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Vibrated Bulk Density using Semi-automated Device: Simplifying Sample Preparation while Improving Accuracy and Precision

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Abstract

Vibrated Bulk Density (VBD) of calcined petroleum coke is widely utilized as one measure of quality and is useful for quantifying binder demand in the anode production process of the aluminum reduction industry. ASTM D4292-10 is one of the methods used widely by the carbon and aluminum reduction industries to measure VBD. Recently the carbon producer and consumer industries have jointly worked together to increase the reliability and benefit of this method. Geopyc is a semi-automated device that potentially yields better precision compared to traditional vibrator device specified in D4292. This paper presents an innovative use of the Geopyc instrument in measuring VBD, the results of which correlate well with D4292; especially to the 28x48 Tyler Mesh size fraction. This novel sample preparation does not involve complicated roll crushing steps as specified in the ASTM method(s) and thus simplifies the method while improving the accuracy and precision.

Introduction

The importance of Vibrated Bulk Density (VBD) in predicting the porosity of coke, pitch demand for production of anodes, and green anode density is well known and has been discussed several times recently and over the past several decades. Industry has developed ASTM and ISO methods to predict these parameters and several improvements in the methods have been published in the past.

The basic principle of these methods (1,2) is crushing and segregating a specific size fraction, and measuring the density by reading the volume of a known mass of sample in a vertical graduated cylinder vibrated at constant amplitude. Vibration was introduced to compact the coke particles and reduce and standardize the interstitial voids. The density of an individual size fraction is used as a measure of the quality of the coke, predicting pitch demand, and several other quality parameters. The crushing step in the methods was introduced because the coke is crushed in actual anode fabrication. The reason for selecting the specific size fractions commonly used in the test methodology was determined from empirical commercial data and laboratory correlations to predict pitch demand for a given plant aggregate granulometry. For example Soderburg Anode Plants used (8X14 Tyler Mesh (TM)) as their aggregate formula was coarse and Prebaked Anode Plants historically used (28X48 TM) as their commercial aggregate formula was finer. Sample mass, cylinder dimensions, particle size and shape, vibration amplitude and frequency as well as many other variables all have great influence on the density result and so it is important to obtain the narrow size fraction to reduce variability and obtain consistent data. Apart from these parameters, variation in crushing equipment, particle size and crushing steps (3), degradation in coke quality, and blending shot coke and sponge coke requirements have questioned the reliability of the current VBD methods.

Recently the Micromeretics Geopyc semi-automated instrument has gained attention in the calcined coke industry for its application of measuring VBD (4). The unique feature of this instrument is that it uses transverse axial pressure to measure bulk volume. The cylinder used by this instrument is 1.5 times the diameter of the cylinder used in the conventional method, and the cylinder is horizontal and applies transaxial pressure gradually to compact the particles as close as possible.

With the increase awareness of concerns related to the sample preparation, VBD measurement and lack of estimating the actual porosity of coke using current VBD methods (5), it is desirable to include an automated or semi-automated device for measuring the vibrated bulk density. This motivated our team at the A J Edmond Company to review the development of the ASTM D4292 procedure.

In the 1970's, Belitskus (1-2) found an alternative way of measuring the bulk density of coke aggregates using available technology. He used a unique sample preparation technique, a specific particle size fraction, vibrating table and a graduated cylinder. He determined that the 28x48 TM size fraction is the best single fraction for indicating the overall bulk density of a coke aggregate. He rewrote his method as ASTM D4292 and since then the industry has been using this method at various stages starting from production of calcined coke to anode fabrication. The basis of his theory was the correlation of bulk density of a specific size fraction with the maximum achievable bulk density of the aggregate granulometry. This inspired us to experiment with his concept using the latest available technology.

This paper will present the VBD data obtained by the Geopyc instrument on samples prepared by jaw crushing and screening only. The data indicates that simply jaw crushing and screening out a portion of the fines is sufficient sample preparation to yield repeatable data. The density obtained by the Geopyc on these samples showed very good correlation with ASTM D4292-10 (28x48 TM) and thus this data represents the actual maximum bulk density of coke aggregate.

