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Anode Raw Materials

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Pilot Anode Testing of Alternative Binder and CPC Raw Materials

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Abstract

The value of pilot anode testing for evaluating raw materials, anode recipes and mixing/forming changes has been well documented in previous studies. Since early 2013, RÜTGERS and Rain CII have invested significant effort into developing a new pilot anode facility at Castrop-Rauxel, Germany. With the ability to mix and form paste at temperatures up to 300 °C and bake anodes at temperatures up to 1300 °C, this new facility is now being used actively across a wide range of carbon raw material R&D projects. Pilot-scale anode results will be presented to expand on previously reported work on low-PAH binder systems along with tests on different quality CPC raw materials. The good reproducibility of the pilot anode process allows better correlation of raw material properties and processing parameters on anode quality with a well targeted testing program.

Introduction

Calcined petroleum coke (CPC) and coal tar pitch (CTP) are the key raw materials used for the production of carbon anodes in the Hall-Héroult process. Both raw materials are produced from byproducts of other industries and as a result, are subject to quality changes. The shortage of traditional quality, green petroleum coke (GPC) used for the production of CPC has been well documented [1]. This shortage has led to a general increase in trace metal impurities and sulfur levels, and a decrease in bulk densities. Aluminum producers have adapted well to these quality changes and there is a growing trend for smelters to continue relaxing CPC specifications in an effort to source lower cost raw materials.

CTP continues to vary in QI content as a result of coal tar quality changes from cokeries [2]. The primary concern with CTP today however, is not performance, but the presence of polycyclic aromatic hydrocarbons (PAHs). The regulatory focus on PAH reductions in the environment is targeting coal and petrochemicals containing PAHs. In 2009, CTP was classified as a PBT (**P**ersistent, **B**io-accumulating and **T**oxic) substance in Europe [3]. As a consequence, authorization of CTP for all applications according to European legislation (REACH, Regulation (EC) No 1907/2006) has been proposed [4].

PAH reductions have been achieved already for Soederberg anodes by mixing CTP and petroleum pitches from fluid catalytic cracker (FCC) or steam-cracker streams [5,6]. Another option is to increase the CTP Mettler softening point (SPM) [7,8]. The toxic PAHs are the low boiling constituents of CTP that can be removed by advanced manufacturing technologies. The drawback here is that many of today's carbon plants have a limited ability to increase mixing temperatures, so equipment upgrades may be required.

In early 2013, RÜTGERS was acquired by Rain CII Carbon and the two companies worked together to upgrade the pilot anode production capabilities at the RÜTGERS Castrop-Rauxel facility. Prior to 2013, all anode testing was done with small, bench scale anodes (50 mm diameter). This small size makes it difficult to evaluate the impact of changes like baking rate on anode properties since the volatile release pathway is so short. Many anode property tests are done on 50 mm Ø (diameter) samples so it is difficult to avoid mold edge-effects when using 50 mm Ø anodes. Larger, pilot-scale anodes (150 mm Ø) where 50 mm Ø samples can be removed by core-drilling, are much preferred for the above reasons.

The following paper describes the pilot anode facility at Castrop-Rauxel and gives examples of some of the work being done on a range of CTP and CPC materials. The paper highlights the close cooperation between RÜTGERS and Rain CII on anode related R&D. Combining the coke and pitch know-how of both companies is helpful in dealing with current and future CTP and CPC raw material challenges.

Equipment for Pilot Anode Production

The pilot anode production line consists of a high-temperature mixer, a high-temperature press and a baking furnace, Figure 1. The Eirich Mixer prototype (RV02/E) was developed in cooperation with Eirich. The mixer has been in operation since 2012 and allows preparation of paste at temperatures up to 300 °C using an infrared (IR) heating system. This system has the advantage of being relatively small compared to an inductively heated setup used previously for testing with very high SPM pitches [7]. Additionally, the paste temperature can be easily controlled and monitored during mixing. With its 10 liter mixing pan, the mixer is capable of preparing 6 -7 kg batches of paste.

