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# Development of a soft sensor for detecting overpitched anodes: Detailed investigation of an anode sticking event

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**Abstract** Adjusting pitch ratio in green anode formulation is becoming difficult due to the increasing raw material variability. The optimal quantity of pitch yielding the best anode properties for a given aggregate, known as the optimal pitch demand (OPD), changes more frequently and is unknown *a priori*. Exceeding the OPD increases the risk of generating post-baking anode sticking events. Previously, the potential of a Principal Component Analysis (PCA)-based monitoring scheme for detecting the onset of these undesirable events was assessed by using a set of five green anode resistivity measurements collected from over 120,000 anodes produced over a two-year period. The Squared Prediction Error (SPE) was shown to be sensitive to abnormal events such as anode sticking. The objective of this paper is to further validate the soft sensor by studying the SPE dynamic behaviour during a post-baking sticking

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event when changes in the anode paste formulation were introduced as part of normal operation. Descriptive and statistical analyses demonstrate that the SPE metric reacts significantly to changes in the recipe. The provided example illustrates how the SPE metric used together with pitch ratio data could help advised the operators of manufacturing conditions posing a higher risk of generating post-baking sticking problems.

**Keywords** Anode sticking · Principal Component Analysis · Squared Prediction Error · Green anode formulation · Resistivity measurement · Optimum pitch demand

## 1 Introduction

The pre-baked carbon anode is a fundamental component in aluminum electrolysis by the Hall-Héroult process for two main reasons: it provides the carbon source for the electrolytic reaction and allow the electrical current to flow through the cells. The quality of carbon anodes is known to influence cell performance and energy consumption [1,2]. To maximize process efficiency and to ensure optimal production, individual anode quality control is therefore essential.

The increase of the world demand for calcined coke and coal tar pitch, the two main raw materials used in anode production, leads to a decrease in their quality while their purchasing costs are increasing [3]. To ensure process profitability, the industry needs to buy materials from multiple suppliers. As the raw material quality influences anode properties [4,5], this increase in raw material variability is a major problem for the industry.

To maintain a certain level of quality, the pitch content in green anodes needs to be adjusted carefully to mitigate the more frequent variations in coke properties, such as its porosity. Maintaining pitch content near the so-called optimum pitch demand (OPD) is important for producing consistent quality anodes. Some studies suggest that for a given dry aggregate mix, the OPD is reached when the baked anode density (BAD) is maximal [4,6,7]. Drift from this optimum may lead to different problems. Under-pitched anodes have low mechanical properties [8], high permeability [9] and resistivity as well as low density [10]. In the case of over-pitched anodes, extreme shrinkage, stub hole deformation, cracks caused by degassing of pitch volatiles, sticking of packing material [10,11], and increased air reactivity caused by impurities are known issues.

Anode sticking during baking may also occur [12,13]. Such an abnormal event requires additional operator interventions during furnace unloading because stuck anodes cannot be placed directly on conveyors. A mechanical separator is needed to release the stuck anodes which increases scrapped material and losses and corresponding extra energy consumption and GHG emissions. As there is no online measurement indicating in real time whether anodes are produced at their optimum pitch content or are under- or over-pitched, post-baking sticking problems can currently only be detected by the operators after baking during furnace unloading.

Various methods have been discussed in the literature to estimate the ODP. Mathematical models based on raw material properties have been developed [7,14,15]. As their properties are not known frequently, these techniques are not suitable for a

rapid optimum identification. Laboratory determination of the OPD is time consuming and the size of the equipment used to produce small scale anodes does not allow formulating dry aggregate mixes that match those used at industrial scale [16]. Dynamic optimization was also proposed to identify the pitch ratio maximizing baked anode density but this is time consuming [13]. A similar approach is periodically used by operators after raw material or recipe changes are implemented, but they rely on visual inspection and dimensions of the anodes, because baked anode density is unavailable [17]. Simplified models are also used. The main goal of all these techniques is to find or to be as close as possible to the optimum, but not directly to prevent post-baking sticking problems before baking.

As an alternative, Lauzon-Gauthier et al. [17] proposed a vision-based sensor to predict deviations from the OPD using paste image texture. Anode paste images were collected from a lab scale anode manufacturing setup for different formulations. Textural features were extracted from these images using discrete wavelet transform (DWT) and gray-level co-occurrence matrix (GLCM). A Partial Least Squares (PLS) regression model was then built for predicting deviations from optimal BAD based on paste image textural features. The vision sensor was later tested using industrial samples collected from an industrial plant to determine its sensitivity and robustness. This method is interesting, but hurdles associated with collecting high quality paste images in a harsh environment limits its application.

In a recent preliminary study [12], the authors assessed the potential of using green anode resistivity measurements for detecting the onset of post-baking anode sticking events. A Principal Component Analysis (PCA) model was built on electrical resistivity data collected from about 120 000 industrial green anodes produced over a two-year period, thus fabricated using different raw materials. It was shown that the Squared Prediction Error (SPE) statistic calculated from PCA was higher during known sticking events. The SPE indicates that a break in the correlation structure between resistivity measurements occurs before the abnormal events are confirmed when the baking furnaces were unloaded. Hence, the SPE could provide an early warning of baked anode sticking problems. However, in this preliminary study, the impact of changes in green anode formulation on the SPE were not considered nor its statistical assessment.

