

## ANODE AGGREGATE BULK DENSITY DETERMINATIONS USING A Y-BLENDER

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### Abstract

Optimizing the composition of the calcined coke aggregate blend used in anode manufacture is important from several aspects, including production of high density anodes, reducing binder pitch requirement, and/or minimizing problems such as cracking or dusting. Plots of aggregate density as a function of composition have been experimentally determined for a number of carbon plants using the Y-blender method. Important experimental considerations will be discussed and representative results given.

### Introduction

There are two potential advantages to converting from an anode aggregate composition having a low-to-medium bulk density to one having a higher bulk density [1, 2, 3]. A higher aggregate bulk density provides the potential for an increase in anode baked density, which could result in increased anode campaign life as well as improvements in other performance criteria. Additionally, a higher bulk density generally provides the opportunity for use of a reduced amount of binder pitch, which is the most expensive component in terms of absolute cost, and increasingly more expensive when a pitch coke yield of only about 70 % is factored into the cost equation.

Probably all anode-producing operations have investigated aggregate bulk density as a function of particle size distribution at some time or another, and many may continue to do so on a regular basis. However, at some locations the use of different cokes and/or new green mill equipment or operating conditions of existing equipment, have rendered past results of historical significance only, and up-to-date measurements have not been made. This has provided the opportunity to determine composition-density relationships for clients, using the Y-blender technique.

This paper reviews some of the key factors affecting bulk densities of calcined petroleum coke aggregates, discusses the equipment and techniques utilized in aggregate bulk density determinations, and gives examples of selected determinations.

### Calcined Coke Aggregate Density Considerations

There are several considerations involving bulk densities of packed particles. One consideration is how the particles fit together to affect inter-particle void space. When all particles have identical densities, the maximum density is achieved by the particle size distribution that minimizes the amount of inter-particle void space. With spherical particles for which particles of all sizes have identical densities, aggregate density as a function of particle size distribution can be predicted. Complications arise when (a) the particles are irregularly shaped and (b) particle

density differs according to the particle size. Both of these situations are inherent to calcined petroleum coke aggregates.

Although highly isotropic shot coke particles can be more-or-less spherical in shape, even these cokes do not lend themselves to calculations based on spheres, since coarser particles may be fused agglomerates of smaller spheres and the finest particles are usually fractured sections of spheres. At the other extreme, highly anisotropic needle cokes tend to fracture into elongated particles, which may increase the difficulty of modeling.

For the most part, calcined petroleum cokes used in anode manufacture are somewhere in between these two extremes, and density as a function of particle size distribution is difficult to predict with sufficient accuracy to be a good indicator of high-density particle size distributions.

Another complication in successfully predicting calcined petroleum coke aggregate bulk densities lies in the fact that coarse coke particles are more porous than smaller coke particles, since larger pores are successively removed during the comminution process. The effect can be small with highly isotropic cokes, but very significant with cokes more commonly used in anode manufacture. In the latter case, it is obvious that the largest pores will be annihilated during comminution. The photomicrographs in reference [4] provide good visual illustrations relevant to the above discussion.

Due to these factors, experimental determinations, rather than predictive methods, are commonly used to determine relationships of anode aggregate particle size distribution and bulk density. Although small scale density testing in glassware is convenient for a laboratory environment and can give satisfactory results [1], there are several limitations. For example, there can be a difficulty in acquiring representative and reproducible aggregate fraction samples due to the small test sample size. In addition, it is normally necessary to scalp off the largest butt particles to avoid bridging in the test glassware, so that there is an inherent, but unproven, assumption that the absence of these largest particles does not affect the relative results.

To avoid these difficulties a larger scale testing device, the "Y-blender" (named for the shape of the apparatus) was developed by R.W. Peterson [5]. This device requires a much larger sample size and is not ideal for a laboratory environment, but it eliminates the problems outlined above. This report gives insights into more recent Y-blender density determinations by the current author.

### General Comments on the Bulk Density Determinations

The Y-blender aggregate bulk density determinations exemplified herein will not be attributed to specific carbon plant operations. In general, though, it can be mentioned that clients have included

aluminum producers based in Asia, Australia, North America, and South America.

Most investigations involved butts-containing aggregates for prebaked anodes although several determinations have been made for Soderberg operations. Complexities have ranged from examining compositions utilizing a butts fraction and only two coke fractions to compositions involving five varying fractions.

