

Anhydrous Carbon Pellets – An Engineered CPC Raw Material

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Abstract

The paper reports on a new technology Rain Carbon has been developing to produce an engineered calcined petroleum coke (CPC) product. Agglomeration of green petroleum coke (GPC) fines through either granulation/pelletizing or briquetting can be used to produce a CPC product with improved properties. Pelletizing GPC fines can produce high bulk density pellets hereafter referred to as anhydrous carbon pellets or ACP. ACP densifies when calcined to produce a high bulk density, free flowing CPC product. The spherical particle shape provides improved particle packing densities to irregular shaped CPC particles during anode production. The paper will summarize key results including pilot anode properties showing improvements in baked anode density, electrical resistivity and other properties when using ACP. A key benefit of ACP is the ability to produce a fully engineered CPC product and Rain Carbon is currently working on building a full scale, commercial plant in the US.

Keywords: Petroleum Coke, Calcination, Anode, Density, Agglomeration, ACP

1. Introduction

Agglomeration of fine particle size, bulk solids is a well-established process technology used by many different industries. One of the most well-known applications is granulation or pelletizing of iron ore fines. This is done on a massive scale with some plants producing more than 9 million tons per year. At the other end of the spectrum in terms of volume and product value, is the pharmaceutical industry which typically uses high speed pellet presses to make tablets. Between these two extremes many different agglomeration technologies are used including briquetting, spray drying/granulation and pellet mills which are now routinely used to make wood or biomass pellets for heating and/or power generation [1].

In 2011, Rain Carbon started to experiment with the agglomeration of green petroleum coke (GPC) fines as a way to improve calcining economics. When a rotary kiln is used for calcining, around 10% of the finest particle size GPC becomes entrained in the counter-current flue gas stream inside the kiln. The fines are carried out the back end of the kiln and into a high temperature combustion chamber or pyroscrubber where they are combusted along with any remaining volatile matter (VM). The heat generated in this process is typically recovered and used to produce steam in a heat recovery steam generator (HRSG). Most plants with a HRSG generate electrical power via a steam turbine generator. The sale of power (or steam) is normally an important economic component of the calciner. GPC fines loss is much lower in a shaft calciner (typically <3%) due to the absence of a counter-current flue gas stream [2] inside the calciner.

As GPC prices increase, rotary kiln calciners are incentivized to reduce GPC fines carryover. Since power prices are normally fixed, it is more favorable for a calciner to convert as much GPC to calcined petroleum coke (CPC) as possible. In 2011, low sulfur GPC prices increased dramatically with US Gulf prices hitting US\$400/ton in Q3 2011 [3]. The idea to separate out and agglomerate GPC fines using one of the commercially available technologies was conceived

during this period. It provides a way to reduce fines carryover and recover the fines as CPC product instead. Granulation to produce spherical shaped pellets was selected as the first technology to evaluate since there are some potential benefits in producing round particles in terms of particle packing density and powder flowability. A recent paper [4] highlights the particle packing benefits of spherical shaped CPC particles.

A second benefit of agglomerating GPC fines is an environmental one. When fine particle size GPC is combusted in a pyroscrubber, any sulfur in the GPC will be fully converted to SO₂ in the flue gas stream. During calcination, all cokes lose some sulfur so the sulfur level of CPC is always lower than the GPC sulfur level. For low sulfur cokes (<2.5 %), the sulfur loss is in the range of ~8-10 % and for higher sulfur cokes, ~11-14 %. The generation of SO₂ via this process is unavoidable and occurs in both rotary kiln and shaft calciners. Fines carryover, which takes place predominantly in rotary kiln calciners, results in a further increase in SO₂ emissions and the higher the GPC sulfur level, the higher the SO₂ emissions via this route. Agglomeration of fines therefore eliminates SO₂ emissions associated with fines combustion. For calciners that scrub SO₂, this will reduce scrubber operating costs and for calciners without scrubbers, it will reduce stack SO₂ emissions.