Figure 1: Pilot anode facility showing IR heated Eirich mixer, hydraulic press and baking furnace

After mixing, the green paste mix is transferred to a high temperature pilot press for forming. This custom-built press was commissioned in 2013 and the mold can be heated and maintained at temperatures up to 320 °C during pressing. The press is used for

both pilot anode and refractory development work and can produce bench scale and pilot scale anodes. Both plungers are CNC controlled allowing a variety of pressing cycles. The height of the anode during pressing is also monitored, so that the spring-back can be measured. This allows monitoring of paste rigidity. Typical green anode sizes are - diameter: 146 mm; height: 190-200 mm; weight: 5.2 – 5.5 kg. In combination, the mixer and press are capable of processing CTP with SPMs up to 230 °C.

The last piece of equipment added to the pilot anode line was an anode baking furnace in 2014. The furnace is capable of baking 12 anodes/batch at temperatures up to 1300 °C which provides flexibility when simulating the different baking levels found in industrial scale furnaces. The design allows for easy loading and unloading of anodes from a standing position, which was an important ergonomic goal. The furnace is calibrated using the Equivalent Temperature (°E) method according to ISO 17499 [9].

Synergies in Raw Materials Supply

Access to new qualities and a reliable stock of standard CPC and CTP is important for conducting raw material focused R&D work. The preparation of aggregate fractions (CPC and butts) for pilot anode production is done at Rain CII´s laboratory in Lake Charles, Louisiana. The fines fraction is prepared in a ball mill using laser particle size analysis for control but RÜTGERS also measures the Blaine Index of all fines batches before making anodes.

Materials and Methods

Binder

Two different CTP based binders were used for the work reported in this study with properties summarized in Table I. Bx 95KS is a standard CTP binder used in many anode plants. HMR 180M is a commercially available binder used in the specialty graphite industry but not yet used for anode production. The HMR 180M binder contains a lower amount of volatiles and has a significantly higher coking value. Volatiles are less in volume and the release is shifted to higher temperatures, Figure 2. According to the CANTOX classification [10,11], the material exhibits a 62% reduction in average toxicity compared to a standard binder pitch.
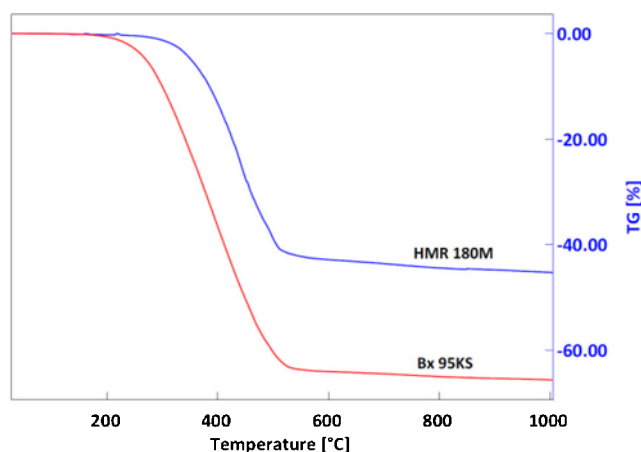

Figure 2: TGA of Bx 95KS and HMR 180M

Table I: Properties of different binders used for pilot anode production and CANTOX-Calculations according to [10,11]

			Bx 95KS	HMR 180M
Softening point Mettler	DIN 51920	[°C]	112	181.6
Coking value (Alcan)	DIN 51905	[%]	57.6	75.7
Toluene Insoluble	DIN 51906	[%]	25.7	37.7
Quinoline Insoluble	DIN 51921	[%]	5.1	8.5
Ash (800°C)	DIN 51922	[%]	0.17	0.26
XRF-Analysis	DIN 51418			
S		[%]	0.59	0.52
Na		[ppm]	80	114
Si		[ppm]	153	309
P		[ppm]	<5	5
Ca		[ppm]	36	80
Fe		[ppm]	142	199
Zn		[ppm]	284	350
Pb		[ppm]	222	242
Distillate 020 - 230°C	GC	[%]	0.03	0.00
Distillate 230 - 270°C	GC	[%]	0.01	0.00
Distillate 270 - 360°C	GC	[%]	0.84	0.03
Distillate 360 - 440°C	GC	[%]	5.18	0.11
Distillate 440 - 500°C	GC	[%]	15.60	2.32
Distillate >500°C	GC	[%]	13.70	15.79
Sum of Distillates	**GC**	**[%]**	**35.36**	**18.24**

