

## Acoustic Emission Techniques to Measure the Properties of Coke Particles – A First Foray

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### Abstract



The performance of the Hall-Héroult aluminium reduction process is strongly influenced by fluctuations of the baked carbon anode properties. The currently decreasing quality and increasing variability of the anode raw materials, coke and pitch, combined with the frequent changes by anode manufacturers to meet their specifications and reduce purchasing costs make it very challenging to produce anodes with consistent properties. Furthermore, the coke quality control scheme traditionally used by aluminium smelters involving infrequent coke sampling and characterization in the laboratory is inadequate for tracking coke variability when formulating the anode paste, and applying timely corrective actions when necessary. Developing new rapid and non-destructive sensors for measuring key coke properties such as density and porosity directly from the production line is highly desirable. This work investigates the possibility of using acoustic emission techniques for measuring physical and/or mechanical properties of coke particles. A set-up was developed to record the sound made by coke particles dropped on a metal sheet. The potential of the approach was tested on coke samples having different physical properties (several sizes and suppliers). The acoustic signature of each type of coke particle was correlated with their physical properties using regression analysis.

**Keywords:** Petroleum coke, pre-baked carbon anodes, acoustic emission, PCA, PLS.

### 1. Introduction

Carbon anodes are used in the aluminium industry for the electrolysis of alumina by transferring the electrical energy to the electrolytic bath, and providing the carbon necessary for the reaction. Prebaked anodes are made of petroleum coke aggregates, coal tar pitch and anode butts (recycled anode particles). The mix of these materials in certain proportions, called the anode paste, is formed in a mould and generally densified by vibro-compaction. The resulting block is called the green anode. The last step of anode fabrication is the baking in large furnaces, in which hundreds of anodes are baked for about two weeks. During all the above mentioned

stages of anode production, materials are characterized, from the raw material, such as coke, coal tar pitch and butts, to the anode paste, the green anode, and then the baked anode. The different analyses are performed to characterize the product quality, as the anode efficiency is related to its physical properties, such as density and porosity [1-3], reactivity (air [4, 5], CO<sub>2</sub> [6]), mechanical strength [7], and electrical resistivity [8]. In order to maintain consistent anode quality when facing changes in raw materials properties, adjustments to the anode paste formulation are implemented. This requires characterizing the raw materials as early as possible in the process. Coke particles are shipped to the plants by train and while unloading the shipment, coke particles flow from their container to specific storage bins in the plant. During the transfer of raw material from transport container to the bins, falling particles emit sound due to interaction with each other as well as falling on sieving grids. The sound emitted is expected to be an intrinsic characteristic of the material analysed. Acoustic chemometrics for fluid and solid media was used to perform either qualitative [9] or quantitative [10] measurement of flows. These methods allow a non-invasive and non-destructive analysis of the materials. Bruwer et al. [11], as well as Haltesen and Esbensen [12] performed sound analysis on materials such as particles having a wide size range, or snack foods in order to determine either the size distribution or the mechanical strength of the studied material, respectively. Based on these studies, it appears that using sound analysis would be a feasible approach to perform a preliminary characterization of coke particles conveyed to the anode plant. Samples could be collected at different locations in the green mill and submitted to sound analysis using the set-up described later in this paper, or emitted sound could potentially be collected directly from the line. The proposed approach could help estimate coke density and porosity before the anode formulation step, which could guide the operators in making feed forward adjustments when necessary. It is also believed that the method could detect changes in coke source, for instance due to a supplier change or when cokes from more than one source are stored in the same bin. The sound emitted by the particles may be used to distinguish coke sources, and estimate their proportions in the mix. To validate these assumptions, tests were performed on several cokes of similar size fractions that were previously investigated in detail [1]. Sound emission analysis of each source was carried out. Then, the cokes having the most different apparent densities were blended and their acoustic signatures were measured.

## **2. Material and Method**

### **2.1. Acoustic emission measurement**

To perform the measurements, the same cokes as those used by Azari et al. [1] were investigated. These correspond to cokes labelled A, C, D, and E in [1]. To facilitate the analysis and the comparison between both works, cokes designation is the same. To perform the sound analysis, the anechoic chamber shown in Figure 1 was built using stainless steel tubes with a square cross-section. The internal surfaces were coated with convoluted acoustic foam. The bottom of the box was placed on a low density Styrofoam plate. A two-inch hole was drilled through the top of the box to allow the particles to flow from the feeder to the reception plate. The set-up was placed on a table separated from the feeder to avoid vibrations coming from the feeder motor. To reduce even more the influence of surrounding sound, a small box was placed onto the arm of the feeder, above the feeding hole. The feeder consists of a flat arm, vibrating at a certain frequency controlled by a variable frequency electrical motor. The reception plate,

measured porosities. This difference is corroborated by the measurement of porosity found by Azari et al. [1].