To improve monitoring and optimization of the process in terms of maintaining the green anodes close to their OPD, it is important to understand the dynamic relationships between anode formulation and SPE, the latter being used as a proxy for deviations from OPD. The objective of this paper is to validate the PCA-based soft-sensor by comparing trends in the SPE against changes made in the anode paste formulation as part of normal operation, and interpreting the impact that these changes might have had on the OPD during a sticking event based on process knowledge. The goal is first to show that when changes in the formulation likely made the paste overpitched, an increase in SPE is observed once passed the transportation delay. Then, statistical tests are used to determine whether changes in the recipe formulation have significant effect in the SPE during a sticking event.

The paper presents first the anode manufacturing process and the collected data. Then, a brief description of the latent variable methods and the soft sensor develop-

ment is provided. Thereafter, the results are analyzed through a discussion and the main conclusions are drawn.

## 2 Anode Manufacturing Process and Data Collection

Data used in this work were collected at Alcoa's Deschambault smelter in Quebec, Canada (ADQ). This section presents a brief overview of the pre-baked carbon anode plant, a description of the apparatus used to measure green anode resistivity, the procedure used to retrieve past anode sticking events and finally how the anode manufacturing data were synchronized.

### 2.1 Pre-baked Anode Manufacturing Process

Figure 1 shows the pre-baked anode manufacturing process as well as the main time delays between the units and key instruments. This information was essential for data synchronization. The fresh coke particles are divided in three size classes : coarse (C), intermediate (I) and fines (F). Recycled materials are also used in the process. Baked recycled particles (BR) are composed of crushed anode butts and defective baked anode unused in the electrolysis cells. Green recycle material (GR) consists of the anode paste obtained from rejected green anodes that were deemed out of specifications for various reasons. According to the specified formulation, coke particles from the different size classes and baked recycled materials are portioned out, mixed and preheated to obtain the desired dry aggregate mix. Liquefied coal tar pitch is then added to the aggregate mix. The resulting paste material is blended in two consecutive mixers before forming through vacuum vibrocompaction. To achieve the desired production rate, the process line is split in two. This allows an alternate use of the two vibrocompactors.

For tracking anodes during their service life, each anode is stamped with a serial number just after forming by a vibrocompactor. A first camera reads the anode number prior to the cooling process to ensure tracking of the anode because immersion time in the water bath is variable. However, in normal operations as it is the case in this study residence time in the water bath fairly constant. When the anode leaves the bath, a second camera reads the anode number in order to link it with anode resistivity measurements collected by using a four-point probe device (4PP). Finally, the anodes are stored prior to baking.

### 2.2 Anode Sticking Events

To analyze the post-baking sticking events, it is essential to retrieve the moment when they occurred. At the plant, occurrence of sticking is registered in a database on a daily basis when the event was deemed important by the operators. However, no indication as to which individual anodes were involved is available. Based on the comments left by the operators in the database, a one-day accuracy backtracking

method is used to assign labels to indicate if the green anode has stuck or not during baking.

Starting with comments left on a given day which, in turn, is associated with an unloading date, the furnace number and the chamber associated with the sticking anode event can be retrieved. With this information, the loading information containing the date and the number of the first and last anodes loaded in this given chamber can be found. Using those and the number read by the 4PP camera, the fabrication day is retrieved. Finally, all the anodes that were produced during this specific day were assigned the label "sticking".

As a high amount of scrapped anode and an important number of anode separator strikes also provide quantitative evidence indicating that a sticking event occurred in absence of operator's comment, an analysis of these variables was performed to retrieve missed or minor events on a weekly basis. When the two parameters are high, all the anodes produced in these weeks were also assigned to the "sticking" class.

To facilitate the distinction between the two types of anodes, the following acronyms are used in the following section. Post-Baking Sticking (PBS) anodes were assigned a sticking event label either by the backtracking method or the strike/scrap analysis. Other anodes are considered asymptomatic and are labelled as Non-Sticking (NS) anodes.

It is important to note that assigning the anodes to a class (PBS or NS) requires information from the anode baking step. However, this is required only for the soft-sensor model building phase and not for its on-line application to detect the onset of sticking events. For the latter, only the resistivity measurements collected on the green anodes by the 4PP device are used. Thus, a decision to apply a corrective action can be made rapidly, before baking the anodes.

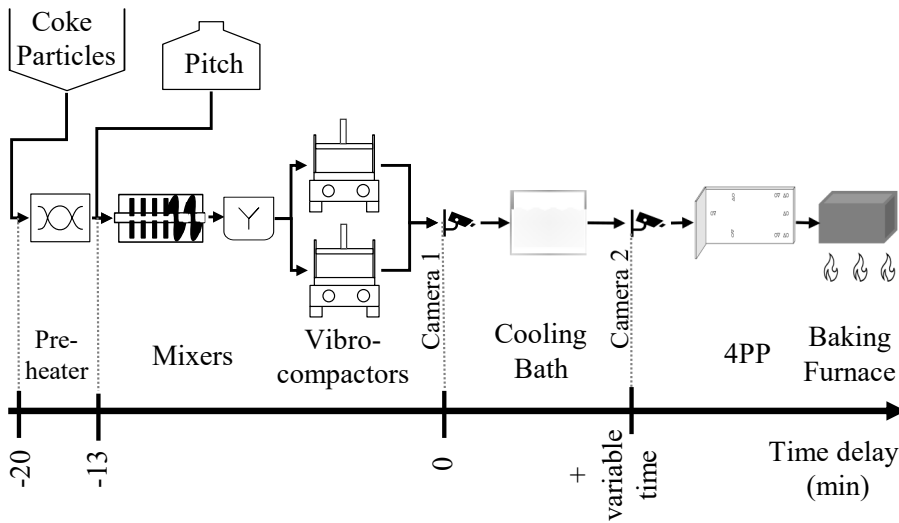
### 2.3 The 4PP device

The 4PP apparatus was patented by Ziegler and Secasan [18]. This non-destructive technique used the linear four-points probe method, which is based on the hypothesis that green anodes follow the Ohm's law [18]. The device is installed on the manufacturing line and allows measuring the green anode resistivity automatically at five different positions as shown in Figure 2.