2. Rationale for GPC Fines Agglomeration

Although agglomeration is a well-known technology, the idea to agglomerate GPC fines was based on an understanding of what would likely happen to these agglomerates during calcination. It was expected that the agglomerated fines would densify and form a calcined product with good bulk density and strength. The agglomeration tendency of GPC has been described previously [5] in relation to its contribution to problems like coke ring formation. GPC fines typically contain a higher volatile matter (VM) content than coarser coke particles due to the heterogeneity of coke formation in the delayed coker. Well-coked material at the bottom of the coke drum has a lower VM and is harder than coke at the top of the drum. When the drum is de-coked with a high-pressure water jet, the softer, higher VM coke breaks down into fines and the coarser, lower VM coke remains in larger coke pieces.

If the fines are agglomerated and heated, the relatively high VM material softens and generates condensable tars which make it sticky. With further heating, these tars undergo cracking reactions which generate lighter hydrocarbon molecules like CH₄ and H₂ [6] which are then combusted in the kiln and pyroscrubber. With further heat treatment, the tars form solid coke which binds particles together in close proximity to each other. This phenomenon also leads to the formation of an agglomerated product from a shaft calciner [2] and is the fundamental basis for the technology development described in this paper. If GPC fines can be agglomerated successfully into a high bulk density precursor, the agglomerate should densify and develop strength during calcining.

Preliminary agglomeration trials were undertaken using granulation/pelletizing equipment similar to that used by ceramic proppant manufacturers [7]. Initial results were encouraging and Rain Carbon filed two patents on the concept in 2011 which were granted in 2013 and 2014 [8,9]. The scale of work done on the project gradually increased after that with the ultimate goal of developing a commercially viable process technology. Rain Carbon is now in the early stages of constructing a plant to make ACP.

3. Initial Results

In the first set of experiments, GPC was screened at a particle size of 2 mm. The -2 mm fines were pelletized using a wide range of binders such as PVA (polyvinyl alcohol), CMC (carboxymethyl cellulose), molasses, dextrin (sugar) and coal tar pitch. Most of the binders worked quite well except coal tar pitch. The GPC had to be heated first to remove the moisture and then heated to

around 150C for pelletizing. A ~110 °C softening point pitch was used and added in a solid form. It took some time to melt the pitch, but the pellets formed readily after this.

All pellets were dried first and the resulting ACP calcined in a static lab furnace to a temperature of 1300 °C. Pellet strengths and bulk densities were generally excellent after calcining. The bulk densities varied depending on the method used but for Rain Carbon's standard 8x14 Tyler mesh (1.18 – 2.36 mm) vibrated bulk density test (VBD) the bulk densities were in the range of 0.80 to 1.00 g/cc. This compares with a typical range of 0.78 – 0.82 g/cc for the same GPC calcined in the standard rotary kiln process. Static lab calcining always gives higher bulk densities than rotary kiln calcining since it is not possible to emulate the rapid heat up rate and devolatilization that occurs in a rotary kiln.

Figure 1 shows a photograph of the starting GPC fines, ACP and calcined ACP. The green pellets are always dried after pelletizing to develop sufficient green strength for further handling. A simple crush strength test was developed to test the pellet strength after drying and calcining and this was useful to check the performance of different binders and pelletizing conditions. The crush strength typically increases by a factor of 3-4 times after calcining due to pellet densification.



Figure 1. GPC fines, ACP and Calcined ACP

4. Larger Scale Trials

In addition to a faster heat-up rate, the dynamic conditions inside a rotary kiln are very different to those in a static laboratory furnace. One early concern with ACP was that the pellets would not survive the aggressive mechanical tumbling and devolatilization in a full-scale kiln. To get more confidence on this before, several batches of ACP were calcined in a small pilot scale rotary calciner. For these trials, a blend with 75 % regular GPC and 25 % ACP was used. The kiln was operated in a batch mode with a continuously rotating drum and moving coke bed. A large gas-fired burner was able to achieve similar heat-up rates to a full-scale kiln. A photograph of the equipment used is shown in Figure 2. Previous studies with this kiln [10] had shown comparable VBD results to a full-scale kiln which gave confidence in the method.