As the results show, the PLS-DA analysis can discern between the clustering of individual sources and mixtures (both size wise on single source and source mixing of one size). The PLS analysis gave promising results indicated by the strong correlation between measured values and projections of density and porosity, respectively. It is thus possible to estimate the density and porosity of mixtures.

#### 4. Conclusions

The results of the study on acoustic emissions of coke particles to estimate their physical properties were presented in this article. After acoustic emission acquisition as well as PCA, PLS and PLS-DA modeling, the results show the clustering of different coke sources and sizes. Furthermore, PLS analysis shows that it is possible to predict density and porosity of different mixtures and sizes with good accuracy. The density model shows that the projected mixture data fall in between the values of the single coke sources that were mixed. Regarding porosity, results for coke A were found consistent with process knowledge, since porosity increases with particle size, and the porosity of mixtures of different sizes of coke A fall in between the single size samples that were blended. This trend, however, is not verified for coke D as shown in Figure 7. Further experimentation would be needed to explain better the phenomena and build a more robust model. In future work, a more sensitive and accurate model could be built by deconvoluting the acoustic signal and thus investigating single particles instead of a sample as a whole. Multivariate specification regions for each size fraction and source would be clearer.

#### References

1. K. Azari, H. Alamdari, D. Ziegler, M. Fafard, Influence of coke particle characteristics on the compaction properties of carbon paste material, *Powder Technology*, 257, 2014, pp 132-140.
2. B. Majidi, K. Azari, H. Alamdari, M. Fafard, D. Ziegler, Simulation of vibrated bulk density of anode-grade coke particles using discrete element method, *Powder Technology*, 2014, 261, pp 154-160.
3. K. Azari, W. Bogoya-Forero, C. Duchesne, J. Tessier, Measurement of vibrated bulk density of coke particle blends using image texture analysis, *JOM*, 69(9), 2017, pp 1613-1623.
4. R. Ishak, D. Picard, G. Laroche, D.P. Ziegler, H. Alamdari, Application of boron oxide as a protective surface treatment to decrease the air reactivity of carbon anodes, *Metals*, 7(3), 2017, 79.
5. F. Chevarin, R. Ishak, D. Ziegler, M. Fafard, H. Alamdari, Evolution of anode porosity under air oxidation: the unveiling of the active pore size, *Metals*, 7(3), 2017, 101.
6. F. Chevarin, K. Azari, L. Lemieux, D. P. Ziegler, M. Fafard, H. Alamdari, Active pore sizes during the CO<sub>2</sub> gasification of carbon anode at 960 C, *Fuel*, 178, 2016, pp 93-102.
7. S. Thibodeau, H. Alamdari, D.P. Ziegler, M. Fafard, New insight on the restructuring and breakage of particles during uniaxial confined compression tests on aggregates of petroleum coke, *Powder Technology*, 253, 2014, pp 757-768.
8. G. Rouget, B. Majidi, D. Picard, G. Gauvin, D. Ziegler, j. Mashreghi, H. Alamdari, Electrical resistivity measurement of petroleum coke powder by means of four-probe method, *Metallurgical and Materials Transactions B*, 2017 (published on-line, DOI: 10.1007/s11663-017-1022-9).

9. M.-J. Bruwer, J. F. MacGregor, W. M. Bourg Jr, Fusion of sensory and mechanical testing data to define measures of snack food texture, *Food Quality and Preference*, 18, 2007, pp 890–900.
10. K. H. Esbensen, B. Hope, T. T. Lied, M. Halstensen, T Gravermoen, K, Sundberg, Acoustic chemometrics for fluid flow quantifications—II: a small constriction will go a long way, *Journal of Chemometrics*, 13, 1999, pp 209–236 .
11. M.-J. Bruwer, J. F. MacGregor, W. M. Bourg Jr, Soft Sensor for snack food textural properties using on-line vibrational measurements, *Industrial & Engineering Chemistry Research*, 46, 2007, pp 864-870.
12. M. Halstensen, K. Esbensen, New developments in acoustic chemometric prediction of particle size distribution, the problem is the solution, *Journal of Chemometrics*, 14, 2000, pp 463–481.
13. Eigenvector Research, Inc., eigenvector.com
14. S. Wold, K. Esbensen, P. Geladi, Principal component analysis, *Chemometrics and Intelligent Laboratory Systems*, 2, 1987, pp 37-52.
15. S. Wold, M. Sjöström, L. Eriksson, PLS-Regression: a basic tool of chemometrics, *Chemometrics and Intelligent Laboratory Systems*, 58, 2001, pp 109-130.
16. P. Geladi, B.R. Kowalski, Partial least squares regression: a tutorial, *Analytica Chimica Acta*, 186, 1986, pp.1-17.
17. A. Höskuldsson, PLS regression methods, *Journal of Chemometrics*, 2, 1988, pp. 211-228.
18. M. Barker, W. Rayens, Partial least squares for discrimination, *Journal of Chemometrics*, 17, 2003, pp. 166-173.