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Characterisation of coke packed beds after liquid slag flow at 1 500°C by image analysis

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Abstract

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Keywords

image, c, 500, 1, analysis, flow, characterisation, slag, liquid, after, beds, packed, coke

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Characterisation of Coke Packed Beds after Liquid Slag Flow at 1500°C by Image Analysis

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Abstract

The flow of slag in the lower zone of the ironmaking blast furnace was experimentally simulated by adding slag to a high temperature laboratory scale coke packed bed. The flow of slag through packed beds of coke with packing densities varying from 50% to 65% was examined at 1500°C. Since the liquid flow through a packed bed depends on packing properties such as particle size, particle shape, pore size and pore neck size, it was necessary to characterise these properties of the beds.

In this work, image analysis of successive sections of the tested beds was utilised to characterise the bed packing properties. In addition, the slag distribution and holdup was also measured. The procedure was that at the end of each experiment, the beds were cooled down, cold mounted in resin, then sectioned into approximately 4 mm thick slices. Each section was then analysed to measure particle and pore geometric properties, and slag distribution. It was found that the slag holdup was well distributed within the bed and was varied in position between bed cross sections, indicating no significant wall effect. The average pore size and average pore neck size were found to decrease as the packing density increased. Slag flow caused the particle sphericity and the average pore neck size to increase in comparison to an unreacted bed due to the slag-coke interaction.

Keywords: blast furnace; slag; coke; liquid flow; liquid holdup; image analysis; packed bed.

1. Introduction

In the lower zone of an ironmaking blast furnace, the flow of liquid slag and iron and their holdup affect furnace permeability, performance and productivity. Due to the limited accessibility of this zone under the operational conditions, experimental and mathematical simulation approaches have been adopted to study liquid flow in this zone. For this, the lower zone is often approximated to flow through a coke packed bed [1, 2]. Consequently, studies of liquid flows in a packed bed and the channels developed between packed coke particles should give insight into blast furnace performance and operation.

Early studies of the flow of liquids through packed beds were based on room temperature experiments and have attempted to relate the liquid holdup to bed packing properties and liquid properties (density, viscosity and surface tension) in addition to the interfacial properties between liquid and bed material via empirical mathematical formulae [2-4].

Bed, liquid and flow parameters have been utilized in the form of dimensionless numbers (modified Reynolds Number Re_m, modified Galileo number Ga_m, Interfacial Force Number Ne and modified Capillary Number Cp_m). These dimensionless numbers have been utilized in the mathematical formulae to predict the liquid holdup and are defined in Equations 1-4.

Modified Reynolds Number, $\mathrm{Re_m} = \frac{\rho_l.u.d_P.\varphi}{(1-\varepsilon).\mu_l}$ (1)

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

Interfacial Force Number, $\mathrm{Ne} = 1 + \cos\theta$ (3)

Modified Capillary Number, $\mathrm{Cp_m} = \frac{\rho_l g(d_P\varphi)^2}{(1-\varepsilon)^2\sigma(1+\cos\theta)}$ (4)

where ρ_l: liquid density, kg/m^3, u: liquid superficial velocity, m/s, d_p: particle diameter, m, φ: particle sphericity, ε: bed voidage, g: gravitational acceleration, m/s^2, μ: liquid viscosity, Pa·s, σ: liquid surface tension, N/m, and θ: liquid contact angle with bed material, rad. The conduit diameter for the flow condition was taken as the particle diameter.

In a packed bed, bed voidage, ε, is the ratio of the pore (or void) volume to the bulk volume of the bed [5]. Alternatively, bed packing density (ρ_B) is a measure of how closely packed a bed is and is defined as the volume fraction of the bed that is occupied by the packing material [6]. The relation between both terms is defined by Equation 5.

$$\varepsilon = 1 - \rho_B \quad (5)$$

In considering flow through a packed bed, liquid holdup is divided into two main types, static and dynamic. Static holdup (H_s) is the amount of liquid that does not freely drain from the bed, but instead remains within the bed's voids after stopping the flow. Dynamic holdup (H_d) is the liquid that flows out of the bed after stopping the flow.

Mathematical description of liquid holdup has been based on room temperature-based packed bed experiments [2-4]. Examples of these models are given in Equations 6 and 7 by Fukutake [2] for H_s and H_d respectively.

$$H_s = \frac{1}{20.5 + 0.263\ .Cp_m} \quad (6)$$

$$H_d = 6.05 Re_m^{0.648} Ga_m^{-0.485} (Cp_m(1 + \cos\theta))^{0.097} N_e^{0.648} \quad (7)$$

The above empirical models treat the bed as a single unit. However, the macroscopic flow of the liquid through packed bed particles is governed by the localized liquid flow patterns at the

level of the individual pores that form between bed particles. These pores are generally inter-connected and are accessible through their relatively narrow pore necks where the flow is mostly governed by interfacial phenomena [7, 8]. A recent study by the current authors [9] focussed on quantifying the minimum pore neck size for flow of liquid slag through a coke channel revealed that while there is a minimum pore neck size for slag to pass through, this size was time dependent. The minimum diameter at 30 minutes and 1500°C for a number of different slag and coke types was approximately 4.4 mm. At longer times the slag was able to flow through smaller channels. There was evidence of reaction at the slag-coke interface that resulted in what was thought to be SiC formation. The time dependence of this minimum pore neck size was explained in terms of changes (lowering) of the contact angle, also measured in the study, between the slag and coke with time as a consequence of the interfacial reaction. This resulted in the slag-coke system changing from a non-wetting to a wetting system and favouring pore infiltration.

From Equations 1-7, packing density and particle sphericity needed to be measured for a bed so that H_s and H_d could be calculated. Also, the pore neck size distribution needed to be characterised to investigate the accessible pores and estimate possible flow channels in a packed bed.

The characteristics of liquid flow through packed beds depend on liquid properties, liquid-bed interaction and bed packing properties. Due to this it is necessary to characterise the actual bed packing properties and pore structure for each flow condition. Unfortunately the bed characteristic data such as voidage, pore size and pore neck size distribution are not known *a priori*, but can only be determined after the bed is constructed and analysed [10].

In a set of high temperature experiments to investigate the slag flow through a laboratory scale coke packed bed, the effect of bed packing density on packed bed flow was assessed. It was found that an increase in packing density resulted in an increase in H_d and a neutral/slight decrease in H_s. As a result the total holdup increased as bed packing density increased [11].