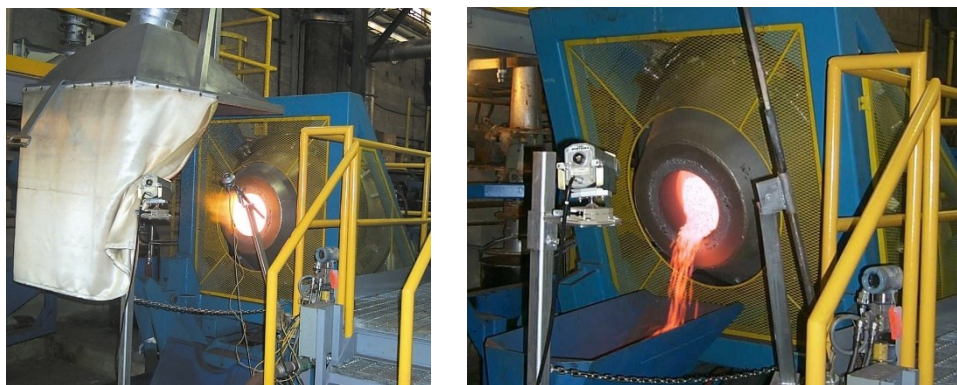


Figure 2. Pilot Scale Batch Rotary Kiln (COREM, Quebec City, Canada)

The pellets calcined in the above kiln survived well and retained their original spherical shape. The bulk density of the blend containing 25% ACP was similar to a batch of GPC calcined in the same furnace under the same conditions. The key finding from the test was that the pellets were not destroyed from the rapid VM loss and tumbling in the kiln. This provided the confidence to produce much larger batches of pellets (requiring significantly more effort) for calcination trials in a full-scale rotary kiln.

5. Full Scale Rotary Kiln Trials

Several 4-10 ton batches of ACP from different green cokes sized to -1.4mm were produced and calcined in a full scale rotary kiln at the Rain Carbon Lake Charles calcining plant in Louisiana. The two kilns at this facility normally operate with a feed rate of 32-35 WMT/hr (wet metric tons per hour) so a method was developed to break the GPC feed stream to add a batch of ACP for calcining. The residence time through the kiln is well known, so it was a matter of waiting for the calcined ACP to exit from the cooler for sample collection. There was co-mingling of CPC at the start and end of the trial but it was quite easy to collect a sample of 100% pellets mid-way through the run.

Every batch of ACP calcined in the full-scale kiln produced good quality CPC product with no significant degradation of the pellets despite the aggressive heat-up rate and tumbling. Results for ACP produced from a low sulfur coke are shown in Table 1. Coke A was a good quality GPC with a VM level of ~11%. The first row shows VBD results for the regular, non-pelletized version of Coke A. The second row shows VBD results measured on CPC pellets (ACP) produced from Coke A. For comparative purposes, a baseline rotary kiln blend is also included. Rain Carbon has been using this blend since 2013 for pilot anode studies and it is used a reference CPC blend for studies with different CPC, coal tar pitches, petroleum pitches etc.

Table 1. Bulk Density Results from Full Scale Calcination Trial

CPC Sample	CPC Sulfur %	Vibrated Bulk Density g/cc		Naturally Occuring Fractions Bulk Density g/cc			
		0.3-0.6mm	1.2-2.4mm	4-8mm	2-4mm	1-2mm	0.5-1mm
Regular A	1.8	0.909	0.781	0.627	0.681	0.746	0.813
ACP A	2.0	0.909	0.833	0.868	0.853	0.806	0.829
Baseline BL	2.8	0.901	0.775	0.685	0.722	0.769	0.802

Several different methods were used to measure the CPC bulk densities in Table 1. The 0.3-0.6mm VBD results were measured via the common ASTM D4292 test on a sample crushed to 30x50 Tyler mesh (0.3-0.6mm). The 1.2 – 2.4 mm VBD results were measured on samples crushed to 8x14 Tyler mesh. This method gives good information about macro-porosity in larger coke pieces and has been used by Rain Carbon for more than 40 years to monitor daily CPC bulk

densities. The other four bulk density results were measured on naturally occurring fractions screened from the CPC with no crushing. A trans-axial density analyzer was used to measure the bulk densities on these fractions via the ASTM 8097-17 method [11].

