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PILOT SCALE ANODES FOR RAW MATERIAL EVALUATION AND PROCESS IMPROVEMENT

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Abstract

Primary and secondary raw materials and carbon plant practices are of critical importance for anode quality. Frequent testing of cokes, pitch and production factors in a full scale plant would be feasible, but might be high risk and expensive. It could also be time consuming as the testing would be subject to the demand of production for priority. For over twenty years, Hydro has run systematic pilot scale tests in Årdal, Norway, and since 2005 the facility has been upgraded with intensive mixers and vacuum vibroforming. Today the pilot production simulates full scale operation, and pilot scale results are successfully implemented in carbon plants. The paper discusses the factors that ensure quality practices for pitch level evaluation, aggregate screening curves and baking level control. Examples are from tests of new material sources, a study of secondary raw materials quality related to carboxy reactivity, and studies of production parameters.

Introduction, Anode Development

Background, Hydro in the Anode Plant

Hydro Aluminium is well known as an aluminium producer and developer of cell technology such as the HAL300 and HAL4e cell technology and auxiliary operations technology. In parallel with this, Hydro has run - less well-known - programs for developing anode production technology. Through the last decades, as the cell amperages increased dramatically, parallel anode improvement programs played an important role in this success. The results of the Høyanger smelter exemplifies this: In the beginning of the 80`s most trials of increasing the current above 205 kA in Høyanger were a failure, and insufficient anode quality was one of the contributions to these failures. Today this line is operated at 285 kA and this success has to a high degree depended on the ability of the carbon producers to meet the quality required of anodes that must sustain current densities at 0.90 A/cm² and above. The amperage load for Hydro pots are world class for end to end cells, and this can not be achieved with second class anode quality.

For the anode producer, maintaining and improving anode quality is always a challenge, partly due to the very long cycle time in production. The feedback loop for baked anode properties is counted in weeks, making full scale studies of material and process changes very time consuming. To speed this up, Hydro has turned to pilot scale. Over twenty years the work has evolved from using dry pitch, sigma-mixers and atmospheric vibroforming of cubic blocks to today's use of liquid pitch addition, lab scale Eirich mixers, pre-heated aggregate and close control of energy input and temperature including a controlled cooling stage. For vacuum vibroforming both die and plunger are thermally controlled.

Pilot Scale Studies

The pilot scale studies are important in the materials part of anode development. For the last six years the annual scale of testing has been 60 to 90 batches with over 1400 pilot anodes produced. Quite a wide range of raw materials have been tested, and Hydro has gained good experiences through cooperation with coke, pitch and equipment suppliers.

Pilot scale anode testing is common in the industry, and Hydro has run pilot scale testing and has several publications from this work [1,2,3]. The perhaps best known test equipment is the R&D Carbon bench scale system with roots as far back as 1978 [4]. Work utilizing pilot scale testing includes studies of isotropic cokes [5], pitch studies [6], reactivity studies [7] and even anode baking furnace optimization [8]. The relationship between anode and coke properties is studied using pilot scale testing in another paper in this conference [9].

Pilot Scale Anodes, Challenges

Below is an overview photo showing important steps in the pilot anode production. Each step has challenges relative to quality, and to achieving a realistic simulation of full scale production.

In the foreground (1) are many barrels and buckets and these represent half the number of fractions required in a recent study - note the labels; building up and tracking inventory is critical.

On the left, the multideck screening machine (2) for preparing aggregate fractions is a straightforward devise - the challenge here is ensuring that out-of-the-drum or out-of-the-bigbag material is homogeneous throughout a study.

Suitable mixers are a key to useful pilot scale testing and (3) is an Eirich intensive mixer, run with test portions of 40 kg - there are many challenges among which we can mention estimating the pitch level, simulating the correct temperature and not overdoing mix energy input.

Figure 1: 1) Part of aggregate inventory; 2) Multideck screening machine; 3) Mixer Eirich RV08; 4) Vacuum vibroformer.