The objective of this work was to quantify the amount and the distribution of the liquid slag holdup through a coke packed bed and to characterise bed packing properties for a number of bed packing densities. This has been done by physical inspection and image analysis of successive cross sections of packed beds post experiment. The image analysis was carried out to provide knowledge about the coke bed structure and to characterize packing properties of the beds that affect the flow of liquid slag, namely packing density, bed pore size, bed pore neck size and coke particle sphericity.

2. Experimental

High temperature laboratory experiments have been carried out to investigate the flow of slag through packed beds of coke where packing density was varied from 50% to 65% at 1500°C under a high purity (99.99%) argon atmosphere. The argon was passed through ascarite, drierite and then copper turnings at 300°C prior to entering the furnace.

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. Details of the packing

method are found elsewhere [11]. The nominal bed packing density, $\rho_{B,}$ was initially established by Equation 8.

$$\rho_B = \frac{(Packing\ material\ weight / Total\ bed\ volume)}{\rho_p} \quad (8)$$

where ρ_p is the particle density of the packing material.

The slag composition was 40.7% CaO, 37.4% SiO_2, 12.5% Al_2O_3 and 8.8% MgO. The bed was packed in a 55 mm inner diameter graphite crucible using particles of synthetic coke (coke analogue with 4.4 mass% of CA6 (CaO.6Al2O3)). The particle density was 1.08 g/cm^3 as measured in accordance with Australian Standard AS 1774.5-2001 [12]. The nominal particle size was 8-10 mm and bed height was 70 mm. Full details of the packed bed experimental set-up are given elsewhere [11].

The slag feeding time was approximately 90 minutes. The bed was held at the experimental temperature for 60 minutes after slag feeding had stopped then cooled to room temperature at a rate of 5°C/min. Once cooled, the bed was removed for inspection.

After each experiment the static holdup $H_{s,}$ was initially determined by estimating the slag mass trapped in the bed after flow via Equation 9.

$$H_s = \frac{(Mass\ of\ slag\ trapped\ in\ the\ bed / Slag\ solid\ density)}{Total\ bed\ volume} \quad (9)$$

The beds were then mounted in cold resin containing florescent dye which served to distinguish resin from slag. . Mounting was done under vacuum to aid impregnation of bed pores with resin. After the resin had hardened, the bed was sectioned with a diamond saw (of 1mm thickness) into on average 13 slices of approximately 4 mm thickness, yielding 22 faces for analysis. Care was taken to ensure that both the vertical and rotational position of the

slices in the original bed was maintained. The sections' faces were then scanned using a flatbed scanner (Canon iR3025 UFR II) and the scanned images were stored as colour 300x300 dpi JPEG files. A typical set of post experiment cross section faces of the packed bed is given in Fig.1. The sections were sorted from top to bottom according to their position in the bed keeping the relative orientation of the sections fixed.

The images were then processed to achieve contrast separation for each phase so that threshold based analysis via the particle analysis tool in the program ImageJ 1.38 [13)] could be applied.

In determining the pore and pore neck sizes, the definition of a pore in a packed bed as presented by Dullien [5)] and schematically illustrated in Fig. 2 was adopted. From this, a pore was defined as the space between coke particles when it was surrounded by coke particles and when lines could be drawn at the local minima of the hydraulic radius between particles.

This was done manually for each section image by drawing lines whenever a local minimum between coke particles was identified. These lines represented the pore necks and the analysis of the length of these lines was taken as a measure of the pore neck size for the sections analysed.

In addition to the irrigated beds, a non-reacted bed that had experienced the same thermal history of the irrigated packed bed experiment but without the addition of the slag was evaluated to provide a comparison with the reacted beds. Image analysis was also used to characterize the local area-based packing density, ρ'_B, and slag static holdup, H'_s, with the results compared to values measured from slag mass [11)]. ρ'_B and H'_s were estimated as the

area fraction of the coke particles and the trapped slag respectively to the total area of the bed sections as defined by Equations 11 and 12 respectively. Fig.3 illustrates a typical example of an image of a slice of coke bed after an experiment and the separated layers of coke particles and slag.

$$\rho'_B = \frac{\sum_{i=1}^{n} Coke\ particle\ area\ in\ section\ i}{\sum_{i=1}^{n} Cross\ sectional\ area\ of\ bed\ section\ i} \qquad (11)$$

$$H'_s = \frac{\sum_{i=1}^{n} Slag\ phase\ area\ in\ section\ i}{\sum_{i=1}^{n} Cross\ sectional\ area\ of\ bed\ section\ i} \qquad (12)$$

where *i* is the bed slice number and *n* is the total number of the bed slices.

On a two dimensional projection (as in bed sections), the bed pore size was estimated as the equivalent diameter of a circle with the same area as the 2-D projection of the pore projected area. The coke particle sphericity was estimated as the average circularity defined by the ratio of the perimeter squared to the projected area times 4π as given in Equation 13 [14].

$$\text{Sphericity} = {}^{4\pi A}/_{P^2} \qquad (13)$$

where *A* is the particle projected area and *P* is its perimeter.

3. Results and discussion

The analysis of all cross sections of the beds revealed that the pore size, pore neck size and particle sphericity were all of a distributed nature. A typical example of the distribution of the equivalent pore diameter for the sectioned packed beds is given in Fig.4 showing the distributed nature of the measured variable. Similar distributions were obtained for pore neck size. Four different bed packing densities (ρ_B = 50, 55, 60, 65%) have been tested in the packed bed experiments. The cumulative distributions of pore equivalent diameter and pore

neck size are given in Fig.5 and Fig.6 respectively. It can be seen that there is a general trend for the equivalent pore diameter and pore neck size to both decrease with increasing ρ_B.

3.1 Effect of packing density on bed pore size and pore neck size

For the fixed and relatively narrow particle size range used to pack all beds (8-10 mm), packing parameters, specifically pore size and pore neck size, were generally dependent on the prepared bed packing density ρ_B as it was shown in Fig.5 and fig.6. To show the effect of ρ_B on the equivalent pore diameter and the pore neck size, the pore equivalent diameter and pore neck size as measured by image analysis of the sliced bed sections for each of the four ρ_B tested are given in Fig.7 and Fig.8 respectively. In both cases, both the average value and the maximum observed value are plotted to provide an indication of the data range. As expected, it can be seen that an increase in ρ_B resulted in a decrease in the pore equivalent diameter and the pore neck size. It can be observed that the increase in porosity as the packing structure becomes less dense is dominated by the larger pores as the maximum values of pore size seemed to increase the most. The minimum pore size and pore neck size were less than 0.07 mm in general and are not shown.