Results are discussed and plotted in terms of actual fractions used in the determinations. For a more complete understanding of the relationships involved, compositions in terms of the more closely sized fractions typically used in characterizing particle size distribution in aggregates (e.g. +4, 4x8, 8x14, etc., where the numbers refer to Tyler series mesh sizes) is required. To achieve density improvements, it may be necessary to alter grinding and sieving equipment to change the compositions of the fractions. In the majority of cases, the author did not participate in subsequent detailed use of the density determinations. In cases where the author was involved, results are not included herein.

There have been several variations in conducting the Y-blender determinations. Approximately three-fourths of the investigations have been conducted at the author's location, i.e. coke and butts were shipped from green mills. Other determinations have been carried out at green mill facilities. In the latter case, a Y-blender was transported to the facility or was constructed or already possessed by the client. Several contracts have included provisions for training personnel in the techniques involved in the determinations, as well.

### Typical Y-Blender Construction and Operation

Peterson [5] showed a schematic diagram of a Y-blender design. However, with respect to size and detailed design specifics there is no standard Y-blender. For most of the density determinations by the present author, internal diameter of the device was 10 cm, the straight section was 61 cm long and each of the two legs was 13 cm long. In a departure from Peterson's design, in which bed height was determined using a closed cylinder having height markings inserted into the long tube of the device after blending, this Y-blender used a cylindrical plate welded onto a steel rod attached at the center of one face. After filling, sealing, rotating, and opening the blender, a rod-centering device was used at the open end to keep the plate horizontally positioned at the top of the bed. Distance between the top of the plate and the top of the blender was measured with a ruler. The height of the aggregate bed was then determined by subtracting the thickness of the plate.

### General Experimental Details

Several parameters were fixed, based on work conducted using the Y-blender version described above or other Y-blenders. Details are given below for this specific Y-blender, and differed somewhat for other versions.

It is useful from a material handling standpoint and, more importantly, from a blending standpoint to use as small a charge size as practical while ensuring that with a dense aggregate the level will not fall below the long section of the Y. Based on experience of typical densities of coke aggregates, a charge of 4000 g (8.8 lbs) was used.

For a fixed composition, rotations were conducted in increments of 50 and density was determined after each increment. Density increased for up to 150 rotations, so this number of rotations was selected as standard.

Absolute Y-blender bulk density values are dependent upon the apparatus and procedure. A precise blender volume calibration was not attempted, since relative densities of compositions are adequate. However, it was deemed useful to at least approximate realistic bulk density values, so a rough calibration was made using a material of known density.

One change from Peterson's procedure for selection of trial compositions was made for all but the first few determinations. For a three-fraction aggregate, for example, he started with an arbitrary composition of only two of the fractions. After determining density, 10 % of the material was removed and replaced with an equal quantity of the third fraction. This process was continued multiple times, with the aggregate composition linearly moving toward the pure third fraction. A second and a third series of compositions, beginning with a blend of the other two combinations of fractions, completed the overall determination. The reason for this procedure was to reduce material shipping and handling, relative to using new aggregate blends for each determination.

This procedure has two shortcomings. In removing material each time, there is no guarantee that a representative sample is withdrawn. Hence, after the replacement process, the actual composition might not be the same as calculated. The error can become larger with each removal/replacement sequence. This problem was demonstrated by comparing composition and density of an aggregate after one of these removal/replacement series with a newly prepared aggregate having the calculated final composition. Results for the two types of aggregates differed. Another shortcoming is that there are appreciable data gaps, necessitating considerable interpolation in analyzing results.

It is preferred to plan and use a systematic grid of compositions, which ensures that the compositions are correct and that there are no gaps in regions of interest. With the Y-blender design described above, about 180 kg (400 lbs) of material was required for a bulk density vs. aggregate composition determination, compared with 90 kg (200 lbs) for the procedure used earlier.

### Representative Results

#### Example 1

This example involves aggregate to be used in prebaked anode production. Butts content was held constant at 20 % and three calcined coke fractions were used. Figure 1 is a ternary diagram based on 25 density determinations. (Note that in all the ternary diagrams to be shown, total of the three coke fractions is normalized to 1.0) Due to both experimental variability and the "connect-the-dots" nature of the plotting program, the plot does not provide a clear picture of the relationship between composition and density. Although the plot shows a small high-density area, it is based on a single composition. The difference in density between this composition and several others is not statistically significant based on a standard deviation calculation. Also, this apparent maximum is far from centered within the next density contour, which seems unrealistic.