In the back we see a sophisticated vacuum vibroformer (4) design made by Siegfried Wilkening at the VAW Bonn lab; the vibroformer has oil heating both in die and load - similar to mixing, a challenge here is to run at correct temperature and not overdo the vibroforming energy input.

Working Environment

Compared to the anode plant, technicians running pilot studies will be more exposed to the raw material dust and fumes; HES issues need to be addressed and followed up closely. The laboratory has considerable experience in this area and the photo above shows five point suction devices for fume collection. However, some stages in the preparation still require use of breathing masks with appropriate filters and further equipment development aims to reduce all open material handling. This will also yield better temperature control in the production.

In the lower right corner is a barrel on a wheeled stand; the pilot work can involve some heavy manual work, and lifting is now mostly motorized; and wheeled barrels are preferred for stock.

Simulating a Full Scale Process with Small Devices

Some steps are simple to simulate in laboratory scale, such as fractionation or preheating, and some are difficult, either due to scale, like the milling of fines, or to the complexity of the process itself like the mixing step.

Anode Fines

Stability in the anode fines production impacts positively on the whole downstream line; process stability; anode mechanical strength and crack resistance. The laboratory mill is a batch device, several hundred times smaller in volume than a wind-swept mill. To make the lab production of fines realistic the milling ball size distribution is important. Milled product is controlled with sieve analysis, grain size analysis down to 0.001 mm and with Blaine number.

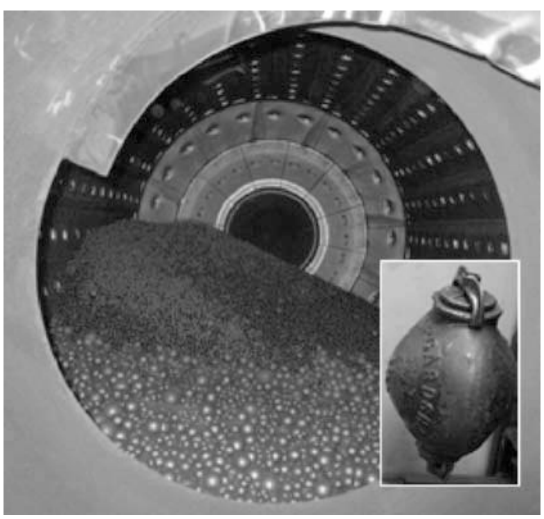

Figure 2: A full scale wind-swept ball-mill, a thousand times larger than the lab mill with capacity 7-kg per 2-hour milling.

The vibroformer

A limitation in simulation is the size of the pilot anode compared to a full-scale anode. Full-scale vibroforming creates gradients due to the cooling, drag and push of the steel walls; the sheer mass of the free-swinging paste in the center of the form, and unavoidable anisotropy of packing. Compared to this, a 3.80 kg pilot with thermally equilibrated freshly oiled walls yields a nearly homogeneous test piece with very little gradients. In cracking studies, this is a disadvantage, but in most investigations a homogeneous pilot anode with good repeatability ensures good quality comparison.

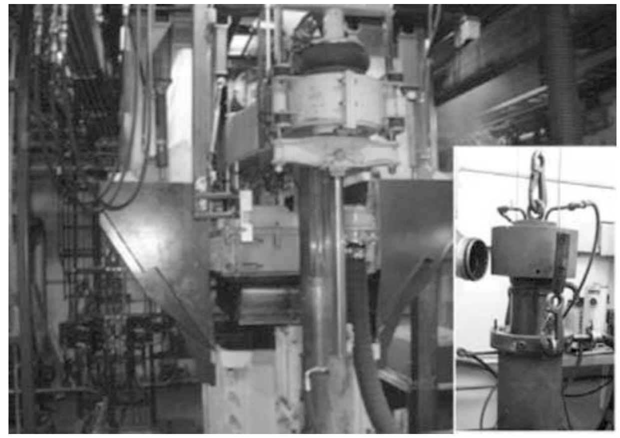

Figure 3: A Hydro vibroformer, making anodes 300 times larger than the labscale vacuum-vib (insert).