3.2 Characterization of slag static holdup by image analysis

Image analysis was used to estimate the H'_s after experiments by Equation 12. The results were compared to H_s values obtained from estimating the trapped slag mass after the experiments using Equation 9 and that calculated by Fukutake's model (Equation 6). As given in Fig.9, the comparison indicates that there was generally good agreement between the H_s and H'_s. However, both values were appreciably higher than that obtained from the room temperature experiment-based models such as described by Equation 6. It would appear that

the room temperature-based models do not adequately describe the liquid retention mechanisms in the current hot experiments or of other published high temperature works [11, 15].

3.3 Characterization of the effect of slag flow on bed packing properties

The ρ'_B, particle sphericity and pore neck size measured by image analysis for a bed of ρ_B=55% for an unreacted bed and one after liquid slag flow are given in Table 1, Fig.10 and Fig.11 respectively. Beds for ρ_B=50,60 and 65 were analysed in the same way.

It can be seen that there was little change in the packing density before and after the slag had flowed through the bed (Table 1). This indicates good consistency in the packing density during the slag flow as an experimental control parameter. It can also be observed that the packing density before slag flow when measured by image analysis was less than when measured by weight and volume method. The difference between the packing densities measured by the two methods is likely related to how the sections were made and the number of sections obtained for each packed bed [5,6].

There is material loss in the sectioning of the bed and there is a practical limit to the number of sections that can be obtained for a given bed. Also, the top and bottom sections of the bed are likely to be less densely packed as a result of an end effect. This is evidenced in Fig.1 by the greater proportion of the resin to coke particles in sections 01 and 02 for the top end and 21 and 22 for the bottom end. It is likely that if we were to increase the number of sections per packed bed and to exclude the end slices, the packing densities by both methods would converge.

From Fig.10 and Fig.11, it can be seen that there was an increase in both the sphericity of the bed particles and the pore neck size after the slag flow through the bed. The average particle sphericity increased from 0.57 to 0.61 and the average pore neck size increased from 1.71mm to 2.11mm as a result of the slag flow through the packed bed experiments. The combined effect of slag flow on particle sphericity and pore neck size is schematically illustrated in Fig.12

In previous studies for experiments carried out under similar conditions[9,11] as this investigation it was observed that there was silicon enrichment at the coke slag interface. This was thought to be a result of reaction of SiO_2 in the slag with carbon in the coke to form SiC. Other workers have shown this is possible[16]. This reaction may cause rounding and (local) loss of carbon from the coke. There is also the possibility of further local carbon loss due to spallation of the SiC layer due to structural mismatch with the coke or further reaction of the SiC to form SiO(g). These possibilities were not explored in the current study. The reaction of the coke with the slag is thought to cause the increase in size of the inter-particle pore necks.

3.4 Characterization of the slag flow pattern using image analysis

In a packed bed the liquid slag flows into a pore through its neck which interconnects it with other pores. Consequently, flow between pores depends on the pore neck characteristics, especially size. For a non-wetting liquid, the accessibility of a bed pore is determined by whether its neck is large enough to allow slag infiltration. For a wetting liquid, the liquid will enter a pore but will not exit it and flow to another unless its pore neck is wide enough to allow the slag to drain. A summary of pore size, pore neck size and particle shape data measured by image analysis for each packing density is given in Table 2. Given the previous

results of the authors [9] that showed the critical pore neck size for slag flow decreased with time, it may be possible to infer the effects of time on flow by considering the percentage fraction of pore necks greater than a range of critical values (see Table 2 for values ≥ 4.4, 3.0 and 2.0 mm.). The values chosen were based on the previous work by the authors [9].

The time dependency of the slag flow through pore necks could affect the flow of the slag through the packed bed experiments. At the early stages of the flow (the first 30 minutes), the liquid slag could only have passed through a limited fraction of available bed pores with necks ≥ 4.4mm. This fraction varied from about 9.1% at a packing density of 50% to 0.9% at a packing density of 65%. At longer times, the smaller pore necks (3.0 and 2.0 mm) would have also been accessible for slag flow, increasing the number of possible flow channels in the bed for the slag.

The data of pore neck size in Table 2 were plotted against bed ρ_B and given in Fig.13. The plot shows that the percentage of pore necks above the critical size decreases with increasing ρ_B, hence increasing the time needed for the slag to pass through these beds. This analysis indicates that residence time of the slag in the bed should increase with ρ_B. This is consistent with recent work by the authors [11].

4. Conclusion

The ability to characterise progressive sections throughout the coke packed beds after liquid slag has passed through at high temperature by image analysis provided significant insight into the slag flow patterns and behaviour through the bed.

The static holdup was measured by image analysis (H'_s) and by measuring the mass of trapped slag after the experiment (H_s). It was found that there was generally good agreement between the two techniques for measuring static holdup across the range of packing densities tested.

The measured pore size, pore neck size and particle sphericity were found to have a distributed nature. The flow of slag through coke analogue packed bed caused the average sphericity of coke particles to increase from 0.57 to 0.61 and the average pore neck size to increase from 1.71mm to 2.11mm. This has been explained by considering reaction at the slag-coke interface observed in previous research.

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List of Captions:

Fig.1 An example of a full set of scanned images of post experiment cross sections of a packed bed. Bed packing density: 55%. Slag, coke analogue and resin appear as white, dark grey and light grey areas respectively. Images are sorted from the top section of the packed bed (01) to the bottom of the packed bed (22). Experimental duration = 90 minutes.

Fig. 2 Two-dimensional presentation of three-dimensional pores and pore necks [5)].

Fig.3 An example of images of a cross section of a coke bed after an experiment - position: 1cm above bed bottom. 1- The image before processing, 2- A layer of manually defined pore necks (black lines), 3- A layer of the identified bed pores (in white) and coke particles (in black), 4- A layer of the slag phase (in white).

Fig. 4 A typical example of the distribution of pore equivalent diameter within the bed - ρ_B: 55%. Experimental duration = 90 minutes.

Fig. 5 Cumulative distribution of pore equivalent diameter within the bed. Experimental duration = 90 minutes.

Fig. 6 Cumulative distribution of pore neck size within the bed. Experimental duration = 90 minutes

Fig. 7 Estimated pore equivalent diameter versus bed packing density. Experimental duration

= 90 minutes.

Fig. 8 Estimated pore neck size versus bed packing density. Experimental duration = 90 minutes.

