# CALCINED COKE PARTICLE SIZE AND CRUSHING STEPS AFFECT ITS VBD RESULT

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#### Abstract

The size of a calcined coke particle used in the Vibrated Bulk Density (VBD) test and the size of the particle before crushing affects its VBD analysis. That is, naturally occurring particles usually have a higher packing density and VBD compared with particles that are crushed to the same size. Consequently, calcined coke preparation crushing steps can dramatically affect the VBD result. Data, showing how calcined coke particle size and crushing steps affect the VBD result will be presented. These data help explain why the roll crushing steps need to be controlled to improve VBD repeatability and reproducibility. In addition, data will be presented showing how the roll crusher operation and maintenance affects the VBD result.

# Introduction

The ASTM D4292 calcined coke Vibrated Bulk Density, (VBD) test defines sample preparation to control the process and improve the repeatability of the test. Although the VBD of a calcined coke is affected by many production variables, only the particle's size and crushing steps will be discussed in this paper.

There have been many references in the literature as to how particle size affects the VBD test results <sup>(1-5)</sup>. These papers show:

- The size of the coke particle affects VBD result-Usually Larger size particles have lower VBD,
- Crushing larger size particles to a given size, usually results in a lower VBD than particles that are naturally occurring.
- Since larger sized particles result in lower VBD, any change in the starting particle size distribution will affect the VBD result. For example samples that are subject to segregation will affect the VBD result. The following Figure 1 shows how the VBD varied as the %+4 mesh changed while a ship of calcined coke was being unloaded <sup>(1)</sup>.

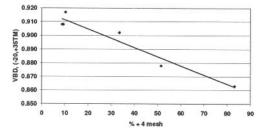


Figure 1 VBD of Calcined Coke vs %+4 mesh

Several papers <sup>(2, 4, 6)</sup> also suggest that particle shape has a significant affect on the packing density and therefore the Vibrated Bulk Density.

# **ASTM Sample Preparation Procedure**

The sample preparation section of ASTM D4292 addresses the crushing steps and gives ranges for roll crusher settings. Even within the accepted range of at least 30% crushed and the ratio of the coarser to finer product 0.8 to 2.0, significantly different VBD results can be obtained. For most cokes analyzed for VBD (-28+48 Tyler mesh), greater % crushed and lower coarser to finer ratios result in lower VBD results. Outside of these specified ranges even larger VBD differences will be observed. Consequently, the crushing steps and roll crusher settings need to be monitored to get more repeatable results.

The crushing steps consist of first a jaw crushing step with a recommended 5 mm at the closest setting. For our lab, the furthest gap is around 5 mm with the closest gap almost 2 mm. Using the jaw crusher closest gap at 5 mm generates a roll crusher feed that plugs the roll crusher. Even though the jaw crushing furthest gap is set at 5 or 6 mm, particles that are significantly larger in length or height get through with the width of the largest particles 5 to 6 mm.

This VBD study was initiated because our carbon lab consistently obtained bias low VBD results compared to an outside lab. For Jan 2010 through early May, our in-house lab weekly VBD monitoring (40 samples) averaged 0.853 g/cc for -28+48 mesh calcined coke whereas an outside lab daily monitoring (104 samples) VBD averaged 0.872 g/cc. That is, our in-house lab averaged almost 0.02 g/cc lower VBD. When the same prepared VBD sample was analyzed at each lab, the results were almost identical. Even though each lab used the same roll crusher feeler gauge gap, it became apparent that the crushing steps were probably affecting the VBD result. This observation led to a close inspection of our roll crusher. By measuring the roll crusher gap at different positions before and after roll crushing, it was found that the rollers had worn unevenly such that the gap varied from the outside edge to the middle and at different positions of the rollers. Feeler gauges were needed to observe this variation. In addition, the gap changed from the beginning of roll crushing to after roll crushing. This change in gap from beginning of the roll crushing to after roll crushing had the largest impact for the closer roll crushing setting. Up to 0.1 mm reduction in gap was measured for the closest roller crusher gap setting of 0.48 mm. A complete rebuild of the roll crusher was performed before performing the tests presented in this paper.

The type of feeler gauges used to set the gap also makes a difference in the actual gap between rollers. Using automotive type feeler gauges to set a roller gap will result in a gap that is slightly greater than using a custom made single feeler gauge. Using automotive feeler gauge often results in using one very thin gauge which can bend. Consequently, the gap is often slightly larger when the automotive feeler gauges are used to set the gap. The final roll crusher setting is the most critical gap in producing the actual size distribution of the coke for the ASTM VBD measurement.

# **Experiment and Results**

Experiments were run to quantify the effect of crushing the larger particles to the desired particle size (-28 +48 Tyler mesh) for the VBD analysis. Four different single source cokes were tested. Their properties are found in the following table:

	Table 1
cal	properties of Calcined Cokes in pan

Chemical properties of Calcined Cokes in paper						
Coke	Α	B	C	D		
Sulfur, %	0.69	2.72	0.96	3.18		
Vanadium, ppm	246	378	133	401		

Initially the ASTM D4292 was strictly followed. However, it became apparent that as the calcined coke was crushed more, the VBD of the calcined coke decreased. This observation led to additional crushing steps at closer roll crusher gaps followed by additional VBD analysis of the newly crushed calcined coke and another VBD analysis of the newly formed crushed coke blended with the formerly crushed coke. The following Figure 2 demonstrates how this was done and the VBD results obtained for Coke C.