GC-analysis		RPF (Cantox)	PAH [ppm]	BaP-equiv	PAH [ppm]	BaP-equiv
Naphthalene		0.000	28	0	0	0
Acenaphthylene		0.000	0	0	0	0
Acenaphthene		0.000	714	0	56	0
Fluorene		0.000	195	0	0	0
Phenanthrene		0.000	2377	0	40	0
Anthracene		0.000	544	0	0	0
Fluoranthene		0.034	9639	328	124	4
Pyrene		0.000	8269	0	125	0
Benzo[a]anthracene		0.033	9123	301	283	9
Chrysene		0.260	9201	2392	334	87
Benzo[e]pyrene		0.050	10173	509	1766	88
Benzo[b]fluoranthene		0.100	10994	1099	1759	176
Benzo[k]fluoranthene		0.010	5224	52	708	7
Benzo[a]pyrene		1.000	12176	12176	2355	2355
Dibenzo[a,h]anthracene		1.400	1907	2670	869	1217
Benzo[ghi]perylene		1.000	9150	9150	6130	6130
Indeno[1,2,3-cd]pyrene		0.100	8354	835	4983	498
Total			98068	29513	19532	10572
Average toxicity index				1.07		0.38

Calcined Petroleum Coke & Butts

The properties of the different CPC's used in this study are summarized in Table II. Coke A is a typical rotary kiln calcined coke and is used as a reference for pilot anode testing. Coke B and Coke D are different shaft calcined cokes, both sourced from China. Coke E is a shot coke which has a different particle shape and a highly isotropic texture relative to the other sponge cokes. Data are also included for the butts material used in all the pilot anode tests which is actually baked anode scrap from an anode plant. It was selected specifically for its low Na level.

Table II: Properties of different CPC & butts material

Physical Analysis		Coke A	Coke B	Coke D	Coke E	Butts
Real Density	[g/cm³]	2.052	2.080	2.128	1.99	2.070
CO₂ Reactivity	[%]	9.5	2.7	39.7	3.8	23.6
Ash	[%]	0.17	0.17	0.38	0.43	0.66
Lc	[Å]	25.2	30.1	40.72	27	30.7
VBD ASTM D4292 28x48	[g/cm³]	0.893	0.971	0.980	1.042	0.935
Chemical Analysis						
S	[%]	2.94	4.19	0.47	4.41	2.13
V	[ppm]	320	408	21	995	241
Ni	[ppm]	189	191	378	370	144
Fe	[ppm]	174	151	168	167	784
Ca	[ppm]	87	53	394	97	211
Si	[ppm]	70	126	96	128	272
Na	[ppm]	87	101	30	90	115

Dry recipe

The dry aggregate recipe used for the pilot anodes is shown in Figure 3. A top size of 8mm is used for all fractions to avoid problems with crushing of larger particles in the Eirich mixer. A standard level of 20 % butts is used in all recipes and added in two different size fractions in the range of 1.7-8 mm.
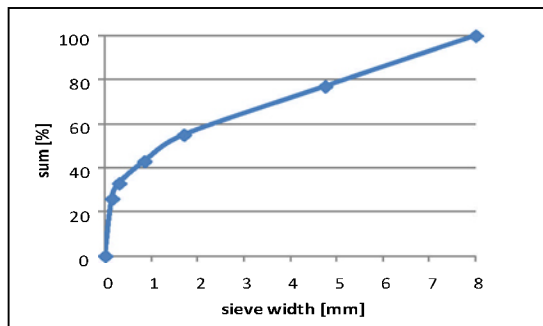

Figure 3: Particle size distribution of aggregate recipe

The CPC fines are milled to a target of 45 % -32 μm. The Blaine value of the ball mill fines used for the pilot anodes produced for this study was ~5000 Blaine.

Pilot anode production procedure

5.5 kg of the dry aggregate is preheated to the mixing temperature, transferred to the preheated mixer and homogenized for one minute. The liquid binder is preheated to a temperature 100 °C above the SPM and added after the 1 minute dry mix. The anode paste is mixed for 10 minutes at 70 °C above the SPM of the binder. After mixing, the paste is cooled to 20 °C above the SPM, transferred into the preheated pilot press and subsequently pressed at 42 MPa for one minute.