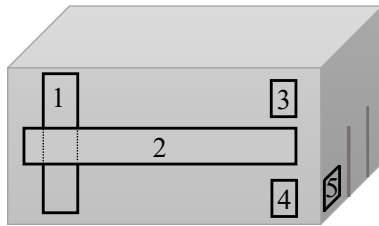
The first four measurements are collected on the long side of the anode while the last one is on the short side. The positions labeled 1, 3 and 4 correspond to resistivity measurements collected vertically, and those labeled 2 and 5 are obtained horizontally. The 4PP measurement principle consists of injecting a known electrical current  $I$  into the anode and to measure the resulting voltage drop  $V$  between two probes (located at the ends of the rectangles shown in Figure 2) sequentially for each labeled position in Figure 2. The resistivity  $\rho$  is then calculated using Ohm's law:

$$\rho = \frac{V}{I} \Phi \quad (1)$$

where  $\Phi$  is a shape factor. In order to ensure the reliability of the data, faulty measurements are flagged and registered in the database when the injected current and



**Fig. 1** Anode manufacturing process at ADQ including the main equipments, the position of the two cameras reading the anode numbers, and the delay between each unit in minutes



**Fig. 2** 4PP measurement position on the green anode

measured voltage drop fall outside predetermined ranges. These faults are usually equipment related (e.g. current is too low or poor probe-anode contact). The binary fault indices, the resistivity measurements and the anode number read by the second camera are stored in a data matrix for further analysis.

For more information about electrical conductivity tests and measurement uncertainties, the interested reader is referred to Ziegler and Secasan [18], and well as Lauzon-Gauthier and Secasan [19]. The latter also illustrate how the 4PP technology is used in pitch optimization experiments.

## 2.4 Anode manufacturing data collection

In order to relate green anode resistivity measurements to change in the anode paste formulation, the proportions of coke particles in the different size classes, recycled materials and pitch in the paste need to be fused with resistivity data. To synchronize these data, the first step is to find the moment when the anode was formed. As presented in Figure 1, a variable delay exists between anode forming and the resistivity measurements. The goal is to retrieve this variable delay by using the two cameras installed on the process. Thus, for each number read by the 4PP camera (i.e. at the outlet of the water bath), the algorithm seeks the same number read by the first camera located at the outlet of the vibrocompactor. If a match is found, the date of the first camera reading is considered to be the fabrication date of this green anode. Otherwise, in the absence of a match, the formulation cannot be linked with the resistivity measurement. Then, for each anode that have receive a fabrication date, the synchronization procedure proposed by Lauzon-Gauthier [20] based on delays indicated in Figure 1 is used. The ratio of each coke particle size fraction and that of pitch are extracted from the database using a time weighted average on a period of one minute where the center of the interval is the time delay. Averaging was used to represent the best possible the materials accumulated in the vibro-compactor mold to form one anode.

## 3 Soft Sensor Development

Since the five resistivity measurements collected from the anodes are strongly correlated, Principal Component Analysis (PCA), a latent variable method, was selected for the analysis of the large and highly collinear dataset, and to develop the post-baking sticking monitoring scheme. First, a description of PCA is presented in this section. Then, a summary of the soft sensor development proposed in [12] is provided.

### 3.1 Principal Component Analysis

The main goal of PCA is to maximize the variance-covariance explained between the variables while reducing the dimensionality of the dataset [21]. The high-dimensional data matrix  $\mathbf{X}$  containing  $N$  observations (rows) and  $K$  variables (columns) is projected on a lower dimensional subspace formed by a set of  $A$  orthonormal loading vectors  $\mathbf{p}$  ( $K \times 1$ ) to obtain the scores vector  $\mathbf{t}$  ( $N \times 1$ ) summarizing the information extracted from the dataset. For each of the  $A$  components, the resulting vectors are stored in the corresponding matrix  $\mathbf{T}$  ( $N \times A$ ) and  $\mathbf{P}$  ( $K \times A$ ). PCA decomposition is mathematically expressed using the following equation:

$$\mathbf{X} = \mathbf{TP}' + \mathbf{E} \quad (2)$$

where  $\mathbf{E}$  contains the model residuals.

The extraction of the loading vector is performed by using the Nonlinear Iterative Partial Least Squares (NIPALS) algorithm or the eigenvalue decomposition of the variance-covariance matrix ( $\mathbf{X}'\mathbf{X}$ ) where  $\mathbf{X}$  was first centered and scaled. For the following study, the NIPALS algorithm is prioritized since each component is extracted one at the time which is advantageous when few of them are considered. More information on this algorithm is available in [22].

The score vectors are orthogonal, and are defined as linear combination of the X-variables where the loading vectors contain the weights of each variables. Hence, the score vectors are obtained by the following equation:

$$\mathbf{t}_a = \mathbf{X}\mathbf{p}_a \quad (3)$$

for the  $a^{th}$  component.