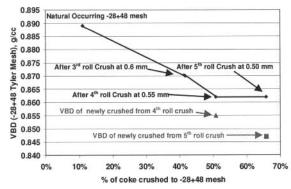


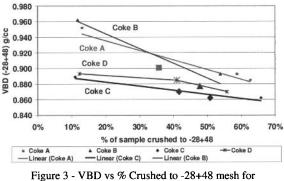
Figure 2 VBD of Coke C as function of % Crushed

This plot shows that 11% of Coke C was in the -28+48 mesh size range and has a VBD of 0.889 g/cc. After following the ASTM roll crushing step with the  $3^{rd}$  roll crusher set at 0.6 mm, 41.6 % of the coke was in the -28+48 mesh range and had a VBD of 0.870 g/cc and a coarser to finer sized ratio of 1.4. Additional crushing of coke larger than 28 mesh at a 0.55 mm roll crusher setting resulted in a coke with a VBD of 0.855 g/cc. When this coke was blended with the previous -28+48 crushed coke, the VBD was 0.862 g/cc with 50% crushed and a coarser to finer ratio of 0.89. Both of these crushed samples meet the VBD results.

Performing a 5<sup>th</sup> roll crushing on the particles 28 mesh and larger produced a calcined coke having a 0.847g/cc VBD.

When this material was blended with the previously crushed calcined coke, its VBD was 0.862 g/cc with 68 % crushed and a coarser to finer ration of 0.12. This experiment shows how continuing to crush larger particles of Coke C results in calcined coke with a lower VBD (poorer packing density).

The same trend was observed for most other cokes tested (See following Figure 3). These four calcined cokes demonstrate that the natural occurring particles have a higher VBD than crushing larger particles to the same size. However, it has also been observed that the natural occurring particles can have lower VBD.



Coke A through Coke D

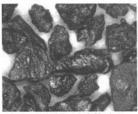
Larger Symbols points meet ASTM Crushing Over/Under Ratio

The Vibrated Bulk Density of cokes crushed in a roll crusher, converge as the cokes are crushed more. See the following Table 2. The natural size calcined cokes have a delta VBD difference of 0.140 g/cc whereas the crushed calcined cokes have a VBD delta of only 0.031 g/cc. Since crushing larger calcined coke particles with greater internal pores opens up these pores, the finer cokes would be expected to have more similar packing densities. In fact, one of the techniques of increasing anode density of calcined cokes with lower packing density is to use them in the fines fraction. However, cokes with higher packing density do produce anodes with higher Baked Apparent Density.

Table 2 Comparing the VBD of Natural Occurring -28+48 mesh calcined coke to VBD of ASTM prepared -28+48 after crushing

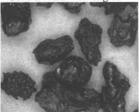
Coke	Natural VBD	% Crushed to	ASTM VBD	
	(-28+48 mesh),	-28+48 for	(-28+48 mesh)	
	g/cc	VBD Test	g/cc	
В	0.962	54	0.893	
Α	0.952	63	0.885	
D	0.893	56	0.870	
C	0.889	68	0.862	

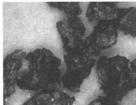
The calcined coke's natural occurring -28+48 mesh particles were examined under 210 magnification to see if particle shape differences can be observed to help understand differences in VBD. These pictures are presented in Figure 4.





Coke B VBD =0.962g/cc





Coke D VBD =0.893g/cc

Coke C VBD=-0.889 g/cc

## Figure 4 - Pictures of calcined coke particles which have different VBD properties

Comparing cokes A & B particle shapes to cokes C & D particles shows cokes C & D particles have many bumps and rounded appendages which could reduce particle packing density.

#### Conclusions

- The sample preparation procedure for ASTM D4922 . defines how to crush the calcined coke, but even in the range specified the VBD can vary. Outside of this range significantly greater variability is observed.
- Crushing larger particles or increased crushing usually leads to lower VBD results.
- It appears that particle shape has an affect on the VBD result with smoother surface of naturally occurring having a higher VBD due to closer packing properties.
- Although often overlooked, the operation of the roll crusher can have a dramatic affect on the VBD analysis of a given coke. Monitoring the consistency of roll crusher gaps can identify when the roll crusher needs to be rebuilt.

#### Recommendations

This study was initiated due to observing that in-house lab obtained VBD results were biased lower than an outside lab. Due to this, a quality control comparison of results between labs has been established that compares the % crushed to the desired particle size and the over/under ratio. In addition, a maintenance monitoring of the roll crusher has been established and includes:

- Daily checking that the roll crusher gap via a feeler gauge is the same before and after crushing coke particles.
- Daily observing the grease around the bearings.
- Monthly adding grease to the bearing casing.
- Installing a clutch so the roll crusher starts smoother instead with a jerk. We suspect the greatest wear is due to the jerking start of the rollers.

We recommend other labs performing VBD tests consider a similar VBD monitoring and roll crusher maintenance program.

## Acknowledgement

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#### References

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