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Improvements of Vibrated Bulk Density Analysis at VM-CBA and Petrocoque S.A

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Abstract

Anodes for aluminum production are composed of coal tar pitch (CTP) and calcined petroleum coke (CPC). The anode composition depends on the reduction technology. For Søderberg anodes, as used by VM-CBA, the coke and pitch contents range from 67 to 79% and from 33 to 21%, respectively. Coke quality control includes sampling and analysis of chemical and physical properties. These tasks are associated with uncertainties, which may lead to wrong decisions. Since 2010, VM-CBA and Petrocoque S.A studied possibilities to reduce the standard deviation (STD) of the vibrated bulk density (VBD) analysis. The result of this work was a reduction of the difference between the VBD results of the two labs from 0.018 to 0.006 g/cm^3.

Introduction

VBD determination is described in standards procedures elaborated by the American Society for Testing and Materials (ASTM), the International Organization for Standardization (ISO), and specialized research centers. These organizations defined at least three different methods for VBD determination: ASTM D4292-10, ASTM D7454, and ISO 10236. The main differences among these procedures are the coke granulometry and the apparatus used.

VM-CBA and Petrocoque have been using the ASTM D7454 method. The VBD results of the two laboratories did not always agree. This issue has been addressed over the years through cross checks within suppliers, costumers, and specialized labs.

The Round Robin #19 report presented results from six labs that routinely use the ASTM D7454 method [1]. The corresponding lab averages results show good agreement between the labs (Fig. 1), suggesting that this method has potential for satisfactory precision.

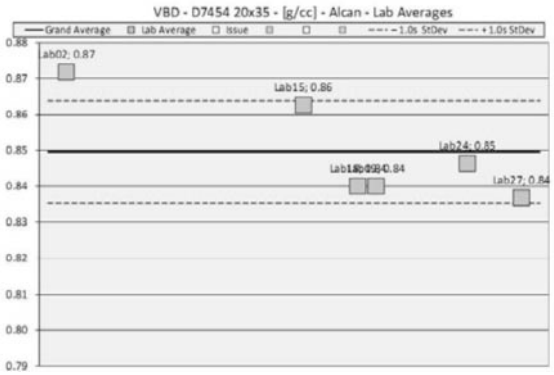

Figure 1. VBD lab averages, determined by the ASTM D7454 method for a CPC sample [1].

Lossius, Spencer, and Øye summarized aspects that might impact agreement between labs [2]:

- Feeding with or without vibration
- Fixing the measuring cylinder to the vibration table or not
- Speed of sample introduction into the measuring cylinder
- Different types of crushers for sample preparation
- Length of vibrating time for the test portion

The authors of reference [3] concluded that the type of crusher and the gap setting used can result in variations of the VDB. Variations can even arise when the same crusher is used to prepare different coke types.

Cannova, Canada, and Vitchus [4] studied the influence of the crushing steps, particle size, and particle morphology. The authors monitored how the coke granulometry changes during ship unloading and measured the impact on the VBD. The influence of particle segregation on the determined VBD is presented in Figure 2. The data clearly show that segregation has a significant impact on the measured VBD.

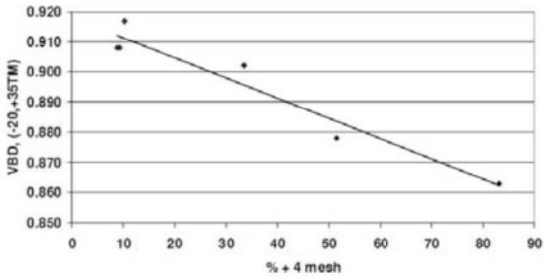

Figure 2. Impact of coke segregation on the VBD [4].

In the same study, the authors also investigated the influence of the degree of crushing on the VBD (Figure 3). It was concluded that "***it became apparent that as the calcined coke was crushed more, the VBD of the calcined coke decreased***". The observation of Cannova *et al.* suggests that crushing during sample preparation should follow the standards and deviations are not acceptable. This statement was reinforced by Laplante *et al.* [5].

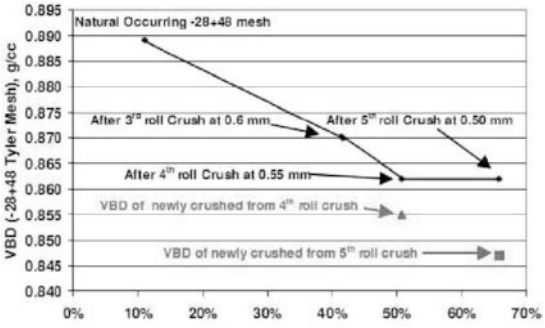

Figure 3. Impact of crushing on the VBD [4].

All standard procedures for VBD analysis address how sample preparation and analysis have to be performed. Unfortunately, the process is not totally automated, which may result in variations of the results. The technicians performing the analysis should have good knowledge of the procedure and of the equipment. Furthermore, the influence of each step on the final result has to be known.