Experimental Details

Rotary calcined and shaft calcined coke produced from different parts of the world were used in this experiment. Each sample was processed up to the jaw crushing step described in ASTM D4292-10 and then screened to remove fines. The raw calcined coke samples were rotary riffled and/or riffled using a table top splitter to obtain small sized representative analytical samples. One split of the sample was analyzed for VBD following ASTM D4292-10 (28x48 TM). The VBD results obtained from the selected set of samples had a range of 0.833 gm/cc to 0.991 gm/cc. The other representative splits were processed and analyzed per the description listed below. All samples were analyzed in duplicate to ensure the quality of the data collected.

1. Jaw crush 1 kg sample slowly (jaw crusher opening was set at 5 mm per ASTM D 4292)
2. Riffle split the minus 5 mm coke to obtain four 200-250 gm splits.

Experiment - 1
1. Riffle split 200-250 gm coke sample to 60-70 gm and analyze for VBD using Geopyc.

Experiment - 2
1. Screen 200-250 gm coke sample to obtain plus 50 mesh fraction
2. Riffle split plus 50 mesh coke sample to 60-70 gm and analyze for VBD using Geopyc

Experiment - 3
1. Screen 200-250 gm coke sample to obtain plus 70 mesh fraction
2. Riffle split plus 70 mesh coke sample to 60-70 gm and analyze for VBD using Geopyc.

Experiment - 4
1. Screen 200-250 gm coke sample to obtain plus 100 mesh fraction
2. Riffle split plus 100 mesh coke sample to 60-70 gm and analyze for VBD using Geopyc.

Results and Discussion

The VBD data obtained by both ASTM D4292-10 and Geopyc device were compared to determine the correlation. Figure 1-4 shows correlation coefficient between two methods for each set of experiments. VBD Geopyc results of samples jaw crushed to minus 5 mm correlates with VBD ASTM D4292-10 with a R^2 = 0.9084, VBD Geopyc results of samples jaw crushed to minus 5 mm and screened to +50 mesh correlates with VBD ASTM D4292-10 with a R^2 = 0.8914, VBD Geopyc results of sample jaw crushed to minus 5 mm and screened to +70 mesh correlates with VBD ASTM D4292-10 with a R^2 = 0.9725, and VBD Geopyc results of samples jaw crushed to minus 5 mm and screened to +100 mesh correlates with VBD ASTM D4292-10 with a R^2 = 0.9812.

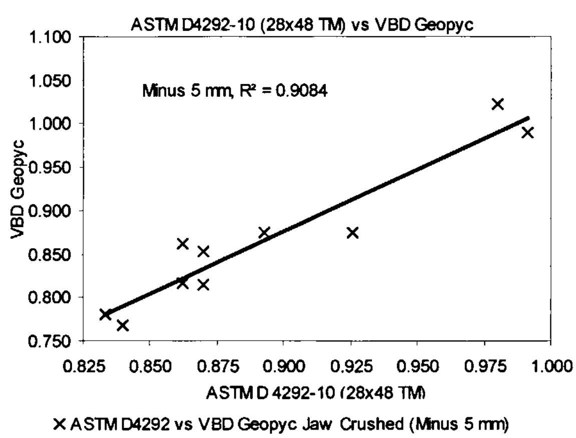

Figure 1. Correlation of VBD ASTM D4292 (28x48 TM) vs. VBD Geopyc (Minus 5 mm)

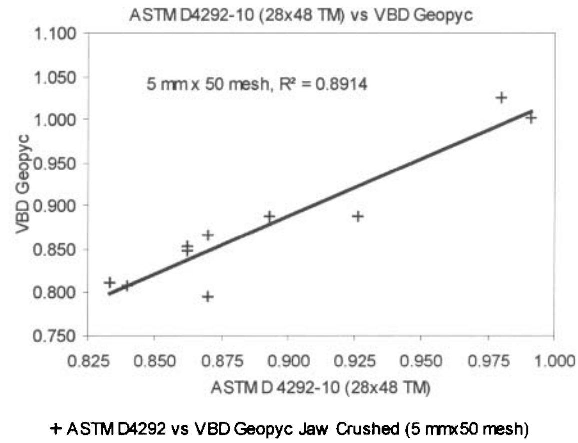

Figure 2. Correlation of VBD ASTM D4292 (28x48 TM) vs. VBD Geopyc (5 mm x 50 mesh)