The green anode is removed, cooled to ambient temperature and the green apparent density measured. Anode batches are baked to an Equivalent temperature of 1220 °E using a gradient of 20 °C/h. After baking, the anode weight and physical dimensions are measured to allow calculation of the shrinkage and baking. Three 50 mm Ø cores are drilled per anode. The cores are analyzed according to the R&D Carbon standard procedure [12]. Baked apparent density, specific electrical resistivity, flexural strength, compressive strength, elasticity modulus, air permeability and CO₂

reactivity of the resulting core samples are determined. Additional measurements can be performed as required.

Pilot Anode studies

PAH reduced binder

The increase of SPM to reduce the PAH content of CTP is not a new idea [13]. With increasing regulatory pressure however, more effort is being made to evaluate potential options. The increase in SPM not only reduces PAHs but also improves anode performance [7]. The current study was intended as a scale-up of previous bench scale studies with a 180 °C SPM binder hereafter referred to as HMR 180M (**H**igh **M**elting **R**esin).

The use of HMR binders for paste production is challenging for the process equipment. Mixing temperatures have to be increased with the higher softening points keeping the paste viscosity within the target range. It is acknowledged of course, that most carbon plants will not be able to preheat the aggregate and mix the paste at the required temperature without some form of upgrade. Some process equipment in common use today, like co-kneaders, are routinely operated at much higher temperatures in industries like plastics manufacturing so there is no technical reason why anode paste cannot be mixed at higher temperatures.

Results and Discussion

The following summarizes pilot anode results of the two binders with coke A. The green apparent density typically increases for both binders with increasing pitch level, Figure 4. The baked apparent density increases by 0.034 kg/dm³ with the use of HMR 180M, Figure 5. The maximum baked density for HMR 180M is achieved at a pitch level of 13.5 % compared to 14.0 % for the Bx 95KS binder indicating a lower binder demand, probably due to a higher concentration of binding constituents. The baked density results are the average of 3 core samples and repeatability testing on 10 anodes returned a 95% confidence level of 0.0027 kg/dm³.
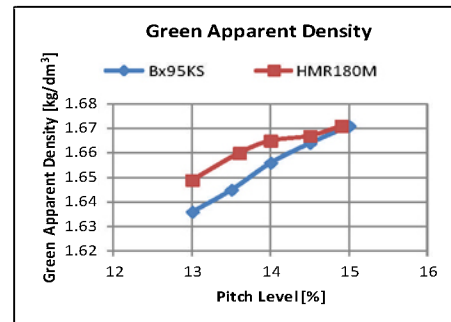

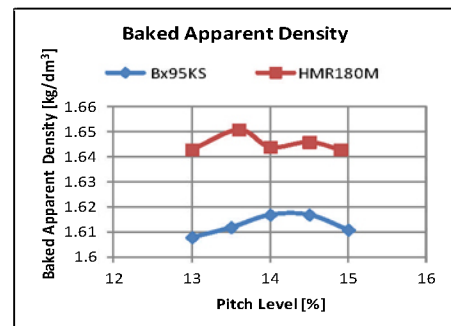Figure 4: Green apparent density vs. pitch level

Figure 5: Baked apparent density vs. pitch level

Results for baking shrinkage and baking loss are shown in Table III. The baking loss of HMR 180M anodes is reduced by 1.8 % compared to BX 95KS at the maximum baked apparent density. This reflects the lower amount of volatiles and therefore the higher coking value of the binder. Pilot anodes produced with Bx 95KS exhibit a higher shrinkage than anodes made with the HMR binders. This is probably due to the decreased release of volatiles.