The number of components retained  $A$  should be carefully chosen in order to model only the structured information and exclude noise. Standard procedures to select the number of components when a model is built for prediction are to use either cross-validation or validation based on an external dataset to calculate the cumulative predicted variance (i.e.  $Q^2$  statistic) on a global basis and the root mean squared errors of prediction (RMSEP) for each variable. As long as adding a new component leads to an increase in  $Q^2$  and/or a decrease in the RMSEP, this component is kept in the PCA model. Otherwise, the procedure stops. If the model is used to make classification, the number of components can be chosen using the classification performances. A component should be retained only if the performances are improved. In this study, the number of PCA components was selected to maximize classification performance.

When the loadings and the scores are determined, the residual should be analyzed. One useful metric is the Squared Prediction Error (SPE) which allows detecting changes in the correlation structure between the variables. In fact, it corresponds to the distance between an observation and its orthogonal projection on the PCA subspace. First, for each observation, the prediction error  $\mathbf{e}_i$  is computed as follows:

$$\mathbf{e}_i = \mathbf{x}_i - \hat{\mathbf{x}}_i = \mathbf{x}_i - \sum_{a=1}^A t_{a,i}\mathbf{p}_a \quad (4)$$

where  $\hat{\mathbf{x}}_i$  is the predicted value of the  $i^{th}$  observation (i.e., projection on the subspace). Then, the SPE is calculated using the following equation:

$$SPE_i = \mathbf{e}_i\mathbf{e}_i^T \quad (5)$$

To detect inconsistent data, an upper confidence limit (UCL) on the SPE metric must be defined. It is known that the SPE values are following approximately a chi-squared distribution [23]. The  $(1-\alpha)$  SPE upper control limit is obtained using the mean and the variance of the SPE based on the calibration dataset, respectively  $m$  and  $v$  in this equation:

$$SPE_{UCL} = \left(\frac{v}{2m}\right) \chi_{\left(\frac{2m^2}{v}, \alpha\right)}^2 \quad (6)$$

where  $\chi_{\left(\frac{2m^2}{v}, \alpha\right)}^2$  is the value of chi-squared distribution for  $2\frac{m^2}{v}$  degrees of freedom and  $\alpha$  confidence level.



### 3.2 PCA-based Monitoring Scheme

Since the work presented in this paper builds on the preliminary study performed by Paris and al. [12], this section summarizes the main step used to built the PCA soft-sensor.

After extracting the five resistivity measurements and the corresponding fault indices =defined in section 2.3, all anodes that have received at least one current or voltage fault were removed since they indicate an improper measurement. In addition, anode having more than one missing resistivity due to an unavailable measurement from one of the 4PP sensor were not considered in further analyses. Following this cleaning step, each set of five resistivity measurements received an anode number by using the 4PP camera reading. This association is necessary mainly to determine the vibrocompactor number. If the synchronization fails for some anodes, they are removed from the dataset. A second cleaning step that consists of removing the data exceeding six-sigma limits is executed for all the synchronized data. The measurements collected from the 120,313 remaining green anodes are stored in matrix  $\mathbf{X}$  where the columns correspond to the resistivity sensors and the rows to the observations. The class assigned to each anode (NS or PBS) are stored in a column vector.

As two known changes not related with sticking issues, namely the impact of the 4PP maintenance and small differences between the two vibrocompactors, were causing systematic shifts in the raw data, the normalization procedure proposed in [24, 12] was applied to remove them. The resulting data free of known biases are then stored in the  $\mathbf{X}_p$  matrix.

To avoid overfitting of the PCA soft-sensor model, the  $\mathbf{X}_p$  matrix was divided in three matrices. The first,  $\mathbf{X}_T$ , is used to train the model while  $\mathbf{X}_{V1}$  and  $\mathbf{X}_{V2}$  served to validate it. As shown in Table 1,  $\mathbf{X}_T$  and  $\mathbf{X}_{V1}$  contain only NS anode. The first data set was obtained by randomly selecting 50% of the days where no sticking problems has occurred. All anodes assigned to the PBS class were collected in the third dataset ( $\mathbf{X}_{V2}$ ).

The mean and the standard deviation of  $\mathbf{X}_T$  was used to mean center and autoscale the data prior to building the PCA model. Note that both validation data matrices were also centered and scaled using the same way as for the training set. After analyzing the classification performance, one component was retained, because subsequent one did not improve detection rate. Using the loadings obtained for each resistivity:

$$\mathbf{p}_1 = [0.45 \ 0.50 \ 0.42 \ 0.45 \ 0.40] \quad (7)$$

the scores and squared prediction errors were calculated for all the observations contained in the three  $\mathbf{X}$  matrices. The upper control limit was set at 6.31 using the SPE associated to the  $\mathbf{X}_T$  matrix. The predicted class for each anode was obtained by comparing their SPE values with the statistical limit; anodes for which the SPE value falls below the limit are assigned to the NS class whereas those violating the limit (i.e. those for which the SPE value greater than 6.31) are assigned to the PBS class. This predicted class is in fact the soft sensor output.

**Table 1** Partitioning of the data in three datasets

Dataset	$X_T$	$X_{V1}$	$X_{V2}$
Anode type	NS	NS	PBS
Number of observations	54,473	41,300	24,540

## 4 Results and discussion

This section focuses on the impact of changes made to the paste formulation on the SPE values. In particular, the goal is to demonstrate that the SPE values are high during a sticking event caused by a pitch ratio exceeding the optimum pitch demand of the dry aggregate. Prior to interpreting the relationships between the trends in SPE and changes in the formulation, the principal actions performed by the operators to minimize known perturbations are stated using the observation made from the dataset provide by the ADQ plant.