Fig.9 A Comparison of the static holdup estimated by bed mass method, estimated by image analysis method and calculated by Fukutake model for different packing densities. Experimental duration = 90 minutes.

Fig. 10 Estimated coke analogue particle sphericity for a bed before slag flow and after slag flow - ρ_B=55%. Experimental duration = 90 minutes.

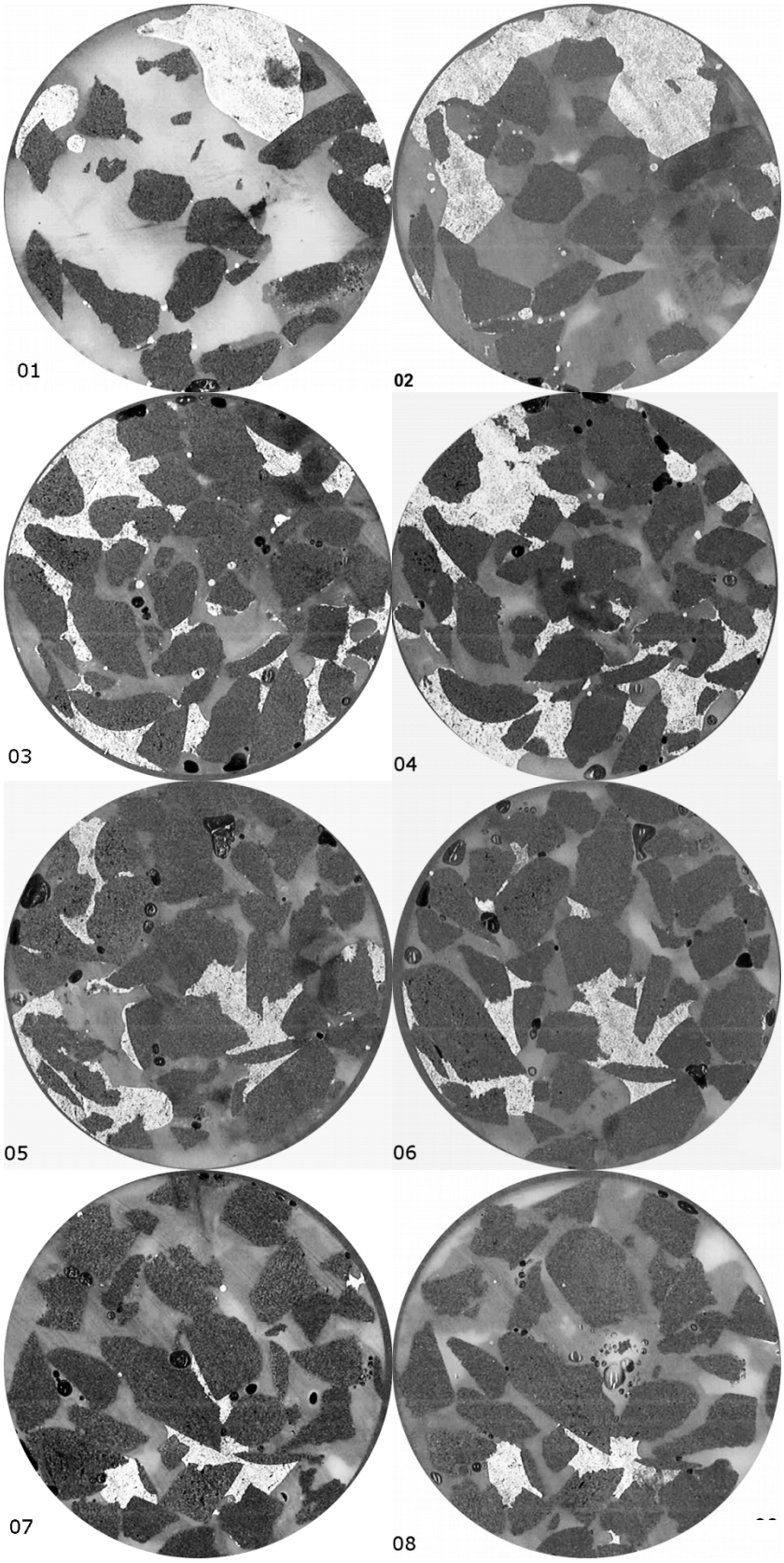
Fig. 11 Estimated bed pore neck size distribution for a bed before slag flow and for a bed after slag flow - ρ_B=55%. Experimental duration = 90 minutes.

Fig. 12 A schematic illustration of the change in coke particle roundness and pore neck size due to slag flow.

Fig. 13 The variation of the estimated pore neck size percentage by image analysis plotted against bed packing density.

Table 1 Comparison of the bed packing density measured by image analysis method before and after experiment. Nominal ρ_B=55%.