Table III: Baking Loss and Baking Shrinkage

Binder Level [%]	Baking Loss [%]		Baking Shrinkage [%]	
	BX95KS	HMR180M	BX95KS	HMR180M
13.0	4.1	2.6	2.5	1.8
13.5	4.4	2.8	2.6	1.6
14.0	4.6	2.8	2.2	1.5
14.5	4.8	3.0	2.3	1.5
15.0	5.0	3.1	1.7	1.3

The lower electrical resistivity and higher flexural strength indicate a better structural integrity of anodes produced with the HMR 180M as well as less defects, Figure 6,7. The 95 % confidence levels are 0.71 μΩm and 0.61 MPa respectively.
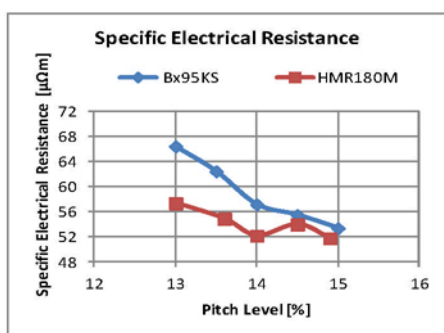

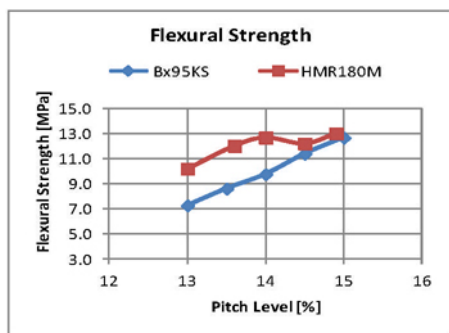Figure 6: Specific electrical resistance vs. pitch level

Figure 7: Flexural strength vs. pitch level

The air permeability level of the anodes was very low with all values in the range of 0.2-0.4nPm (95 % confidence level of 0.02nPm). This is due partially to the use of fines with a Blaine value of 5000 but also highlights the very uniform paste mixing and forming. In modern paste plants, Blaine values up to 4500-5000 are becoming more common. Air permeabilities in the range of 0.3 nPm represent best practice today.

The use of HMR as a binder increases the compressive strength, Figure 8. The modulus of elasticity is still in a reasonable range for all anodes (2.7-4 GPa) and not expected to cause any thermal shock problems [14].
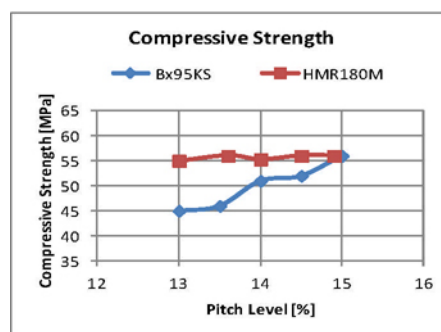

Figure 8: Compressive strength vs. pitch level

The CO_2-reactivity is driven primarily by the sulfur level and trace metal impurity levels in the CPC. As a result, no significant differences were measured between the different binders tested in this study and all CO_2 reactivity residues were measured in the range of 93-96 %.

CPC raw materials

The objective of the CPC investigation in this study, was to look at three quite different cokes – regular rotary kiln calcined coke (coke A); shaft calcined coke (cokes B & D) and shot coke (Coke E). Shaft calcined coke is well known for its lower porosity and higher bulk density relative to rotary kiln and rotary hearth calcined coke [15]. It can be used to maintain, or improve anode densities when used in a blend with other cokes.

Shot coke is a highly isotropic coke which has been well described in a previous paper [16]. It has a high bulk density and spherical particle shape and additions of shot coke can also improve anode density. The use of non-traditional cokes is growing [1], due to their ready availability and significantly lower cost. Controlled smelter trials have been conducted with shot coke additions of 20 % [16] and a full scale smelter trial with a lower level of shot coke has been underway for the last 12 months with good results. The plan is to gradually increase the level of shot coke over a 2 year period.

Shaft Calcined Coke Testing

The two different shaft calcined cokes used in this study were selected to illustrate the impact of very different sulfur and trace metal impurity levels. Coke B is a high sulfur coke produced at Rain CII's shaft calciner in China. Coke D is a low sulfur, low vanadium coke produced in the Northeast of China. It's an example of a coke that can be used in blends to maintain sulfur and vanadium level specifications.