To maintain a stable production and compensate changes associated with baked recycled particles, calcined coke is used in replacement. This leads to a change in the OPD because coke particles are more porous than baked recycled particles. To stay as close as possible to the ODP, pitch ratio is typically increased when the proportion of baked particles in the recipe is reduced.

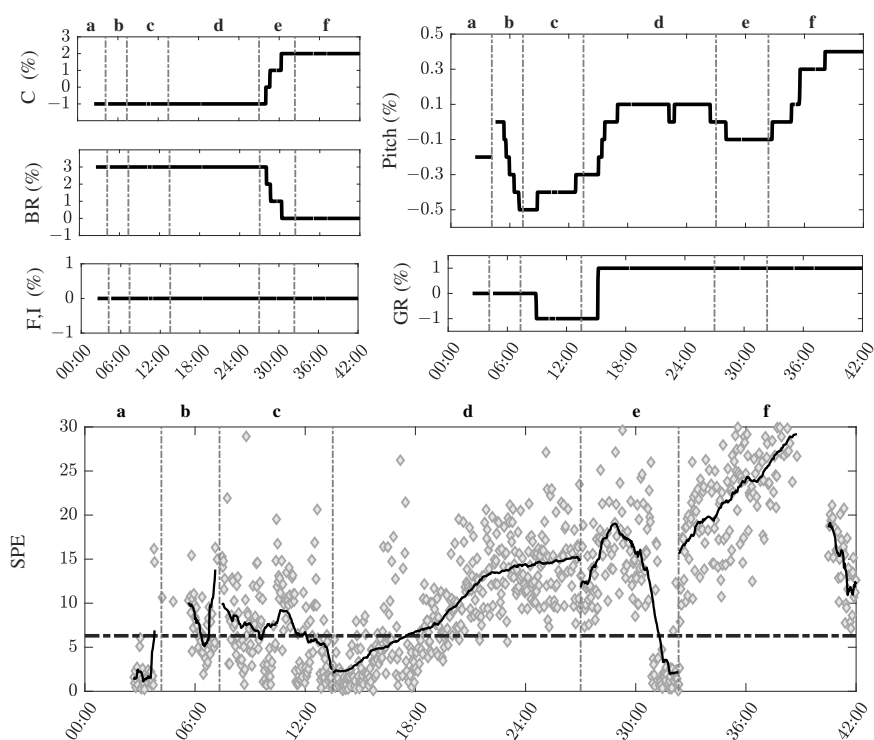
Changes in the quantity of green recycled are not compensated by another particulate component. As green recycled particles already contain uncarbonised pitch in their pores, reducing their proportion in the recipe may require a small increase in pitch ratio, sometimes no action is needed.

To illustrate these practices, an example using operation data collected during a sticking event is presented and interpreted. A descriptive analysis is performed followed by a statistical analysis. It is worth mentioning that time series were scaled to protect confidential information.

### 4.1 Descriptive Approach

In this section, recipe adjustments and trends in SPE are analyzed jointly during known sticking events. The time series shown in Figure 3 are split into 6 time periods (labeled a to f) within which different changes in the formulation were implemented. For the SPE, in addition to showing its values, a moving average is plotted using a solid black line to facilitate the interpretation of the relationships between the trends in SPE and the changes made to the recipe. The size of the sliding window used depends on the numbers of data points contained in the time interval. The criteria to determinate the number of data to calculate the average is to use a ratio of 25% of the number of data points in the interval. The 95% confidence limit on SPE is also indicated using a black dashed line. To simplify the figure's legend, the acronyms introduced in the Section 3 are used to identify the different materials (particle size fractions and pitch).

Initially, each ratio is held constant. The SPE values are below the confidence limit and vary from 0.2 and 2.7. A first adjustment was made to pitch ratio at the



**Fig. 3** Impact of anode paste recipe adjustments (top) on SPE values (bottom) during a post-baking sticking event

end of interval (a) and the beginning of interval (b). As the resistivity measurements were not available at the beginning of interval (b), the SPE values are missing. For the pitch ratio, a synchronisation problem explains why the modification are not displayed. However, the subsequent change made at around 6:00 can be interpreted since the data are available. While the dry aggregate composition remains unchanged, pitch ratio was progressively reduced until the smallest ratio for this event was reached. The impact of the first three steps seems to cause a decrease in the SPE trends which falls below the confidence limit. SPE values then increase for the two last step changes in pitch ratio. The reason behind this behaviour is difficult to explain using the formulation data. As the SPE metric is not specific only to one problem (i.e. sticking events), it may indicate the presence of other variations in the process leading to a change in the correlation structure of the resistivity measurements.