The first point to note in the above, is that the 0.3-0.6mm VBD for Coke A was the same for the regular CPC and the ACP. Since the pellets were produced with -1.4mm GPC fines, much of the starting GPC already falls in the 0.3-0.6mm size range. The fines were agglomerated to produce pellets in the size range of ~2-8mm. To run the VBD test, the CPC pellets are crushed to a size of 0.3-0.6mm. This is why the VBD results from this test show no real change or improvement with pelletizing. Pelletizing GPC does not change the structural form of the starting coke and when the spherical pellets are crushed to a finer size, they produce irregular shaped particles. Any particle packing benefits associated with spherical shape pellets is therefore lost.

The situation is quite different for all other bulk density results measured on the calcined ACP. The 8x14 VBD's results are ~0.05g/cc higher which is a significant improvement. Most of this increase is likely due to the improved packing density with the spherical particle shape but lower porosity may also be a contributor. Not all the particles in the test were spherical since all the +8 mesh material gets crushed to 8x14 mesh for the test. The paper referenced previously [4] discusses the potential benefits of rounding particles through selection of different crushing technologies to improve product bulk densities and this data highlights the positive impact of a spherical particle shape.

The improvement in particle packing density can be seen even more clearly with the bulk densities measured on the naturally occurring fractions. The biggest improvement (+0.24g/cc or ~38%) occurs with the 4-8mm pellets vs the regular CPC screened to 4-8mm. Coarse coke particles typically also have significant open and closed porosity and ACP eliminates some of this porosity. The 2-4mm and 1-2mm uncrushed particles also show a significant improvement relative to irregular shaped particles from the regular CPC product but the magnitude of the improvement decreases as the average particle size decreases. The difference for the finest particle size material (0.5-1mm) is much smaller and within the margin of error for the bulk density test. This is because most of the coke in this size range is non-pelletized given that the starting GPC was sized to -1.4mm.

6. Pilot Anode Results

In this next section, pilot anode results are presented for anodes made with formulations containing calcined ACP made from Coke A in Table 1. For reference, pilot anodes were also produced from the Baseline Coke in Table 1. The pilot anodes were produced according to Rain Carbon's standard pilot anode production methods and aggregate recipe [12] using a 112M softening point pitch. Table 2 includes a summary of the cokes used to make pilot anodes in the study. No butts or baked scrap were used in the anodes to better highlight coke differences,

Table 2. Description of Cokes Used to Make Pilot Anodes

Coke	Chart Label	S %	V ppm	0.3 - 0.6mm VBD g/cc	1.2 - 2.4mm VBD g/cc
Coke A Regular	Coke A	1.79	208	0.909	0.781
Coke A ACP	APC A	1.98	237	0.909	0.833
Baseline	BL	2.78	320	0.901	0.775
Baseline + ACP	BL + ACP A	2.34	274	-	-

Selected pilot anode results for the cokes described in Table 2 are shown in Figure 3.