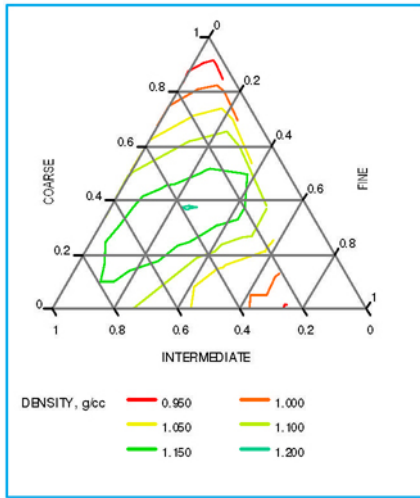


Figure 1. Ternary plot of aggregate bulk density based on 25 experimental points.

Because of this less-than-satisfactory result, a mathematical model was used both to smooth out normal experimental variability and to provide additional predicted densities to enhance the plot. A model of the form given in Equation 1 was used. A through F are constants derived from the experimental data values.

$$\text{Density} = A \times \text{coarse} + B \times \text{intermediate} + C \times \text{fine} + D \times \text{coarse} \times \text{intermediate} + E \times \text{coarse} \times \text{fine} + F \times \text{intermediate} \times \text{fine} \quad (1)$$

Figure 2 is a ternary diagram using 200 values predicted from the model. Resolution is much improved. The oval-shaped highest density contour plotted, with the long axis roughly aligned along the fines-content axis was typical of the majority of determinations for different carbon plants, with the contour more elongated for some locations, as will be the case in the next example.

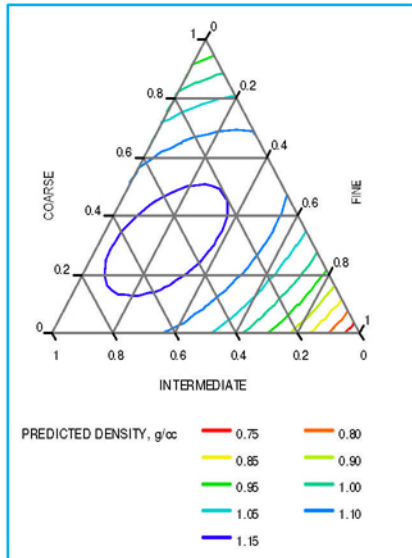


Figure 2. Ternary plot of aggregate bulk density based on 200 values predicted from a mathematical model.

## Example 2

Figure 3 shows the result for a determination for another carbon plant using one coke source. At a later time, the client changed coke source and contracted for a second Y-blender density determination. Result for just the highest iso-density contour for the new coke is shown in Figure 4.

Highest density values for the new coke averaged approximately 0.03 g/cc higher than those with the previously-used coke. In addition, the position of the maximum density region shifted considerably. With the 22 % butts included, coarse has increased by 4 %, intermediates have remained constant, and fines have decreased by 4 %. Also, the density is more sensitive to changes in the fine fraction, as evidenced by the elongation of the density contour roughly along the fine-fraction axis. Without more detailed information (not available to the author) it is not known whether these changes were due to a difference in porosity distribution, particle shape (which could alter packing efficiency), grinding or milling characteristics (which could affect particle sizes within the fractions), or a combination of these factors.

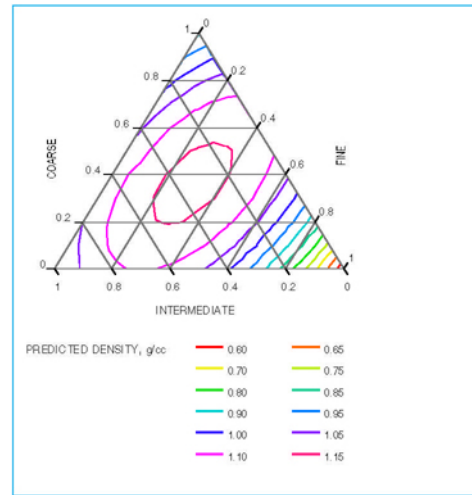


Figure 3. Ternary plot of aggregate bulk density based on 100 values predicted from a mathematical model.