The green recycle ratio is reduced in interval (c) which is compensated by increasing the pitch ratio. This action results in a slight decrease in the SPE until it drops below the confidence limit. Based on process knowledge, the three step changes in pitch ratio performed during the interval should have lead to an increase in SPE. However, the SPE signal decreases. This might be a sign that the adjustment made by the operators to compensate change in green recycled ratio was appropriate. Also, at around 8:30 corresponding of the middle of interval (c), the moving average show

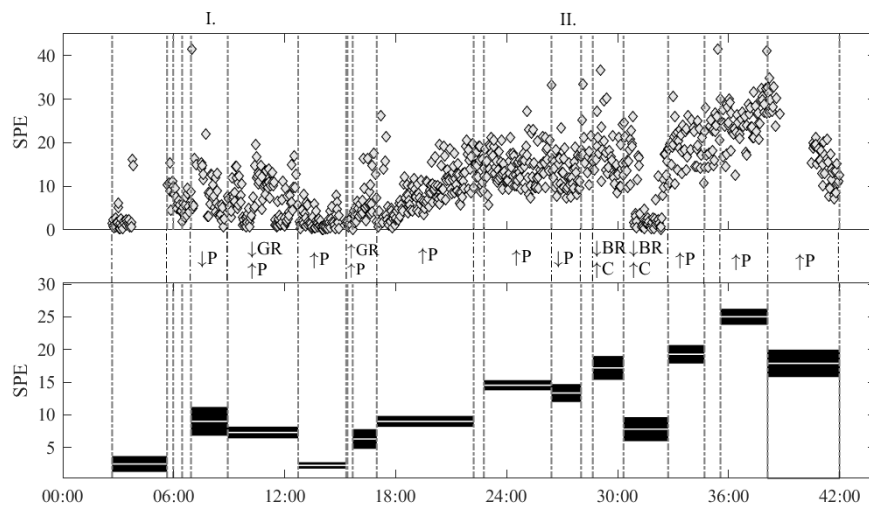
a slight increase in SPE values even if all ratios are held constant. This suggests that other factors may have an influence on the SPE behavior or it is the result of an unknown perturbation. Based solely on the anode recipe, the root-cause cannot be determined. The main hypothesis used in the present analysis is that each size class of coke particles (coarse, intermediate and fines) has a constant mean size. However, the fractions themselves are distributions of sizes. Changes in these distributions may have an influence on OPD and the resulting SPE values.

At the beginning of period (d), the pitch ratio set by the operator seems to be appropriate for the dry aggregate mix because the SPE values are below the confidence limit. Minutes later, the green recycle ratio is increased. Simultaneously, the pitch ratio is also increased drastically. In response to those adjustments, the SPE values start rising sharply and violate the confidence limit. After this important modification, the moving average of the SPE plateaus but the variance of the metric is high. Twice during this period, the pitch ratio is decreased by one increment (0.1 to 0) and held at the new ratio for approximately 30 minutes. The impact of these corrective adjustment is not observable in the SPE because of the high variance and there were implemented for a too short period of time.

The next interval (e) shows another important effect of formulation in SPE values. At the beginning of the period, the proportion of coarse particles is increased by 3% to compensate the equally important reduction in the baked recycle fraction. The adjustment is performed in three consecutive steps of 1% to ensure the stability of process operation because large steps are not recommended. For the first step change in coarse ratio, the pitch content is also decreased by one increment, but remains constant for the rest of the interval. The combination of all these changes results in an important decrease in the SPE values at the end of the time segment. At this moment, the SPE signal drops below the confidence limit and the behavior is similar to the one observed in time interval (a). As the paste obtained is composed of a more porous dry aggregate for the same pitch ratio, the risk of sticking problems is therefore reduced.

Segment (e) also shows that changes in formulation do not affect SPE instantaneously, but only after a time delay (transportation time through the process units). The first step in the coarse aggregate does not result in an immediate decrease in SPE. As represented by the moving average, SPE values increase even if the paste contains less pitch than before in a more porous aggregate. It is only during the second adjustment that the SPE signal starts decreasing. The short period associated with the first step change allows the production of fewer than thirty anodes. It may not be sufficiently long to observe the effect on the resistivity measurements. Another possibility would be data synchronization uncertainties. It may introduce errors since the time delays are based on an approximation of the residence time in each equipment. The presence of a residual dynamic, possibly caused by fluctuation in some process temperatures, may also be in cause, but further analysis would be necessary to validate this hypothesis.

The last interval (f) analyzed in the sticking event shows a corrective action implemented by operators based on their process knowledge. To compensate for the known decrease in the porosity of the dry aggregate caused by the increased proportion of the coarse coke fraction made in interval (e), pitch ratio is increased to the highest level used during the event. The resulting SPE values rise almost instantly



**Fig. 4** SPE means represented by a white line and their respective 95% confidence interval shown by the black area for each process operating point (i.e change in recipe) calculated for the anode sticking event studied previously

to reach 30, which is above the mean observed in interval (d). This behavior is quite different compared to the other changes of pitch ratio applied during the test where an increase of 0.1% is not sufficiently large to observe a direct change on the SPE trend. For this given dry aggregate mix that contains more porous coke particles, the adjustment made in pitch ratio seems to be more effective on the SPE trend.

Finally, a decreasing trend in SPE is observed at the end of the interval. As no change occurs, a variation in the grinding and classification process or an adjustment in a process temperature may introduced disturbances that could explain the trend. Further studies are required to identify the main factor influencing the SPE values other than the proportions of the various components in the formulation, and establish cause-and-effect relationships.

In general, this example illustrates that the proportion of coarse coke particles, baked recycle aggregate and pitch ratio have an impact on SPE values. Changes in the ratio of coarse particles seems to have the greatest impact on SPE.

However, it is difficult to compare which modification has the greatest impact since the size of the step for the pitch ratio is ten times lower than the one for the coke, respectively 0.1% compared to 1%. Sometimes, a step of 0.1% in pitch seems to be insufficient to observe a real difference in the SPE values because of the high level of noise. Knowing the steady-state gain between each type of change in the formulation and the SPE, through performing designed experiments, would help identify which parameters have the strongest impact.

This case study provides a good example of how the proposed soft sensor would have been beneficial for adjusting the pitch ratio to minimize the risk of sticking.