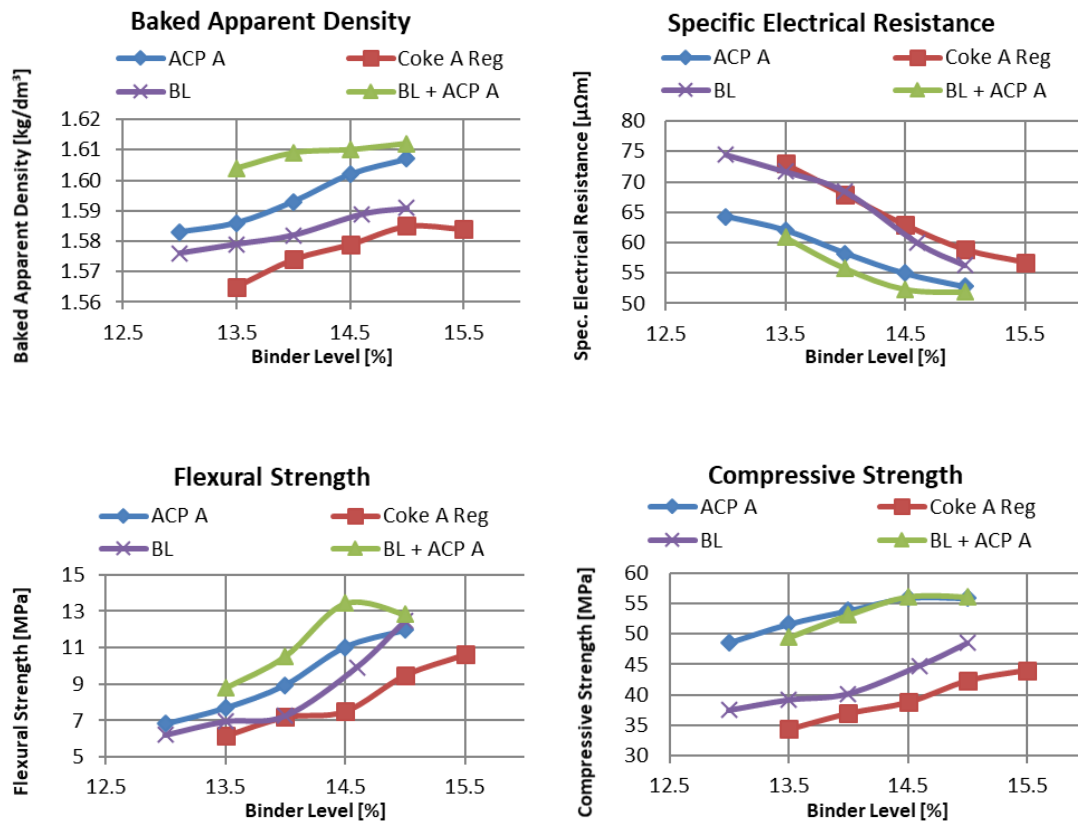


Figure 3. Pilot Anode Results for Cokes Shown in Table 1

The first point to note in the charts above, is the significantly higher baked anode density of the anodes made with calcined ACP produced from Coke A vs. the anodes made with regular Coke A CPC (roughly +0.02 g/cc). The specific electrical resistivity was also significantly lower for these anodes and the flexural and compressive strengths were higher.

The baked density of the anodes made with the baseline coke blend was slightly higher than the anodes made with the regular Coke A CPC but lower than the anodes made with 100% ACP. For the anodes labeled BL + ACP, the two coarser fractions of the baseline aggregate (1.7 - 4.75mm and 4.75 - 8mm) were replaced with ACP. Based on the standard Rain aggregate recipe [12], this meant that 55% of the coke in the 1.7mm to 8mm size range was replaced with ACP. The goal here was to replace the coarser aggregate with lower porosity and spherical ACP. These anodes had the highest baked anode density in this study confirming the particle shape and porosity benefits of using the ACP in the coarser aggregate fractions.

When ACP is used in all aggregate fractions, the finer aggregate fractions have to be produced by crushing and milling the ACP. The particle shape and porosity benefits of using ACP in these fractions is therefore lost, particularly in the ball mill fines fraction and any aggregate fractions <~1mm in size. If it were possible to use ACP in a targeted way to produce anodes, it would be better in principal to use ACP in the coarser aggregate fractions to take advantage of the higher packing density and lower macro-porosity. Milled, irregular shaped CPC would then be used in the finer particle size fractions to fill the void space between the larger spherical calcined ACP.

7. Comparison of Briquetting and Pelletizing

Rain Carbon has focused most of its agglomeration efforts on pelletizing but some work has also been done on testing briquetting as an alternative. Figure 4 shows a photograph of briquettes made

with GPC screened to -2mm and the same briquettes after calcination (in a static lab kiln). Several different binders were tested in this work and the performance varies significantly. Binders that work well in pelletizing do not necessarily work well in briquetting. Compared to APC, the briquettes were more brittle and had significantly lower calcined crush strengths. In each crush strength test, 20 pellets or briquettes were tested to determine the load in kg that the pellet or briquette could support before collapsing. Since the pellets are physically much smaller than briquettes, an equivalent load in kg represents a significantly higher pressure.



Figure 4. Green and calcined briquettes

Table 3 includes some comparative data for briquettes and ACP. The results for Coke B represent a direct comparison between briquetting and pelletizing for the same coke source. Since briquettes are all produced to the same final size and shape, it was not possible to measure bulk densities on naturally occurring particle size fractions. The only bulk densities measured were those on briquettes crushed after calcination. In general, the calcined ACP has significantly higher strength and 8x14 VBD's compared to calcined briquettes. The 30x50 mesh VBD's were similar since the CPC is crushed to a size comparable to the starting GPC size.