Table 2 Summary of the average bed packing properties obtained from image analysis of the cooled down beds.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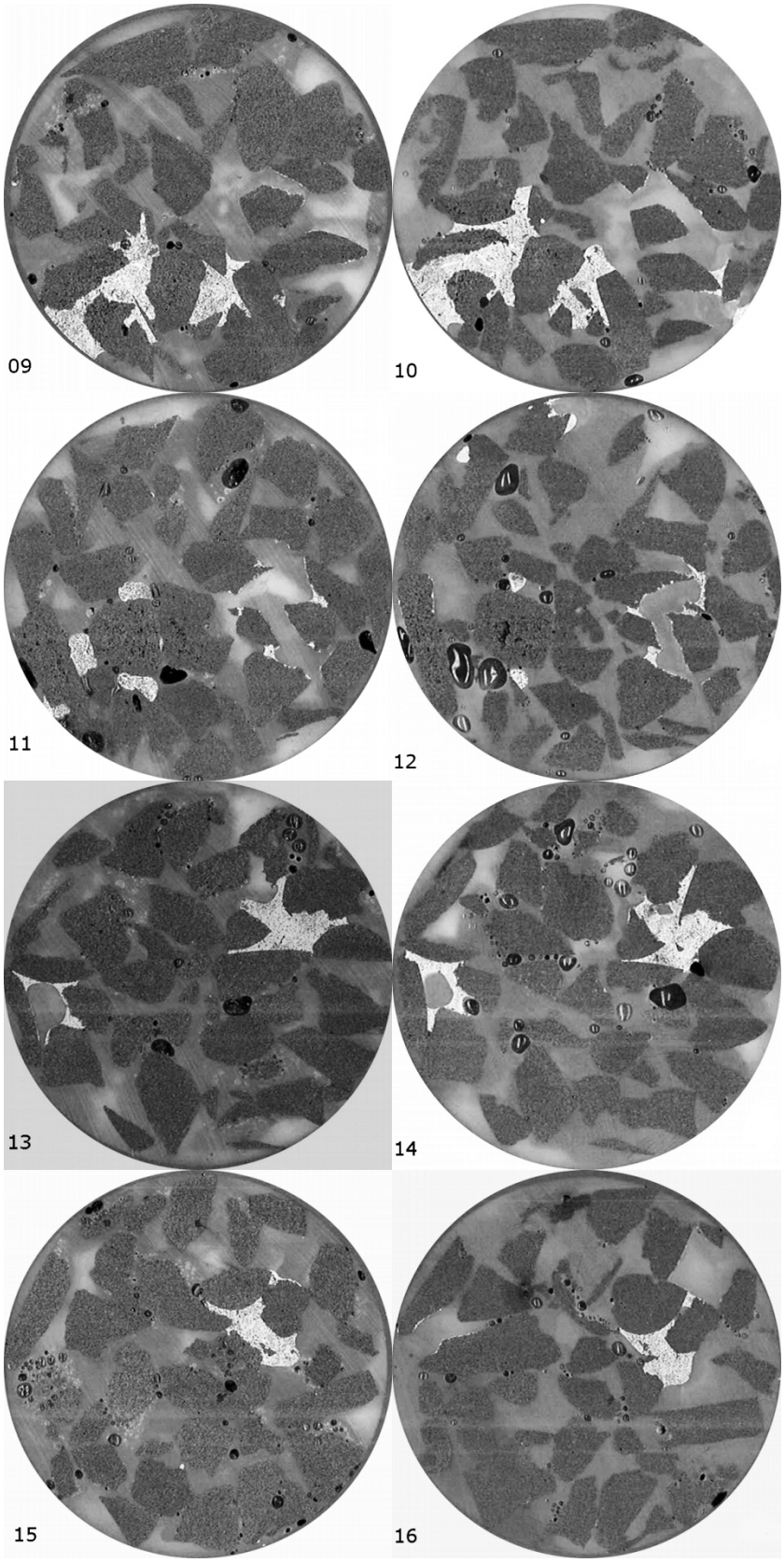
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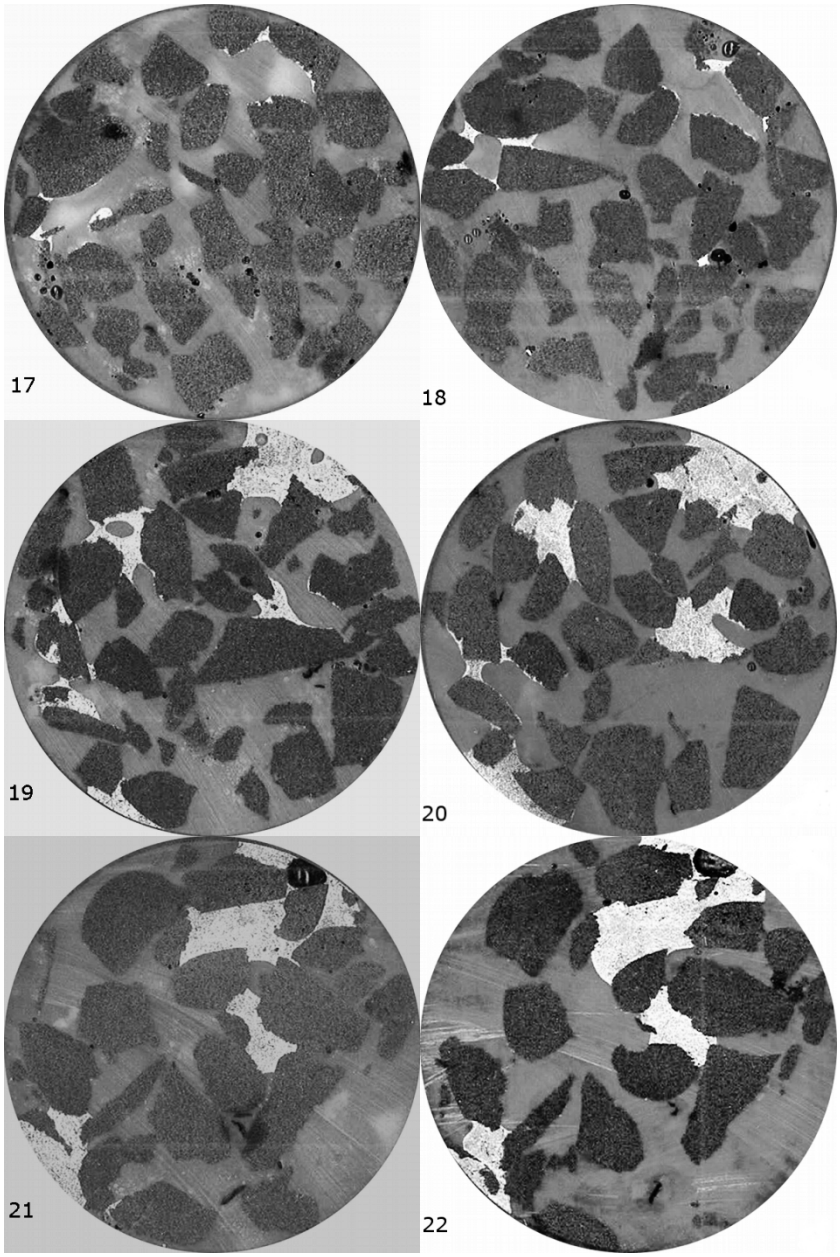

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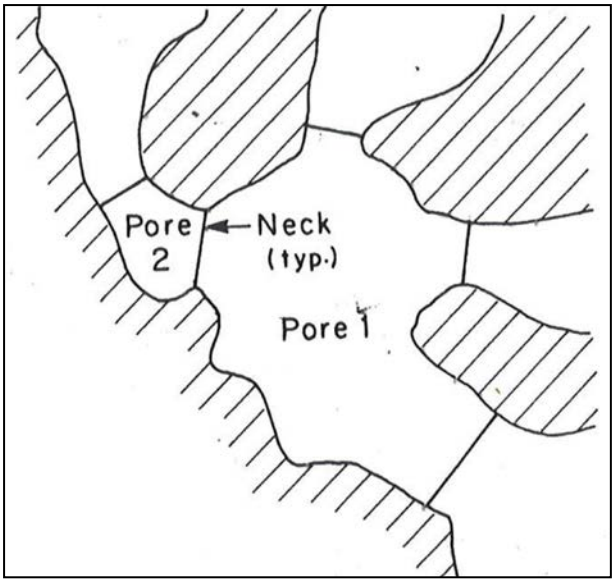