Twice during the presented sticking event pitch ratio should have been held constant rather than being drastically increased because the SPE values were already below the confidence limit prior to the change. It is the case at the beginning of the interval (b) and when the coarse ratio was increased around time 30:00 (interval e). Had the soft-sensor been implemented before this event would have advised the operator not to overreact on pitch ratio to minimize the risk of getting into new sticking events.

Finally, it is important to note that even if the proposed approach will not allow avoiding all sticking events, it can potentially help reducing the number of sticking anodes and the duration of sticking events. In this study, the transport delay between a change in pitch ratio and the readings by the 4PP device is worth about 2 hours of production, whereas documented sticking events in the available historical data last at least a day, and sometimes up to several days in the worst cases.

## 4.2 Statistical Approach

As the previous analysis was based only on a visual approach, the conclusions drawn also need to be statistically validated. This is achieved by computing the mean of the SPE for each combination of operating conditions as well as its confidence interval. It allows to assess whether changes in the SPE are statistically significant.

Initially, some hypothesis are stated. For statistical inference purposes, it is typically assumed that the SPE statistic is an independently and identically distributed random variable following an approximate Chi-Squared distribution [23]. Since the mean corresponds to the sum of random variables divided by a constant, the central limit theorem can be invoked to determine the distribution of the mean of SPE values. It states that the sum of  $n$  random variables sampled from any distribution approximately follows a normal distribution when  $n$  tends toward infinity [25]. Hence, a computational study was performed to determine the number of data points  $n$  (i.e. number of anodes) required to make the mean of SPE values approximately normally distributed. At least 30 anodes produced under normal conditions and with the same raw material should be used to meet these conditions [24]. Therefore, in the remainder of this section, the mean of SPE values calculated based on 30 anodes or more is assumed to follow a normal distribution, and the confidence interval on the mean is calculated using the Student-t distribution.

The results are provided in Figure 4. The graph at the top of the figure shows the same SPE signal as presented in Figure 3, but the time intervals are divided differently. Each step change performed to adjust the recipe is considered a new operating point (i.e. set of process conditions). To simplify the interpretation of the figure the acronyms described in Section 3 are used with an arrow to indicate the sign of the variation for each operating point. The smallest step changes on pitch ratio is 0.1% and 1% for the different fractions of particles. New operating points obtained by changing the recipe, but maintained for the production of less than 30 anodes were not displayed in Figure 4. The graph at the bottom shows the SPE means in white and the confidence intervals in black. It should be noted that the thickness of the confidence interval is varying, because they are calculated using the number of samples and the standard deviation of each process operating point.

The method used to analyze the figure consists of comparing two consecutive means of SPE values to determine if they are significantly different. This is achieved by comparing their confidence intervals to determine the degree to which they overlap. No overlap provides a clear indication that changes in the process had a statistically significant impact on SPE. If they do overlap, this might indicate that the implemented change results in smaller or no impact on the SPE values. For almost the totality of the change performed, the confidence limit does not overlap except for the two time periods labeled as I and II at the top of the figure. This demonstrates that recipe adjustments have a significant impact on the SPE values calculated by the soft sensor.

A more detailed statistical analysis is performed for the periods I and II by using a Student's t-test for unequal sample sizes and variances. For the adjustment of pitch and green recycle ratio identified by the interval I, the p-value of t-test is 0.16. In the case of the period II, which represented the impact of a change of 0.1% in the pitch ratio, the calculated p-value is 0.14. Based on a bilateral student test at 95%, as the p-value is higher than 0.05, the null hypothesis stipulating that the means are equal cannot be rejected. These two changes in the recipe do not have a statistically significant impact on SPE. If the ratio had been maintained longer, the size of the interval would have been smaller, and the change might have been statistically different for these two cases. The plot would have shown confidence interval overlap for these two cases.

The analysis of the confidence limits highlights that most adjustments made on the recipe during the sticking event have had a statistically significant impact on the output of the soft-sensor. The SPE trends observed in the descriptive approach section are the same for operating points held a sufficiently long time period to produce at least 30 anodes. No comparison is possible for the two drastic modifications of the pitch ratio the confidence intervals cannot be calculated because of the non-compliance to the central limit theorem. As each change has an influence on the SPE, the soft sensor may be useful to provide guidance to the operators on how to adjust the recipe to avoid sticking events before baked the anodes. This will therefore minimize the risk of producing a large amount of sticking anode that result in a decrease of scrap material and reduce the economic losses.

## 5 Conclusions

The increase in raw material variability and changes in dry aggregate properties are known to influence the optimal pitch demand. It results in more frequent adjustments of pitch ratio as well as an increase of the risk of post-baking anode sticking problems. Previously, Paris et al. [12] have proposed a soft sensor for detecting sticking issues prior to baking by using green anode resistivity measurements. It was shown that a break in the correlation structure between the resistivity measurements detected by the SPE statistics computed from a PCA model seems to occur when anode sticking problems are observed, but the dynamic behavior of the SPE metric during these undesirable events was not assessed. Using data from a past post-baking event, the

objective of this article was to show that the SPE metric reacts to changes introduced in the anode paste recipe as part of normal operating procedures.