Based on the literature review, the following points are addressed in the present work:

- Equipment evaluation (maintenance and characteristics) in the two labs;
- Sampling
- Sample preparation procedures;
- Cross-checks between the two labs;
- Critical analysis of the whole process to identify differences;
- Training of all staff involved reflecting the points above

As a result of this work the difference between the VBD results of the two labs was reduced from 0.018 to 0.006 g/cm³.

Improvements of the VBD analysis procedure

Equipment evaluation

As mentioned above, the work was divided into several steps. The first one was an evaluation of the equipment and methods used for sampling, sample preparation, and VBD analysis.

The equipment is a critical factor in achieving good reproducibility between labs. All equipment in the two labs was inspected and the equipments specifications were compared. As can be seen in the figures 4(a) (f), the equipment was in good shape and no significant differences were observed.

The instrument for the VBD measurement is semi-automated and consist of a control panel, a vibrating bowl, and a graduated cylinder equipped with a photo detector fixed to an electromagnetic jogger. The control panel allows the adjustment of the vibration set point of the electromagnetic jogger and the control of the material flow passing through the vibrating bowl [5].

In the VBD apparatus, vibration is induced by an electromagnetic field. The vibration frequency was recorded in order to evaluate the stability of the jogger. A pen vibration meter was used and the results showed a wide range of frequencies, which increased variation of the VBD results. Instability of the electricity network supplying the apparatus was identified as reason for the frequency variation. In order to stabilize the power supply a surge protector was installed.

Sampling procedure

The sampling process is critical for the accuracy of the analysis. Several aspects have an impact on this step and may result in wrong results. At VM-CBA, sampling was done on a conveyor belt feeding the carbon plant. This conveyor belt receives coke from a 5 metric tons storage silo. Segregation in the silo introduces variation in the coke granulometry. This could result in variation of the determined VBD and an increased standard deviation.

Figure 4(a) VM-CBA's STAS VBD Apparatus

Figure 4(b) Petrocoque's STAS VBD Apparatus

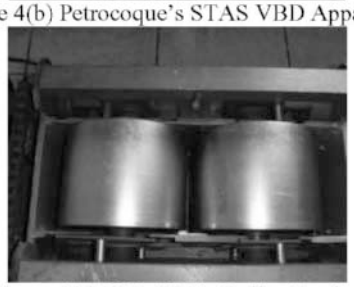

Figure 4(c) VM-CBA's Roller Crusher

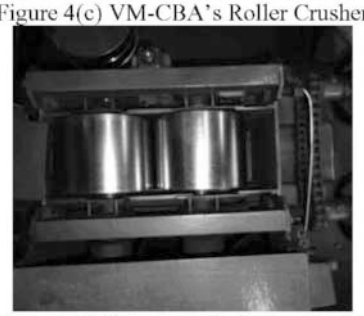

Figure 4(d) Petrocoque's Roller Crusher

Figure 4(e) VM-CBA's Sieving Apparatus

Figure 4(f) Petrocoque's Sieving Apparatus

The granulometry of coke samples from the conveyor belt varied considerably. This was also the case for the -4 +10 Tyler mesh fraction (Fig. 5). The variations have a negative impact on the accuracy of VBD analysis. This could cause wrong decisions, including non-justified process adjustments at the carbon plant or complains to the CPC supplier regarding the coke quality.

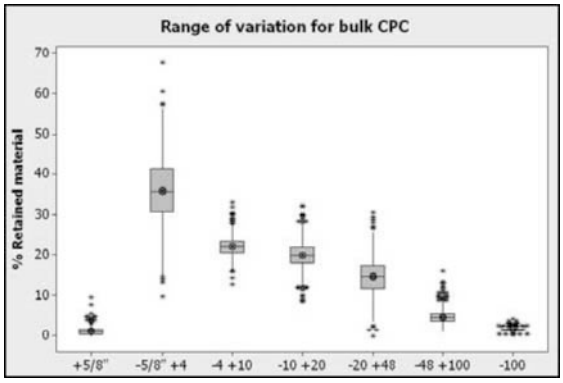

Figure 5. Range of granulometries of coke samples collected from the conveyor belt.

In order to eliminate these shortcomings, the sampling procedure used at Petrocoque was deployed at VM-CBA. Coke is now sampled on trucks at nine locations (Figure 6) just before unloading. A special tool was developed to collect samples 30 cm below the surface of the coke bed. Sampling starts next to the drivers cab (position 1 in Figure 6) until to rear end of the truck is reached (position 9). The nine samples are combined for analysis.