The Baking Furnace

The baking furnace is where the difference in scale is most apparent; the photo shows the 112 ktonne per annum Hydro furnace (ABF3 at Hydro Aluminium Årdal, when new) and the two cupola furnaces at the Årdal testing facility. The baking simulates full scale very well as the heat treatment is tuned using the equivalent temperature method, ISO 17499 [10]; it is raw material independent. This stable baking practice ensures equal heat treatment and allows repeatable baked density, coke yield and anode shrinkage, enabling us to do comparison of batches made months apart.

Figure 4: ABF 3, Hydro Aluminium, Årdal Carbon runs at 112 ktonne per annum with a section load of 168 anodes; in the lab cupola furnace the equal heat treatment zone limits the load to sixteen pilots, by weight less than 1/25000th of full scale.

Scope of Hydro Anode Development

What is the place of the pilot scale testing in anode development? For Hydro anode development has been a wide field in the last two decades, from baking furnaces to vibroformers:

- Anode Baking Furnace capacity increased for the vertical flue Hydro ABFs in Sunndal and Årdal so that amperage increases were covered by retrofitting within existing infrastructure [11]
- ABF refractory qualities developed with suppliers and scientific institutions enabling designs with better heat distribution in all ABFs [12]
- Safety in ABF operations improved through CE certified safety systems for closed top furnaces based on the Directive of Machinery and IEC standards [13]
- Further HES improvements achieved with installation of RTO fume treatment systems which combined with a downstream electrostatic precipitator placed the Årdal plant in world class for low atmospheric emissions
- Baked anode design improved, including deep sawing of slots and a unique system for drilling of stub holes based on Norwegian North-Sea oil-drilling technology [14]
- In the paste plant, stabilizing the aggregate quality through coke blending, stabilizing the fines through better ball mill circuit and air classification; improving the mixing string by adapting Eirich mixers to existing lines, improving butts with eddy current separator [15] and developing the Hydro vibroformer with automatic control of the anode density using CarboMaster

From this list it is obvious pilot scale anode testing till today has been a small part in anode development in Hydro, but currently it is becoming a critical and essential part for anode quality. The reason is the wider range of raw materials being taken into use, and the need to test in ways that ensure the paste plants are prepared.

What Can Be Tested?

This is an interesting and debatable question, what can be tested, realistically, in pilot scale? A list of what has been meaningfully tested till today includes

- Qualification of a new raw materials; pilot testing gives good guidance for what anode quality can be expected when a new raw material is introduced in the full scale production
- Reactivity studies on coke and butts - the butts level, butts contamination level, butts' fines; butts cleaning issues and interaction of sodium and sulfur and desulfurization at higher baking levels
- Aggregate studies, closely reproducing full scale recipes including all fractions; making similar recipes for different cokes; introducing new cokes, varying the fines content and fineness; studying anode physical properties with higher or lower coarse grain content
- Anode properties with unusual raw materials, both pitch and cokes, and even coal and tests with charcoals can be done in pilot scale with no risk of upsetting the anode supply
- Evaluation of process equipment regarding the suitability for use with different raw materials

And What Can Not Be Tested?

The major limitation is the scale; a 4 kg pilot is only $1/250^{th}$ of an anode. The size-dependent gradients will not play a part for pilots; as the thermal strain during baking will not be as large as in full scale the risk of cracking can, unfortunately, not be simulated. There are mechanical properties that can be measured that indicate strength and both thermal and mechanical properties that indicate shock resistance, but the direct feedback of strain through cracks is not possible.