As well as producing pilot anodes with 100 % these cokes, trials were also completed by adding the cokes selectively, to different parts of the aggregate recipe. This can have a significant impact on anode properties such as density and reactivity which needs to be considered when blending different quality CPC. In the case of shot coke, pilot anodes were produced with a blend of 80 % coke A and 20 % shot coke E added to the coarse fraction. Bx 95KS was used as the standard binder for all recipes along with 20 % of the butts material shown in Table II.

Results and discussion

Figure 9 compares the baked density of anodes made with coke A to anodes made with coke B and coke D. Results are also shown for the blend with 80 % coke A and 20 % coke E. The results highlight

the reduction in optimum pitch level for anodes made with shaft calcined coke and the increase in anode density ($0.03 - 0.04$ kg/dm^3 higher). The results also highlight the density benefit of adding 20 % shot coke to a rotary kiln calcined coke blend.
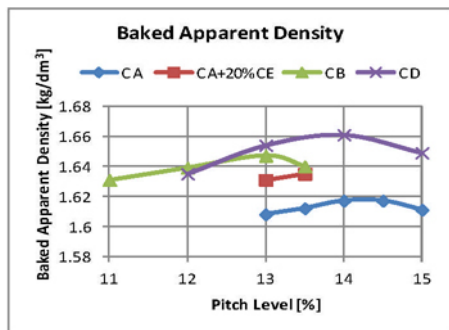

Figure 9: Baked anode density with cokes A, A+ 20% E, B & D

Another property that showed significant differences was anode CO_2 reactivity, Figure 10. Not surprisingly, the anodes made with coke D showed a lower CO_2 reactivity residue than the rest of the anodes (86.7 % vs an average of 94-97 % for the other anodes). This is due to the much lower S level and elevated Ca level of this coke [17] which is also clear from the CPC reactivity results in Table II.
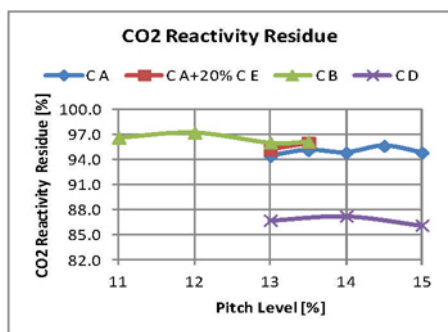

Figure 10: Anode CO_2 reactivity residues

For results like electrical resistivity, air permeability, flexural strength and compressive strength, there were no significant differences between coke A and the two shaft calcined cokes at the optimum pitch levels. Pilot anodes produced with the blend containing 20 % shot coke showed a slightly lower electrical resistivity, Figure 11.
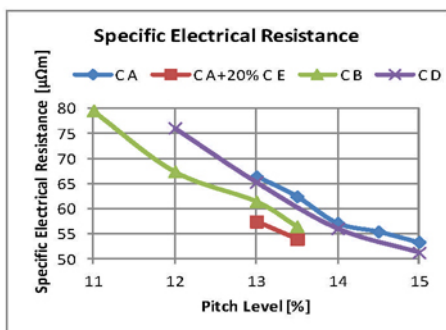

Figure 11: Electrical resistivity of pilot anodes

In Figure 12, the impact of using different cokes in different parts of the aggregate recipe (see Table IV) is shown. It shows baked densities for anodes with coke B in the coarse fraction and coke D

in the fines; coke B in the coarse fraction and coke A in the fines; and coke D in the coarse fraction and coke A in the fines.
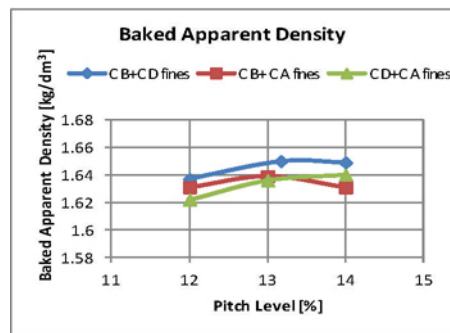

Figure 12: Impact of adding cokes to different fractions

The results are in-line with expectations, where anodes made with coke B in the coarse fraction and coke D in the fines fraction (both shaft calcined cokes) had similar baked densities to the anodes made with coke B and D shown in Figure 9. For the anodes made with the shaft cokes in the coarse fraction and coke A in the fines fraction, the baked densities were a little lower but still significantly higher than anodes made with 100 % coke A.