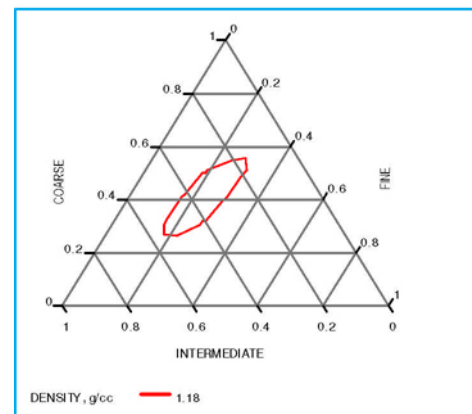


Figure 4. Ternary plot of the highest density contour with a new coke based on 100 values predicted from a mathematical model.

**Example 3**

This determination for another carbon plant used several butts levels (18, 22, and 25 %) and three coke fractions. At least twenty experimental values were determined at each butts level and mathematical models of the type shown earlier were generated.

Figures 5-7 show the ternary diagrams using 100 predicted values for compositions containing 18 %, 22 %, and 25 % butts, respectively. As in the previous examples, density drops off most steeply with fines content, as illustrated by the considerable elongation of the highest-density contour roughly along the fine-fraction axis.

With an increase in the butts fraction, there is also an increase in intermediate material apart from the amount present from the intermediate fraction itself. At the 22 % butts level, the highest density contour has shifted considerably due to this factor, and at the 25 % butts level there is enough excess intermediate material to truncate the highest-density contour. It can be speculated that at an even higher butts level, it might not be possible to produce as high a density as achievable at lower butts levels.

The particle size distribution within the butts fraction is thus shown to have a considerable effect on overall aggregate particle size distribution. More generally, this can apply to coke fractions as well. Excessive overlap in particle size ranges among fractions can affect the ability to produce the highest density aggregate possible from a given coke.

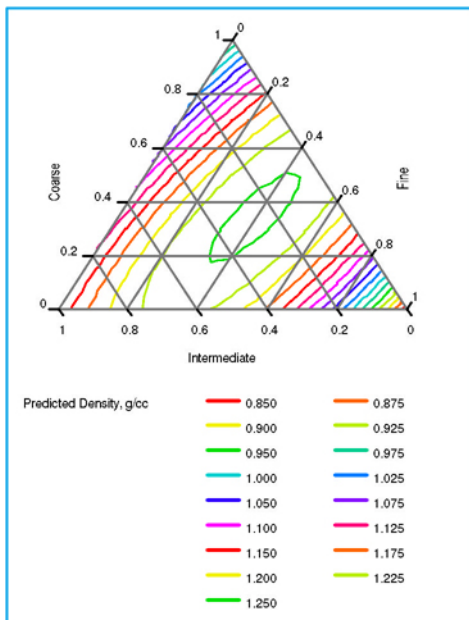


Figure 5. Ternary plot of aggregate bulk density with 18 % butts based on 100 values predicted from a mathematical model.

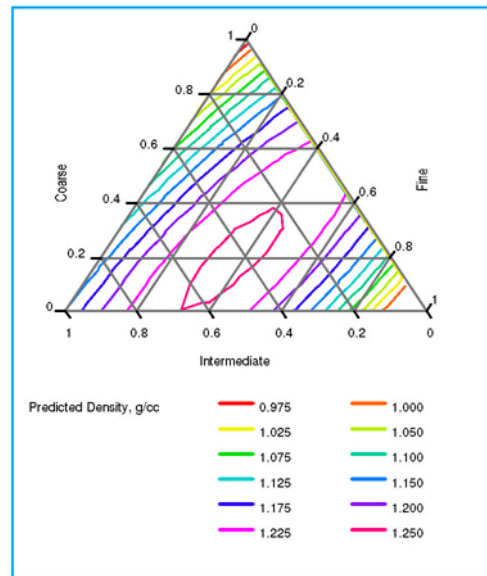


Figure 6. Ternary plot of aggregate bulk density with 22 % butts based on 100 values predicted from a mathematical model.