An analysis of the SPE behavior during a post-baking sticking event was provided in this article to determine the influence of recipe adjustments on SPE. The statistical analysis demonstrated that all change applied to the dry aggregate mix and pitch ratio have a significant influence of the average of SPE values computed over periods of fixed operating conditions. The coarse coke and anode baked recycled particles lead to important variation in the SPE trend. Changes in pitch content also needs to be considered. Even when the variations in pitch ratio are small, a significant effect is observed on SPE values. When the amount of pitch is expected to be above the OPD for a dry aggregate mix, a rise of the SPE values is observed. This make sense since an increase of pitch may lead to overpitched anodes which have a higher risk of causing post-baking sticking issues. The descriptive analysis provided an example of how this soft sensor may have been beneficial for detecting the onset of post-baking sticking anodes and prompt operator's attention to make a corrective action to mitigate the situation.

Since it currently takes about 14 days to determine if the anodes have stuck in the baking furnace, using the soft-sensor may inform on the risk of sticking issue few hours after the anode formulation, which is mainly the delay between the vibrocompaction and the measurement of the resistivity. As the SPE, the output of the soft-sensor, is related with the recipe modification, using these data together may help operators adjust the pitch ratio. This indicator would be beneficial to prevent the risk of sticking problems. Future work will look at implementing the monitoring scheme in a plant to refine the model and limits and further validate the approach by using online data with a set of planned tests on the industrial production line.

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### Conflict of Interest Statement

On behalf of all authors, the corresponding author states that there is no conflict of interest.

### References

1. K. Grjotheim and H. Kvande: *Introduction to aluminium electrolysis: understanding the Hall-Héroult process*, 2nd ed., Alu Media, Düsseldorf, 2010, 260 pp.
2. A. Charette, Y. S. Kocaeffe and D. Kocaeffe: *Le carbone dans l'industrie de l'aluminium*, Les presses de l'aluminium, Saguenay, Canada, 2012, 320 pp.
3. L. Edwards, N. Backhouse, H. Darmstadt and M.-J. Dion, C. E. Suarez, ed.: *Light Metals 2012*, The Metallurgical Society of AIME, New York, NY, 2012, pp. 1207–12.
4. D. Belitskus and D. J. Danka: *JOM*, 1988, vol. 40 11, pp. 28–29.
5. U. Mannweiler: *Anodes for the aluminum Industry*, R & D Carbon Ltd, Sierre, Switzerland, 1995, pp. 197–202.



6. D. Belitskus: *Metall. Trans. B*, 1981, vol. 12 1, pp. 135–39.
7. A. L. Proulx, S. K. Das, ed.: *Light Metals 1993*, The Metallurgical Society of AIME, New York, NY, 1993, pp. 657–61.
8. E. Selding: *Proceeding of the First and Second Conference on Carbon at the Univ. of Buffalo*, The Waverly Book Company Ltd., 1956, pp. 217–22.
9. W. K. Fischer and R. Perruchoud: *Anodes for the aluminum Industry*, R & D Carbon Ltd, Sierre, Switzerland, 1995, pp. 141–48.
10. U. Mannweiler and F. Keller: *JOM*, 1994, vol. 46 2, pp. 15–21.
11. K. Hulse: *Anode manufacture : raw materials, formulation and processing parameters*, R & D Carbon Ltd, Sierre, Switzerland, 2000, 416 pp.
12. A. Paris, C. Duchesne, É. Poulin and J. Lauzon-Gauthier, A. Tomsett, ed.: *Light Metals 2020*, Springer International Publishing, Cham, 2020, pp. 1176–82.
13. C. Vanvoren: Procédé de réglage de la teneur en brai des anodes destinées à la production d'aluminium par électrolyse, patent: EP 0252859 B1, 1987.
14. M. Benton, C. Bickert, ed.: *Light Metals 1990*, The Metallurgical Society of AIME, New York, NY, 1990, pp. 657–59.
15. L. Castonguay and P. J. Rhedey, D. W. MacMillan, ed.: *Proceedings of the International Symposium on Quality and Process Control in the Reduction and Casting of Aluminum and Other Light Metals, Winnipeg, Canada, August 23–26, 1987*, Pergamon, Oxford, 1987, pp. 67 – 84.
16. K. B. Wright and R. Peterson, P. G. Campbell, ed.: *Light Metals 1989*, The Mineral, Metal and Materials Society, Warrendale, 1989, pp. 479–88.
17. J. Lauzon-Gauthier, C. Duchesne and J. Tessier: *JOM*, 2020, vol. 72 1, pp. 287–95.
18. D. P. Ziegler and J. Secasan: Methods for determining green electrode electrical resistivity and methods for making electrodes, patent: US 20140183770A1, 2014.
19. J. Lauzon-Gauthier and J. Secasan, L. Perander, ed.: *Light Metals 2021*, Springer International Publishing, Cham, 2021, pp. 951–956.
20. J. Lauzon-Gauthier: *Multivariate Latent Variable Modelling of the Pre-Baked Anode Manufacturing Process Used in Aluminium Smelting*, Master thesis, Laval University, 2011.
21. I. T. Jolliffe: *Principal component analysis*, 2nd ed., Springer-Verlag, New York, NY, 2002, 487 pp.
22. P. Geladi and B. R. Kowalski: *Anal. Chim. Acta*, 1986, vol. 185, pp. 1 – 17.
23. P. Nomikos and J. F. Macgregor: *Technometrics*, 1995, vol. 37, pp. 41–59.
24. A. Paris: *Contrôle de qualité des anodes de carbone à partir de méthodes statistiques multivariées*, Master thesis, Laval University, 2020.
25. D. C. Montgomery and G. C. Runger: *Applied statistics and probability for engineers*, 5th ed., John Wiley & Sons, 2010, 784 pp.