<u>Can Anode Density be Tested?</u>
Care must be taken when testing for higher anode density. Often high densities are considered a gain, and in pilot scale it is no problem to supermix the paste and flatten the anode in the vibroformer to green densities of 1.70 g/cm^3 and above. But that is not the purpose of pilot scale simulation of a real paste plant. For simulation, a too high density is equally unsuited as a too low density; high energy input will give high density, but will not aid production!

Making Green Pilot Anodes

A typical batch is 40+ kg with 35.0 kg aggregate, allowing four pilot anodes to be made with moderate spread in green density. The photo shows 20 pilot anodes from five batches, and illustrates the sequential numbering system, with a letter referencing the material. The green pilot anode is 3.80 kg; this is kept constant due to an increase in the density of 0.008 kg/dm^3 per kg paste. The spread of the green density within the 4-pilot batch is low; in 2009 the average standard deviation within 59 batches was 0.008 kg/dm^3, or 0.21 %rel. Previous to establishing a constant in-weight, the standard deviation within a batch could be 0.026 kg/dm^3.

Figure 5: Five batches - each with four pilot anodes, individually marked. Mass is 3.80±0.01 kg/dm^3. Over 1400 have been made in the last six years.

Mass and dimensions are measured pre- and post-baking to determine green density, green aggregate density (GAD), baked density and changes during baking such as coke yield (CY) and shrinkage.

<u>The Raw Materials; Coke and Butts Fractions</u>
The practical solution for large studies has been to collect ready fractioned reference material from Årdal Carbon, e.g. 2.5 metric tons all together of coke fractions, mill product and butt fractions. This is material for 60-80 batches.

<u>Pitch Level</u>
Correct pitch level for good comparison is a challenge to get right. The literature refers to pitch optimization procedures where anodes are made over a range of pitch levels and the resulting

peak of the baked density curve indicates an optimum pitch level. However, for Hydro, when running studies simulating full scale production this has not turned out to be a good solution. In full scale, the pitch level can be 1-3 % down on the left shoulder of the optimization curve, and in addition the anode plant pitch control is very tight; *e.g.* the ramping interval for pitch control might be 0.05 wt% or 0.1 wt% around an average of 14.0 wt%.

When testing with paste plant fractions, the practice has been to use the same pitch level used in the plants, with a small addition due to a batch being made in a clean pan. When testing a new coke, the practice has been to select a likely pitch level based on coke type and inspect the paste for consistency and the vibroformed green anode for surface appearance. A next batch will be made with a small shift in pitch level. Work is underway to replace this practice with analytical procedures and methods for improved control including viscometry, wettability and even pitch distribution studies by quantitative image analysis.

The Baseline

Testing is started with establishing a good simulation of the current paste plant production in an experimental design for pitch and butts percentage, at several baking levels. Testing then proceeds - all the time with the advantage of having the original simulation as a baseline for effects. Standard testing are series with different recipes; series with blending different out-of-the-drum materials into the reference aggregate; alternative pitches; testing the effect of butts adjustments on reactivity and adjustments of process energy input. In these tests the system with the reference baseline composition is a great help in evaluation; and repeating the reference batch at any time is a help to check that the pilot line is stable and comparable.

Brand New Materials; Standardized Testing and Trends

Sometimes little or no ready made coke fractions and fines are in the aggregate - then a set of standard parameters for aggregate recipe, preheating temperature, pitch temperature, mixing energy input and vibroforming energy input are used. The material will be tested at 2-3-4 levels. This is a less certain simulation of the anode plant; what is observed will not be as accurate. But a series' trend will be relevant, pointing the direction for full scale results.

Baking Level

The baking is done, packed in coke within a refractory container. in cupola furnaces of 16 or 8 pilot anode capacity, determined by the size of the zone of equal heat treatment. The zone of equal heat treatment was mapped using the equivalent temperature method [1,10]. The repeatability is better than 10°E enabling reliable comparison at different baking levels, typically normal 1230°E, underbaked 1150° and overbaked 1330°E, very useful for coke studies [2].