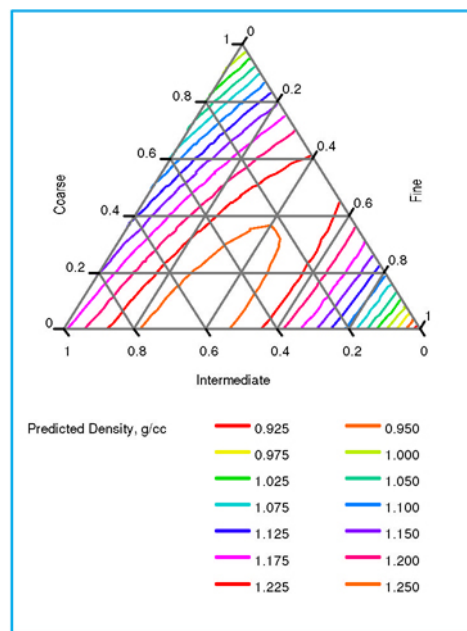


Figure 7. Ternary plot of aggregate bulk density with 25 % butts based on 100 values predicted from a mathematical model.

**Example 4**

The last example involves a Soderberg anode plant which uses four variable coke fractions in paste formation. Although the same approach as in Example 3 (selecting several arbitrary levels of one of the fractions and determining densities when varying the other fractions) could have been used, it was decided to use a four-factor designed experiment since there are no set levels for any of the fractions. With typical maximum and minimum levels for each fraction incorporated into the design, a 39-point experimental grid

was generated using a statistics program and density values were determined. A mathematical model of the form in Equation 2 was used, with letters A through J representing constants determined from the model equation and the experimental density values. F1 through F4 represent fractions used, in increasing order of fineness.

$$\begin{aligned} \text{Density} = & A \times F1 + B \times F2 + C \times F3 + D \times F4 \\ & + E \times F1 \times F2 + F \times F1 \times F3 + G \times F1 \times F4 \\ & + H \times F2 \times F3 + I \times F2 \times F4 + J \times F3 \times F4 \end{aligned} \quad (2)$$

After data analysis, densities were determined for nine more compositions near the highest density region in an attempt to improve resolution. Ternary plots can be calculated from the predicted density values by varying amounts of three of the fractions at any level of the fourth fraction but are not shown for this example.

Figure 8 compares predicted density values with the experimental values. Agreement is reasonable, but might improve if retesting of a few of the compositions resulting in the largest deviations were found to change the experimental value significantly.

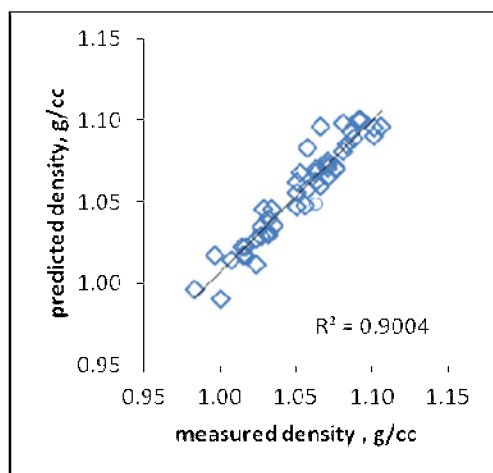


Figure 8. Agreement between experimental values and predicted values based on a designed experiment and a mathematical model.

### Summary and Discussion

Numerous Y-blender anode aggregate bulk density determinations have been carried out, with several of them included as examples in this report.

Although the experimental density values provided satisfactory results in some cases, use of values generated by simple mathematical models provided improved resolution, both by eliminating the effect of random experimental variability and by providing more values for plotting purposes.

In all experimental determinations, high density compositional regions were successfully found. In no case was there an indication of a sharp maximum peak region. Rather, there was a relatively large compositional region in which densities did not differ appreciably.

In most cases, the fine fraction content had the largest effect on density. It can be speculated that packing of the larger particles establishes the basic aggregate structure and the fine fraction "fills in the holes." Too little fines and the holes are not totally filled in. Too many fines and the efficient packing of the larger particles is disrupted.

For analyses using only three variable fractions, intuitive planning of the experimental test compositions was satisfactory, with about 25 compositions sufficient for a good analysis. With a greater number of variable fractions, a computer-generated designed experiment may be preferable.

While not a direct conclusion from this report, it seems reasonable to assume that carbon plants with relatively poor control should operate at a composition near the center of the maximum density region to decrease the likelihood of producing low density compositions at times.

In contrast, plants with good control can select a composition closer to the edge of the high density region, sacrificing a little density, but (if shown by experimentation) possibly improving some important anode properties and/or reducing binder pitch requirement

### Acknowledgement

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