The CO_2 reactivity results on the other hand, are quite different as shown in Table IV. In this case, the CO_2 reactivity residue of anodes where coke D was added to the fines fraction (Trial 1) were significantly lower (90.4 %) than anodes where coke D was added to the coarse fraction (Trial 2, 94.8 %). In the latter case, the lower reactivity, coke A, was used in the fines fraction.

Table IV: CO_2 reactivity results for different anode formulations

		Trial set 1	Trial set 2	Trial set 3
Dry aggregate	Fraction 0.3-8mm	Coke B + Butts	Coke D + Butts	Coke B + Butts
	Fraction 0-0.3mm	Coke D	Coke A	Coke A
Average CO_2-Reactivity Residue [%]		90.4	94.8	95.3
Average CO_2-Reactivity Dust [%]		3	0.5	0.5
Average CO_2-Reactivity Loss [%]		6.7	4.6	4.3

The above demonstrates the benefit of blending cokes more strategically, particularly cokes that have different bulk densities and reactivities. To improve anode densities, it is better to concentrate higher bulk density cokes in the coarse fraction. If cokes have different CO_2 reactivities, it is better to use higher reactivity cokes in the coarse fraction and avoid their use in the fines. Likewise, it is better to concentrate lower reactivity cokes in the fines fraction as reported in an earlier paper [18].

The above is important when considering the use of shaft calcined cokes in a blend. Most shaft calcined cokes have good bulk densities but some have elevated CO_2 reactivities as a result of higher than typical Ca levels. This can be particularly problematic if Ca and other impurities are concentrated in the fines fraction as reported previously [19]. This is a feature of a number of Chinese cokes that needs to be considered. The other point to keep in mind when using shaft CPC, is that the coke can be a little harder and contain a higher percentage of fines relative to rotary kiln CPC. This can drive additional variation in the ball mill operation if the blend ratios vary significantly (eg >30 %).

Discussion and Conclusions

A primary objective of this paper is to demonstrate the value of pilot anode scale work when evaluating different CTP and CPC raw materials and different anode recipes and formulations. RÜTGERS and Rain CII have invested significant resources over the last two years in developing a state-of-the-art pilot anode facility that can be used for such evaluations. The results presented in this paper represent a subset of a larger and very active R&D program to investigate alternative raw materials with the aim of improving the economics and performance of anode production. The combined group is in a unique positon of being a supplier of both CTP and CPC raw materials.

The results presented for the HMR 180M binder show the value for the industry of continuing to push softening points higher. This can bring significant value in the form of lower PAH emissions and improved anode quality and performance. Although not discussed in this paper, use of binders like this could potentially allow faster anode baking rates [7] as a result of lower volatile emissions. This could provide a significant productivity benefit for anode plants. The next obvious step is verification at the full industrial scale.

On the CPC side, the density of anodes produced with shaft and rotary kiln CPC are compared. Although the density increase with shaft CPC is well known, almost no quantitative anode density results have been provided in past papers and this paper seeks to address that. The paper also highlights the density benefits of using relatively modest amounts of shot coke in anode blends. Although shot coke typically comes with higher sulfur and vanadium levels, it is a low cost raw material that can bring significant cost savings to a smelter and the interest in this area is growing.

The other issue highlighted in this study is the need to be cautious about impurity levels and reactivities in some shaft calcined cokes. They need to be monitored on a regular basis and particular attention needs to be paid to differences between bulk impurity levels and those in the fines fraction. Any concentration of catalytic impurities like Ca in the fines fraction will increase the reactivity of the binder matrix.

Another takeaway from this paper, is the potential to maximize anode performance by concentrating different cokes in different parts of the anode recipe. It is beneficial to concentrate higher density cokes in the coarse fraction and lower reactivity cokes in the fines fraction. It may be advantageous to look at flow sheet changes that allow this as some smelters have done

For smelters using both rotary kiln and shaft CPC, large swings in pitch demand and anode density can be avoided by blending these cokes at a constant ratio. This can also minimize problems associated with different fines levels and different hardness levels between cokes which can affect ball mill stability and performance.

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