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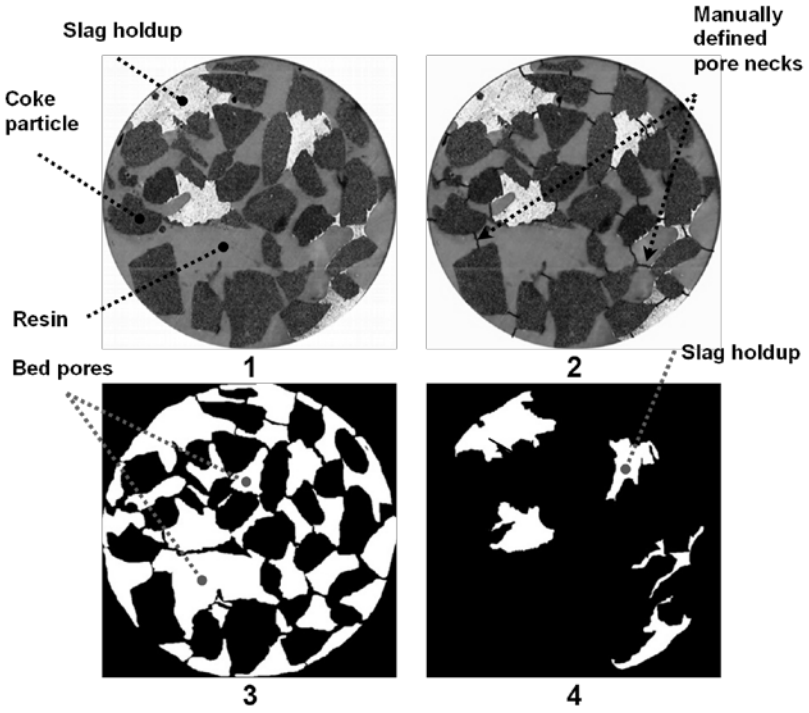

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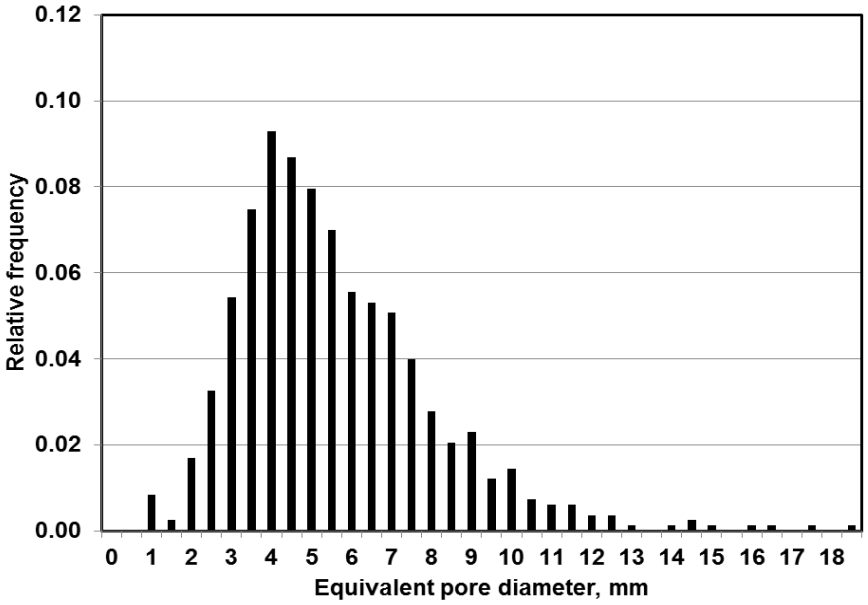

Fig. 4 A typical example of the distribution of pore equivalent diameter within the bed - ρ_B: 55%. Experimental duration = 90 minutes.

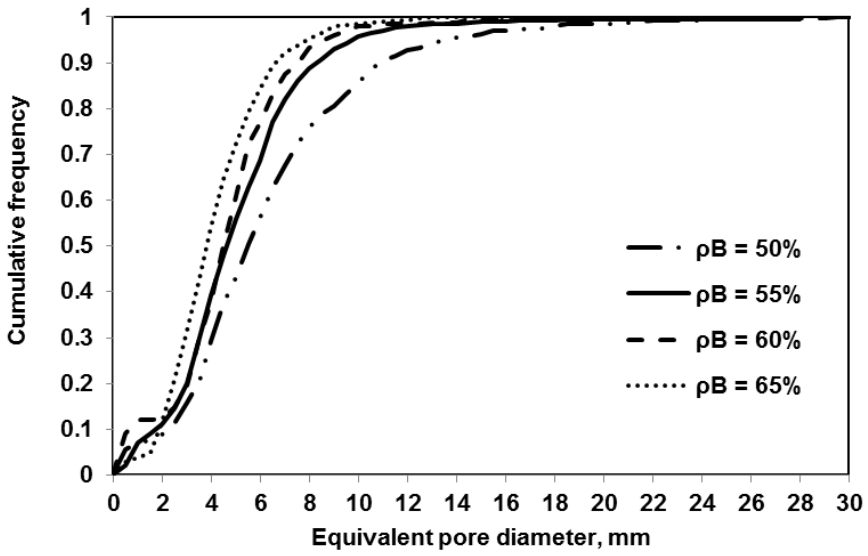

Fig. 5 Cumulative distribution of pore equivalent diameter within the bed. Experimental duration = 90 minutes.