Examples from Running Studies

In the following, some of the tools required for a pilot scale line simulating a full scale line are presented, and some issues encountered when trying to match full scale anode manufacture are discussed.

Example 1, Coarseness of the Aggregate

Using fractions from the paste plant is very suited for recipe studies. A study was run in 2009 to look at effects of adding a higher percentage of +5.6 mm material, both coke and butts.

Table of sieving curve control points
X1 and X2 interpolated between Fine and Coarse.

	Fine	Fine-X1	Fine-X2	Coarse
+5.6 mm	15.5	20.1	24.7	29.3
+1.4 mm	42.6	46.1	49.6	53.0
+0.18 mm	69.5	71.4	73.4	75.3
Bottom	15.4	14.9	14.5	14.0

Figure 6: Four level of coarseness of aggregate.

This was a large study, with the plan shown below; four coke materials (blend/single source), four levels of coarseness, and three baking levels. For a pilot anode study this was relatively straight forward as all cokes were known and it was easily recognizable if results were away from reasonable values.

Produced Week	Sieving Curves & Batch numbers Four pilots per batch Fine	Fine-X1	Fine-X2	Coarse	Baking Level °E Bulk vol & dens 1150	1230	1330
Coke A							
2008-w13	c02				x1	x1	x1
2009-w07		c82				x1	x1
2009-w07			c83		x1	x2	x1
2009-w07				c84	x1	x2	x1
Coke B							
2008-w50	c64				x1	x2	x1
2009-w11		c92			x1	x2	x1
2009-w11			c93		x1	x2	x1
2009-w06				c73	x1	x2	x1
Coke C							
2009-w03	c68				x1	x2	x1
2009-w10		c85			x1	x2	x1
2009-w10			c86		x1	x2	x1
2009-w10				c91	x1	x2	x1
Coke D							
2009-w14	b02				x1	x2	x1
2009-w14		b04			x1	x2	x1
2009-w14			b05		x1	x2	x1
2009-w14				b06	x1	x2	x1

Figure 7: Four cokes; four recipes; each batch four pilot anodes; baking levels 1150° (underbake), 1230°E and 1330°E (overbake).

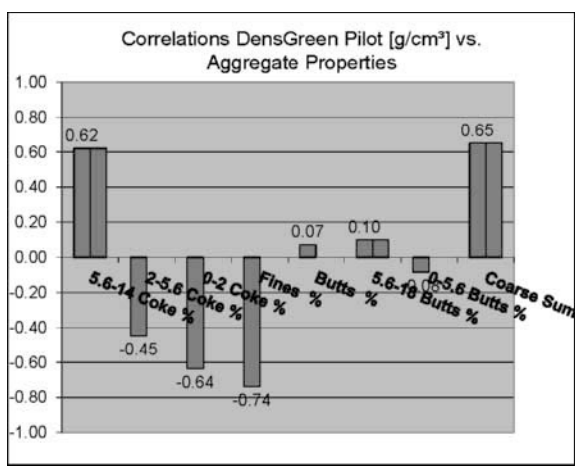

Figure 8: Average correlation of the four coke materials across baking levels - the red indicates the +5.6 mm fractions.

The results shown in Figure 8 are the correlation of the green density with several aggregate properties. This density is the average green density across coke materials and baking levels. An increase in the coarse coke fraction increased the green density.

Example 2, Issue of linearity of coke properties

A result from a small study of coke bulk density showing the effect on VBD when blending two coke materials, LS and NS. It is included to show the versatility of pilot anode testing. For this set the Blend followed the lower bulk density normal sulfur coke material and not the low sulfur, higher density, coke material.