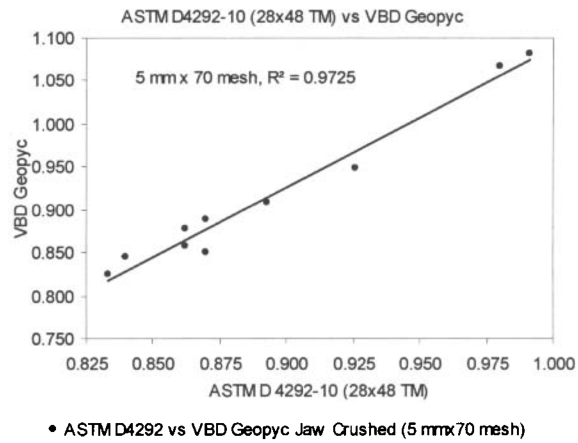

Figure 3. Correlation of VBD ASTM D4292 (28x48 TM) vs. VBD Geopyc (5 mm x 70 mesh)

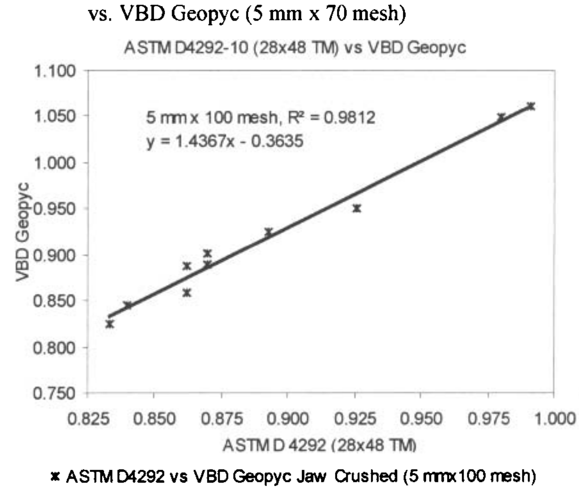

Figure 4. Correlation of VBD ASTM D4292 (28x48 TM) vs. VBD Geopyc (5 mm x 100 mesh)

Based on the initial data it was determined that the jaw crushed to minus 5 mm and screened to plus 100 mesh material correlates best with the VBD ASTM D4292-10 data. Using this as a base line, it was decided to run another experiment with 10 unknown coke samples and calculate the VBD ASTM D4292-10 value from the Geopyc data using the correlation coefficient equation.

Coke	VBD Geopyc (5 mmx100 mesh)	Actual ASTM D4292-10 (28x48 TM)	Calculated ASTM D4292-10 (28x48 TM)	Delta
1	0.895	0.877	0.876	0.001
2	0.898	0.885	0.878	0.007
3	0.906	0.901	0.884	0.017
4	0.921	0.908	0.894	0.014
5	0.947	0.901	0.912	-0.011
6	0.919	0.893	0.893	-0.000
7	0.865	0.847	0.855	-0.008
8	0.934	0.909	0.903	0.006
9	0.913	0.877	0.888	-0.011
10	0.930	0.901	0.900	0.001

Table 1. Comparison of VBD ASTM D4292-10 and Calculated VBD ASTM D4292 from VBD Geopyc

The data shown in Table 1 indicates that the Geopyc VBD obtained on a sample prepared by the method proposed in this paper can calculate VBD ASTM D4292-10 well within the repeatability of the ASTM method.

Conclusions

- Belitskus (1) proved that the VBD of 28x48 fraction determined by Vibrator and Cylinder (revised as ASTM D4292) correlates best with bulk density of coke aggregates. The VBD Geopyc data presented in this paper shows that Geopyc VBD of Jaw Crushed to minus 5 mm and screened to +100 mesh sample correlated best with ASTM D4292-10 and thus Geopyc VBD data represents the maximum bulk density of raw coke or coke aggregate.
- The experimental method is very easy to perform i.e. jaw crushing and screening.
- The Geopyc instrument is a semi-automated device and it eliminates common errors related to Cylinder and Vibrator prescribed in ASTM D4292-10, and thus it provides better accuracy and precision.
- AJE does not recommend the Geopyc instrument as an alternate device to measure VBD, but we foresee potential application of this device in measuring the maximum bulk density of blends of coke aggregates and anode butts used in anode fabrication.

Future Development:

A J Edmond Company will further develop this method and study the application of the data in improving Anode Density, estimation of pitch requirement for high density anodes, and other aspects of making optimum quality Anodes.

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