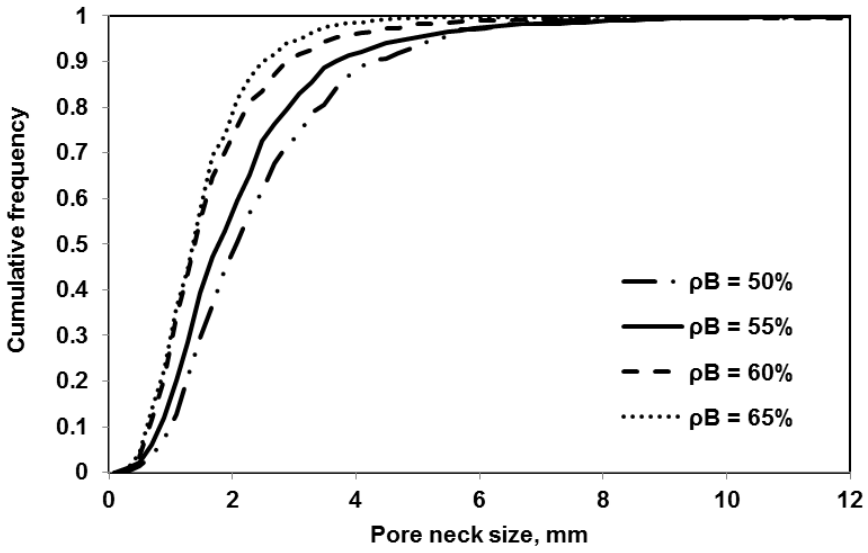

Fig. 6 Cumulative distribution of pore neck size within the bed. Experimental duration = 90 minutes.

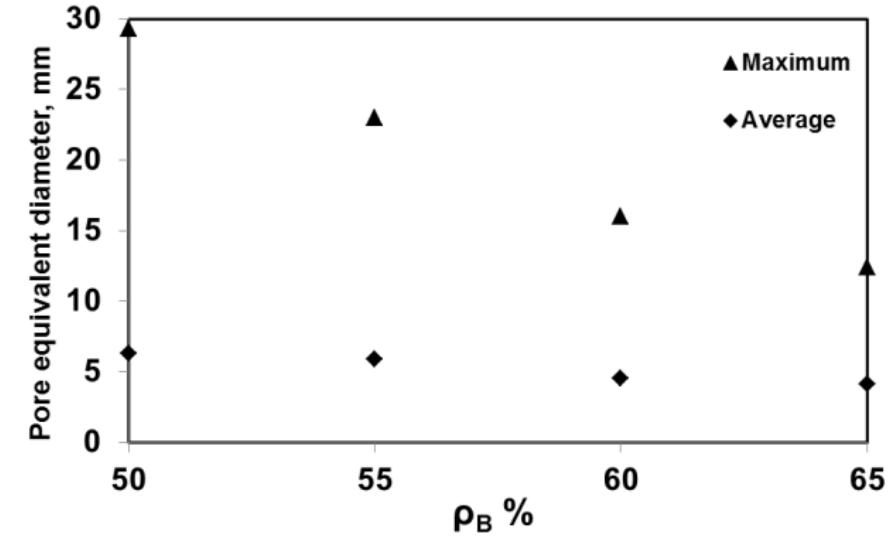

Fig. 7 Estimated pore equivalent diameter versus bed packing density. Experimental duration = 90 minutes.

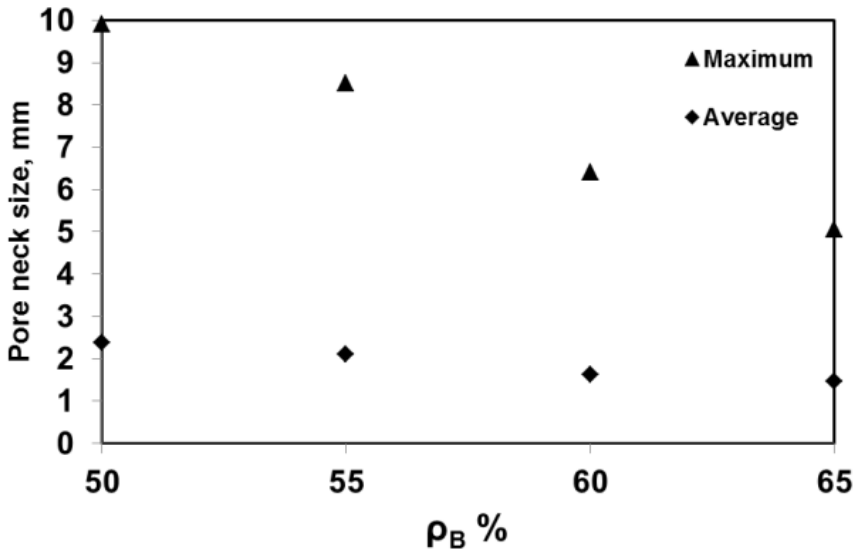

Fig. 8 Estimated pore neck size versus bed packing density. Experimental duration = 90 minutes.

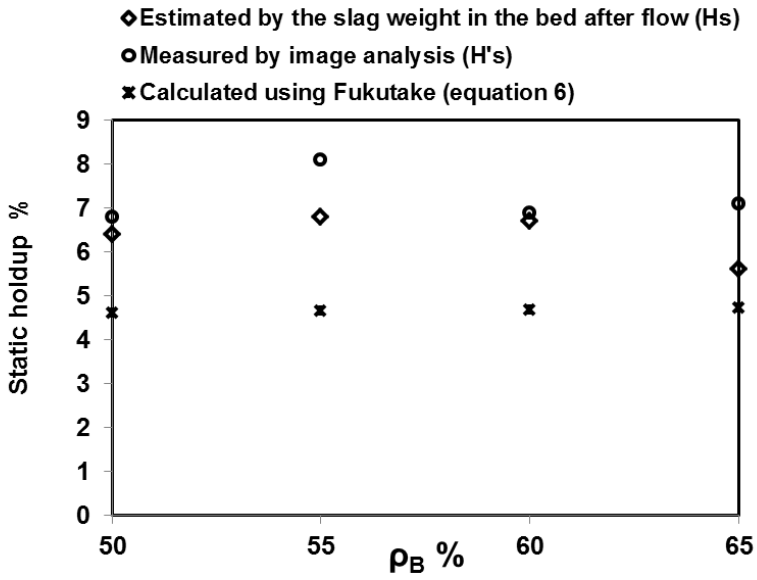

Fig.9 A Comparison of the static holdup estimated by bed mass method, estimated by image analysis method and calculated by Fukutake model for different packing densities. Experimental duration = 90 minutes.