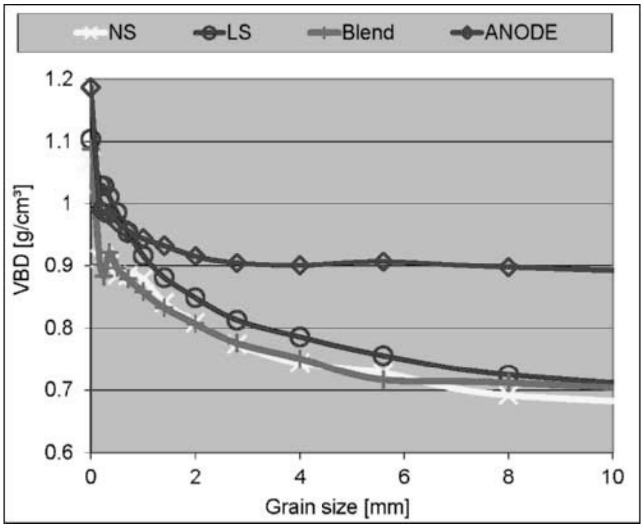

Figure 9: VBD for many fractions; the LN and NS cokes were blended; the ANODE result shows bulk density of crushed anode.

Example 3, Comparing Cokes

Pilot scale allows realistic cost-saving pre-studies that can show if a new material will run normal, or they can give insight early of potential anode issues. One study was run in connection with and previous to renegotiation of a coke supply contract. Two potential cokes C and B were compared with Ref , the current coke.

Coke	Simulate	Mixing input	Vib input	Pitch Level	Pitch Level low	mid	high
Ref	Plant1	High	High	14.1		b50	
Ref	Change	Low	High	14.1		b51	
Ref	Change	Low	Low	14.1		b51	
Ref	Plant2	Low	Low	14.1		b51	
Ref	Plant2	Low	Low	14.1		b52	
Ref	Plant2	Low	Low	14.5			b53
Ref	Plant2	Low	Low	13.7	b54		
Ref	Plant2	Low	Low	14.1		b55	
C	Plant1	High	High	14.1		b56	
C	Plant2	Low	Low	14.1		b57	
C	Plant2	Low	Low	13.5	b58		
C	Plant2	Low	Low	14.5			b59
B	Plant2	Low	Low	13.5	b60		
B	Plant2	Low	Low	14.1		b61	
B	Plant2	Low	Low	14.5			b62
B	Plant1	High	High	14.1		b63	

Figure 10: Test scheme for evaluating two replacement cokes, see results in Figure 12. There are usually several "Change" batches to review effects of process parameters.

Results from this test are shown in the Sharing of Results section below. Note that the purpose of this example is to illustrate systematic use an Excel dashboard type sheet for result presentation and distribution, not to show actual results. However, if the PDF is magnified details are visible. Figure 12 depicts a page with concentrated information, in this case with four groups of the three cokes - three pitch levels, low, medium and high, and the fourth group is a simulation of another paste plant shown in Figure 10 as "High" mix and vib energy input.

Example 4. Dilemma of large experimental designs

As a last example of running studies a special case of too many ideas is shown - the purpose was to test reactivity with coke and butts additions. The study stretched over too much time, invalidating overall comparisons. Individual segments were useful 2x2 factorials, but the scope grew past original planning with many additions such as an extra recipe, extra calcination levels, and this stretched the study beyond the stability of the coke materials.

		Recipe I				Recipe II		
Coke Type	Butts Fines	Na0503	Na0781	Na1681	Na4000	Na0503	Na1681	Na4000
	yes	b23						
	no	b14						
HS-1	yes	b24						
HS-1	no	b16						
	yes			b25				
	no			b18				
HS-1	yes			b26				
HS-1	no			b20				
	yes				b08			
	no				b09			
HS-1	yes				b10			
HS-1	no				b11			
	yes	b23	see above					
	no	b14	see above					
NS-1	yes	b53						
NS-1	no	b54						
	yes		not	b25	see above			
	no		not	b18	see above			
NS-1	yes		b48					
NS-1	no		b49					
	yes				b08	see above		
	no				b09	see above		
NS-1	yes				b51			
NS-1	no				b52			
	yes					b13		
HS-1	yes					b15		
	yes						b17	
HS-1	yes						b19	
	yes							b02
HS-1	yes							b21
HS-1	yes							b22

Figure 11: Test scheme of a study that started simple but ran too far with many extra parameters. Na0503 means 503 ppm in the fine butts fraction.