Table 3. Crush Strength and Bulk Density Results with Briquettes and APC

Sample Description	Crush Strength		CPC Bulk Density	
	Green Crush Strength (kg)	Calcined Crush Strength (kg)	0.3-0.6mm VBD g/cc	1.2-2.4mm VBD g/cc
Briquette Batch 1 Coke A	7.7	5.0	0.893	0.752
Briquette Batch 2 Coke B	7.8	7.5	0.847	0.769
Briquette Batch 3 Coke C	3.5	4.2	0.909	0.752
Pellet Batch 1 Coke B	11.8	24.1	0.893	0.870
Pellets Batch 2 Coke D	6.4	23.1	0.943	0.893
Pellets Batch 3 Coke D	13.2	24.1	0.926	0.893

It should be possible to increase the briquette strength by optimizing the binder content and additional work is planned in this area. Batches of briquettes will also be calcined in a pilot scale rotary kiln calciner to see how well they stand up to the faster heating and devolatilization rates and mechanical tumbling. Briquetting is a much simpler processing technology than pelletizing so it is attractive from this perspective. One significant downside however, is the non-spherical shape of briquettes. The shape selected for the Rain Carbon trials was as close to spherical as practical but did not give the same particle packing benefits as spherical pellets.

8. Pelletizing Process Technology

Rain Carbon has invested significant time and effort into developing a pelletizing process that produces high quality ACP. There are a number of different pelletizing technologies that can be used and it's a matter of selecting the one which gives the best combination of processing simplicity, productivity and product quality. Binder selection is critical for the application and a tradeoff between performance and cost has to be accepted. For anode grade coke applications, a binder with very low trace metal impurities must be used but some of these can be expensive. The binder level also has a direct impact on cost so it's a matter of using just enough binder to give the pellet sufficient green strength for handling after drying. No further details on the pellet production process will be provided in this paper for proprietary reasons but Rain Carbon has now developed a commercially robust process.

The most cost-effective way to incorporate ACP production into CPC production is to include it as a part of a calciner flowsheet. GPC being fed to the calciner is first screened to produce a coarse and fines fraction and the fines fraction is pelletized to produce ACP. The ACP product stream and coarse coke fraction are then recombined and fed to the kiln. Depending on the feed particle size cut point and GPC particle size distribution, the percentage of ACP in the final CPC product can be anywhere in the range of 25-50%. This can be controlled to achieve a specific target with a well-designed flowsheet. Using this approach, the incremental cost of making ACP can be minimized which improves the overall process economics.

9. Discussion on Benefits of ACP Technology

The starting objective for agglomerating GPC fines was to improve the economics of calcining by reducing fines carryover and improving the yield of the process. Another benefit mentioned in the patent applications relates to eliminating the problem that GPC fines creates in a shaft calciner. Since very little fine particle size GPC is lost in a shaft calciner, it stays with the product. It can create a significant dusting problem when the CPC product is handled since the fines are only loosely attached to the surface of coarser CPC. This problem is described in more detail in [2] and can be completely eliminated if the fines are used to produce ACP.

During the development of ACP technology, it has become clear that there are several other benefits. With the right processing technology, it is possible to make high density ACP which then produces a high bulk density CPC product. The high bulk density is driven primarily by the improved particle shape (spherical particle size) which increases particle packing density. For larger particles, ACP also has a lower overall porosity compared to regular, larger CPC particles.

The pilot anode results summarized earlier, demonstrate that the bulk density benefits of ACP translate into measurable improvements in anode quality. The results generated by adding calcined ACP to the coarse fraction of a regular rotary kiln coke (BL + ACP) in Figure 3 are particularly interesting. The starting GPC for the ACP had the same 30x50 mesh ASTM D4292 as the baseline CPC, but the improvement in replacing the +1.4mm BL coke fractions with ACP is clear.

Of particular note is the flatter response for the baked anode density as a function of pitch level. The specific electrical resistivities for the anodes made with this coke combination were also low ($\sim 52\mu\Omega\cdot\text{m}$) for hydraulically pressed anodes. The compressive and flexural strengths were also good. This suggests that using a backbone of spherical CPC particles to make an anode can have some significant quality benefits.