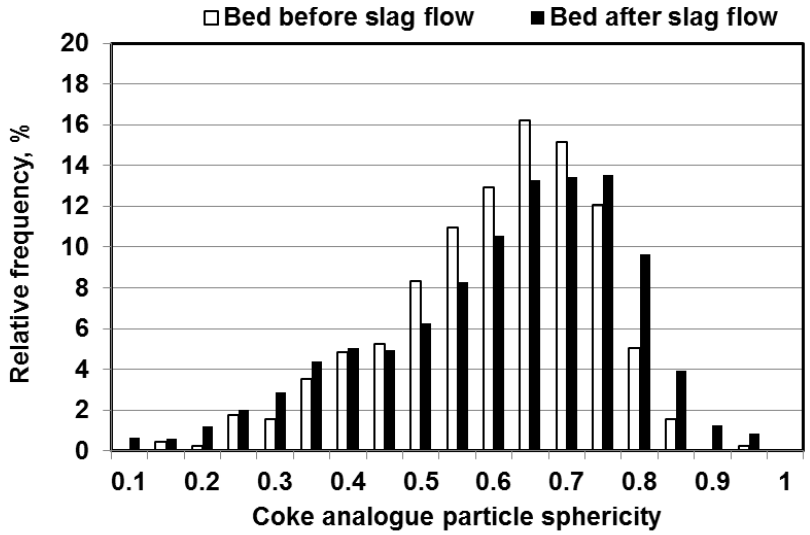

Fig. 10 Estimated coke analogue particle sphericity for a bed before slag flow and after slag flow - ρ_B=55%. Experimental duration = 90 minutes.

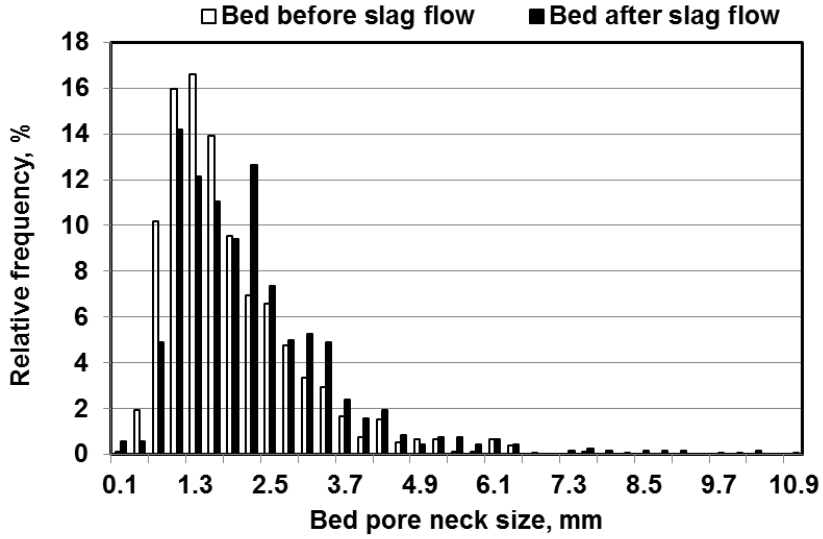

Fig. 11 Estimated bed pore neck size distribution for a bed before slag flow and for a bed after slag flow - ρ_B=55%. Experimental duration = 90 minutes.

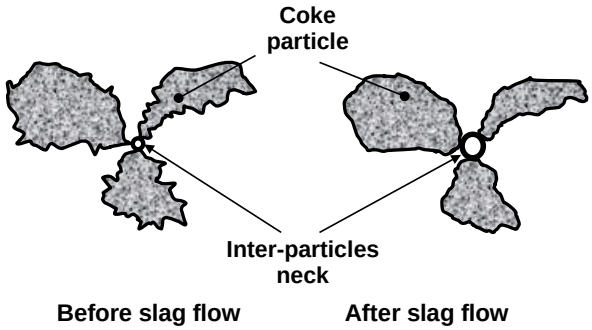

Fig. 12 A schematic illustration of the change in coke particle roundness and pore neck size due to slag flow.

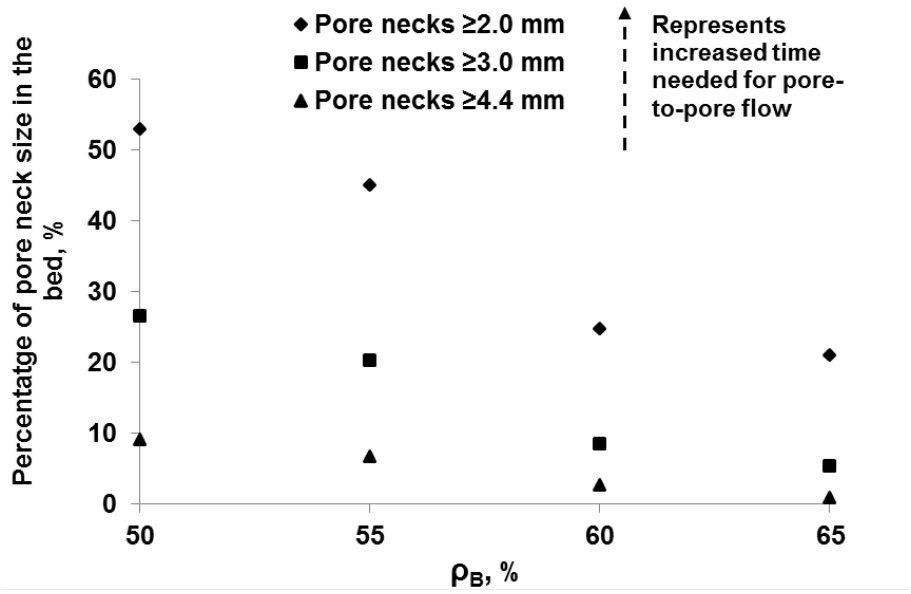

Fig. 13 The variation of the estimated pore neck size percentage by image analysis plotted against bed packing density.

Table 1 Comparison of the bed packing density measured by image analysis method before and after experiment. Nominal ρ_B=55%.

Packing density measured by image analysis ρ'_B, %	**Before Slag flow**	**After slag flow**
	47.8	**48.6**

Table 2 Summary of the average bed packing properties obtained from image analysis of the cooled down beds.

Nominal Packing density	Particle sphericity	Pore size, mm	Pore neck size, mm	% of pore necks ≥4.4 mm	% of pore necks ≥3.0 mm	% of pore necks ≥2.0 mm
50%	**0.62**	**6.3**	**2.4**	**9.1%**	**26.7%**	**53.0%**
55%	**0.61**	**4.8**	**2.1**	**6.8%**	**20.4%**	**45.1%**
60%	**0.59**	**4.5**	**1.6**	**2.7%**	**8.5%**	**24.9%**
65%	**0.63**	**4.1**	**1.4**	**0.9%**	**5.4%**	**21.1%**