerable differences, all the data were plotted together. C_{pm} was calculated from Eq. (2). The density and surface tension were obtained from Ref. 18). The contact angle was separately investigated.¹⁹⁾ The void fraction was calculated using Eq. (5).

$$\varepsilon = 1 - \frac{(W_c / \rho_{app})}{V_t} \dots\dots\dots (5)$$

In the above equation, V_t is the total volume of the packed bed, W_c is the total weight of the carbonaceous particles in the crucible, and ρ_{app} is the apparent density of the carbonaceous particles. All the thermophysical data used in the present study are summarized in **Table 4**. The mean static holdups of slag A in the coke bed consisting of particles with diameters of 8–9, 14–15, and 20–24 mm were 8.55, 3.65, and 3.68%, respectively. However, the slag did not flow through the packed coke bed consisting of particles of diameter 4–5 mm (**Fig. 4(a)**). The measurements were reproducible with the same particle diameter. The maximum difference in the measured static holdup was 24%. These differences were caused by the packed structure and the low height of the coke bed.

Static holdup is generally accepted as a function of a dimensionless modified capillary number as shown in Eq. (6) or (7).

$$H_s (\%) = \frac{100}{7.195 + 0.415 \cdot C_{pm}} \dots\dots\dots (6)$$

$$H_s (\%) = 32.9 \cdot C_{pm}^{-0.562} \dots\dots\dots (7)$$

The values predicted with Eqs. (6) and (7) reasonably agreed with the data reported by Niwa *et al.*¹³⁾ and Husslage *et al.*¹⁴⁾ However, Eq. (6) (dashed line in **Fig. 5**) was found to be unsuitable for the prediction of the slag holdup,

Table 2. Composition of the cokes and chars used in the present experiments (wt%).

Component	Moisture	Volatile Matter	Ash	Fixed Carbon
Coke	0.22	1.76	11.66	86.36
Char	2.71	4.71	17.11	75.47

Table 3. Chemical composition of the slag (wt%).

	Al ₂ O ₃	CaO	MgO	SiO ₂	FeO	Ref.
Slag A	18	40	10	32		Present Work
Slag B	16	44.5	4	35.5		
Slag C	14.4	32	8	25.6	20	13)
Slag D	10	38	12	40		15)
Slag E	12	43	13	32		15)

Fig. 3. Typical scanned cross-sectional CT image of a coke particle.

Fig. 4. Coke beds before and after the static holdup experiments.

Fig. 5. Static holdup as a function of the modified capillary number. (Online version in color.)

because it cannot explain the present experimental results with coke particles of 4–5 mm in diameter. However, Eq. (7) is more suitable for the prediction of the slag holdup (solid line in Fig. 5). The mean static holdups of slag B in the coke bed consisting of particles with diameters of 14–15 and 20–24 mm were 3.33 and 2.93%, respectively. The data obtained with slag B agreed well with the values predicted with Eq. (7).

Table 4. Thermophysical properties used for the calculation of the modified capillary number.

	Slag A	Slag B
Density (kg/m^3) ¹⁸⁾	2 773	2 766
Surface tension (N/m) ¹⁸⁾	0.485	0.480
Contact angle with coke ($^\circ$) ¹⁹⁾	118	122
Contact angle with char ($^\circ$) ¹⁹⁾	122	—
Void fraction (—)	0.46–0.58	0.47–0.49
	Coke	Char
Apparent density (kg/m^3)	921	950
Shape factor (—)	0.59 (± 0.048)	0.52 (± 0.098)

It is noteworthy that the values obtained with Eq. (7) from the high temperature experiments agreed well with the values predicted using the cold model experiments when C_{pm} was larger than 20. However, the hot model data became much larger than the cold model predictions when C_{pm} was smaller than 20. In the cold model studies, as addressed before, the density of the liquid is smaller than that of the solid particles; however, the density of liquid slag is larger than that of the solid carbonaceous materials. In addition, the solid particles used in the cold model studies are wetted by aqueous solutions, while the carbonaceous

materials used in the high temperature experiments are not wetted by liquid slag. Accordingly, when the slag between the carbonaceous particles is molten, it may push the nearby particles and provide larger room. This tendency becomes stronger as the capillary forces increase by decreasing the gap between the particles. Consequently, as the particle size decreases, the static holdup increases more rapidly.

In Fig. 5, the static holdup of slag A in the char bed is shown together. The mean static holdups for particles with diameters of 8–9 and 14–15 mm were 8.89 and 5.17%, respectively. The maximum difference in the measured static holdup was 34%. Due to irregularity in the shape of the char particles, the experimental scatter was slightly larger than that observed for coke beds. All the thermophysical data used in the present study is summarized in Table 4. Differences in the thermophysical properties were found in the contact angle and the shape factor. The contact angle between char and liquid slag A was 122° and the shape factor of the char particles was 0.52. From Fig. 5, Eq. (7) was found to predict the experimental data with char beds as well. Hence, Eq. (7) seems suitable to predict the static holdup of liquid slag in coke and char beds examined in the present study.

5. Conclusions

The static holdup of liquid slag in carbonaceous beds at high temperatures was studied using lab-scale experiments. The effects of particle size, type of carbonaceous materials, and slag composition on the static holdup were investigated. The particle diameter was found to be the most important factor determining the static holdup. From the experimental results, an empirical dimensionless correlation for the static holdup of liquid slag in the carbonaceous beds was derived in

terms of the modified capillary number as $H_s(\%) = 32.9 \cdot C_{pm}^{-0.562}$.

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