Another advantage of ACP is the ability to blend GPC on a micro-scale to produce a uniform CPC product. Today, coke calciners typically blend different quality GPC together to produce a blended CPC product. Although such blends normally perform well in anodes, they consist of discrete particles with different sulfur, vanadium and other impurity levels along with different coke bulk densities and reactivities. If GPC fines are blended together first to make ACP, the potential exists to make a very uniform CPC product with no difference in properties between different particles within the blend. This would reduce the risk of a softer, higher reactivity coke ending up preferentially in the ball mill fines fraction. This can lead to higher anode reactivities as shown in previous work [12,13]. It would also eliminate a problem with shaft CPC where higher reactivity fines on the surface of coarse CPC particles end up in the ball mills fines fraction and increase anode reactivity.

The reduction in GPC fines carryover in a rotary kiln through ACP production gives a direct benefit to the calciner since it results in lower SO₂ emissions. Taking the example of a rotary kiln calciner operating with a federate of 50 WMT/hr with a GPC blend having 3% sulfur, the reduction in SO₂ emissions is around 200 kg/hr. For calciners operating with SO₂ scrubbers, this gives a reduction in SO₂ scrubber operating costs which can include the reagent cost (like lime) and disposal cost assuming there is no byproduct application for the scrubber residue. For older calciners which do not operate with SO₂ scrubbers, the reduction in SO₂ emissions gives an immediate environmental benefit.

At this stage, Rain Carbon has focused only on producing ACP from GPC fines generated through screening of GPC. This represents the lowest cost route to producing ACP but in principal, coarser particle size GPC can also be milled to produce a feedstock for making ACP. Whilst it might seem counter-intuitive to mill coarse material and then spend money to re-agglomerate, there may be some technical benefits in this approach if a product with a wider particle size distribution of spherical particles can be produced. An improvement in particle packing density could in principal lead to a change in aggregate granulometry. If intra-particle void space can be reduced through more efficient packing of solid spheres, the ball mill fines requirement and binder requirement may decrease. No work has been done on this so it is speculative, but it will be investigated in further studies.

Today, all CPC is produced from refinery byproduct GPC with virtually no processing. The only work a calciner does is to crush the incoming GPC to a size of -75mm or -50mm for some shaft

calciners. This does not change the coke in any material way. Producing ACP on the other hand, would be a big step towards producing a fully engineered CPC product. The combination of micro-blending with different green cokes and the production of a high bulk density, spherical product can give some significant potential benefits for anode production and performance. CPC made from ACP is also a free-flowing material which can mitigate some of the problems that occur with CPC related to silo segregation effects.

If ACP was adopted as the primary CPC production feedstock, it would be necessary to broaden the particle size distribution to include spherical particles in the size range of 0-2mm. Without a broader size distribution, the poured bulk density or stowage factor of the CPC product would be lower. A CPC product based entirely on 2-10 mm pellets for example, would require more silo storage volume for a given mass than existing CPC material with fines. It is not difficult in principal to make finer particle size pellets but this would require processing changes which would add additional cost. Producing a product with a broader particle size distribution would also reduce the need to crush large amounts of calcined ACP to produce the finer portions of the aggregate recipe.

10. Conclusions

The results presented in this paper show that it is possible produce a better performing CPC product through the agglomeration of GPC fines to make ACP. This will add some additional cost during calcination but the cost can be at least partially offset by reduced fines carryover and higher CPC production per ton of GPC. At higher GPC prices, the economic benefit of making APC increases. The spherical shape of APC gives particle packing benefits which translates together with reduced porosity into an improvement in CPC bulk density and anode properties like baked anode density, electrical resistivity, flexural strength and compressive strength. Production of ACP opens the door for a more engineered approach to producing CPC including the potential to produce a uniform, micro-blended ACP product where all CPC pellets properties are identical. The technology would also eliminate the fines/dusting problem that occurs with shaft CPC. Moreover, ACP also results in a direct benefit to the calciner in reducing SO₂ production.

Rain Carbon is now embarking on a project to build a commercial scale plant in the US to make ACP. Such a plant will allow a full evaluation of the benefits of this new technology for both CPC and anode production.

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