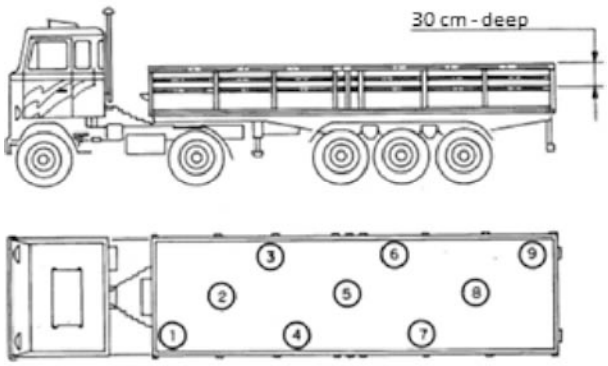

Figure 6. Locations of coke sampling on trucks

Procedures

To ensure consistency of the VBD analysis, statistical process control was introduced (Fig. 7). Prior every VBD test, the technician verifies the equipment parameters and analyses a reference sample, provided by STAS. This sample has a certified VBD of 0.871 ± 0.006 g/cm³. When the measured VBD value is outside these limits, a check list is followed until the problem is resolved.

However, this procedure is not the best approach as it calibrates the apparatus against just one point. This means for samples with VBDs considerably different from the reference, the results are doubtful.

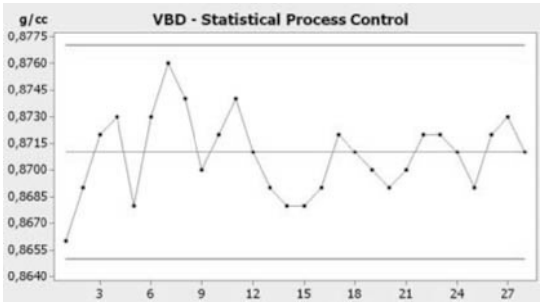

Figure 6. VBD SPC chart.

Up to now no solution for this problem exists. However, at our labs, a second reference sample was selected that has a similar VBD as the CPC supplied by Petrocoque to VM-CBA. The VBD apparatus is now calibrated against the second reference sample.

Agreement between the two labs

Since 2006, VM-CBA and Petrocoque have an agreement to carry out one cross check per year. This allows to compare the VBD results and to determine standard deviation. This evaluation includes preparation of coke samples in one lab and analysis in triplicate in each of the two labs. The data are statically analyzed and the final results are reported in terms of average VDB and standard deviation.

In 2009, a large difference between the average VBD values was observed. This was not a new issue and the program described above was initiated in order to improve the agreement between the two labs. Furthermore, it was agreed to perform two cross checks per year. The differences between the labs decreased (Tab. 1). The stabilization of the power supply of the VBD apparatus had a major contribution.

Table 1. Results of cross checks between VM-CBA and Petrocoque-S.A

Date	Sample preparation at	Number of samples	VM-CBA (V)		Petrocoque (P)	
			VBD (g/cm³)	STD (g/cm³)	VBD (g/cm³)	STD (g/cm³)
Nov. 09	C	20	0.838	0.009	0.856	0.001
Dec. 10	C	15	0.799	0.010	0.804	0.002
Mar. 11	P	5	0.842	0.008	0.836	0.004
Nov. 11	C	20	0.829	0.006	0.837	0.003
Nov. 11	P	20	0.843	0.004	0.837	0.002

In order to verify the agreement between the two labs after the improvements reported above, an analysis of variance (ANOVA) test was performed. It covered 175 coke samples collected at the two companies. With 95% confidence, there was no statistically significant difference between the VBD results of the two labs.

The distribution of VBD results from 134 samples measured at the two labs is shown in Figure 8. The average results were virtually the same with a difference smaller than 0.001 g/cm³ between the two labs.

Conclusions

This study led to the following conclusions:

a) Periodic monitoring between the customer and suppliers labs is an excellent tool for anticipating possible deviations in the VBD measurement;

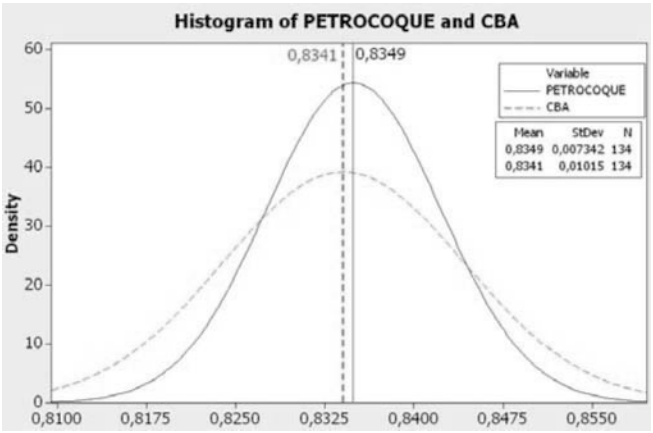

Figure 8. VBD results from 134 samples collected at Petrocoque and VM-CBA, respectively. Averages: 0.835 ±0.007 and 0.834±0.01 g/cm³

b) Perfect conditions and the correct use of VBD measurement equipment are crucial;

c) Cross-checks between customers and suppliers must be made with a minimum frequency of once per year

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