Current Issues

Analysis Development

Development of analysis methods is important in the work; both establishing new methods and learning to use existing analyses better. One must be aware that most standard analysis methods for anodes are made for mass production and are used to report an average for a lot, a week's or a month's anode production. For instance, the specific electrical resistivity has a within-lab precision when comparing two anodes of 1.2 μΩm at 95% confidence level; two anodes can not be distinguished if the difference is below 1.2 μΩm [16]. Using this value of spread to estimate the uncertainty in the average of a lot represented by 25 samples gives $1.2/\sqrt{(25)}$, or 0.24 μΩm; this means the average reported is a good estimate for the lot. But for pilot scale comparison using one measurements for each anode, the uncertainty is 1.2 μΩm at 95% confidence level! Results are not significant if the difference is less so doing more than one analysis can be critical for the strength of conclusions in a pilot scale test program. Awareness of this is important when considering number of parallels, or the need for analysis improvement.

Sharing of Results

As pilot scale testing grows the need for efficient procedures become apparent: systems for planning that are recognized by the team involved; systematic execution of test programs so the work can be delegated; sharing of the results - distribution of results. The Excel sheet in Figure 12 illustrates a system of compact information that is used to distribute results.

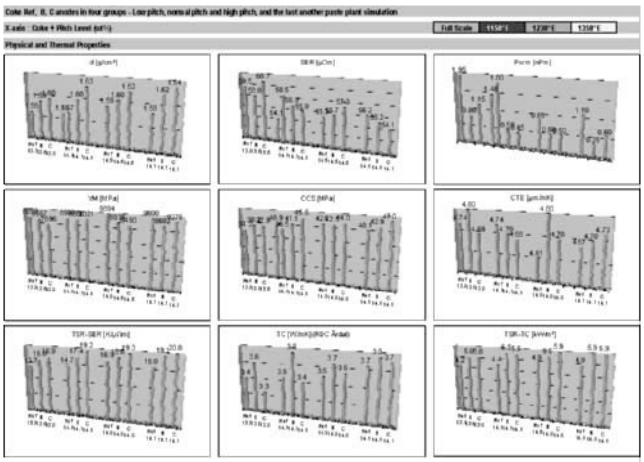

Figure 12: Example of compact presentation of results from the test plan in Figure 10. Comparison of a reference coke, Ref, with two cokes B and C; the green bar is full scale anode results.

Visiting Scholars

The pilot facility is very suited for inviting students for summer jobs; this can be followed with related Project work and a MSc thesis, and even PhD level work. This is a boost for the research group and gives positive signals to the organization. Visiting work sometimes is of a very specialized nature and e.g. addresses special anode materials such as coal materials or charcoal, or unusual binders or other non-traditional applications [3].

Pilot Testing and Full Scale Testing

The final proof for anode quality is the performance in the pot room. The final result is therefore not seen before pilot findings are introduced in regular anode production. These are examples where pilot scale testing has played an important part.

- Pilot scale to full-scale testing of anodes with new raw materials
- Butts cleaning and limiting impurities that are circulated back into the anode aggregate e.g. by butts fines removal
- Adjusting recipes for stability; fines level, fines fineness and pitch level control; limits to gluing together of anodes in the ABF
- Studies of raw materials versus anode properties
- Plant optimization of aggregate, mixing improvements and forming improvements
- In close cooperation with electrolysis, improving rodding produced anode cover materials and general characterization and monitoring of ACM

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