

68-8 "APPLANT" CALCULATION - EDGE MOOR KILN NP-103-D CAMPAIGN, MAY 20-24, 1968 By: B. W. Oetnar 5/20/68 - 5/24/68

1/79 (1)

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Serial No. KN-68-8

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SUBJECT: AFFLAIR® CALCINATION---EDGE MOOR KILN
NF-103-D CAMPAIGN, MAY 20-24, 1968

PERIOD COVERED: May 20-24, 1968

Date: 9/30/68

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SUBMITTED BY: B. W. CETNAR

DATE SUBMITTED: 9/25/68

APPROVED BY:

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DATE RELEASED: 9/30/68

Abstract

The report summarizes an Afflair® NF-103-D calcination campaign at Edge Moor. Operating data and measurements are reported along with the results of a material balance to determine losses and calcination yield.

AFFLAIR® CALCINATION--EDGE MOOR KILN
NF-103-D CAMPAIGN, MAY 20-24, 1968

A task force consisting of Newport research and quality control, and Edge Moor production supervision was assigned to follow the May 1968 NF-103-D campaign at Edge Moor. The objectives of the task force were twofold: 1) produce Afflair® quality equal to standard; 2) calculate a material balance based on current kiln operation. The task force met these objectives.

I. SUMMARY

The presscake from six hydrolysis batches, 3-72 through 3-77, was calcined during the campaign. While some minor color variations were noted, all of the kiln product met the NF-103-D quality standard.

The kiln feed hopper was charged with 140 lbs. of presscake at 15 minute intervals or 560 lbs. per hour. The average product discharge rate was 287 lbs. per hour.

The kiln temperature at the 1-foot thermocouple* was used as the control temperature. A setting of 1020°C. produced the best Afflair® quality, except for batch 3-77 in which 1010°C. was determined as the optimum control temperature. At the 1020°C. setting, the temperature indicated by the 1-foot thermocouple cycled between 1000°C. and 1020°C. with an occasional peak of 1030°C.

Five drums of kiln discharge were recycled when the quality evaluation indicated the material was undercalcined. Four of the drums were undercalcined as the result of a flame-out in the kiln. The cause for the fifth drum could not be determined.

The total presscake charged to the kiln was 48,121 lbs. The moisture content was approximately 39%. Ignition losses due to sulfate removal and dehydroxylation amounted to 9.1% based on dry presscake. The calculated theoretical yield was 26,683 lbs. Product drummed amounted to 22,888 lbs. Losses were 1720 lbs. The overall accountability was 92.2%. The product yield, i.e. product weight collected divided by the calcined product to account for, was 85.8%.

A. Afflair® Quality

The calcined product was collected in 201 drums. The net packed weight was 22,888 lbs. The average product weight in each drum was 114 lbs. Each drum was sampled three times while filling.

* One foot from the discharge chute.

A. Afflair® Quality (Contd)

Each sample was checked for Afflair® quality. The method employed consisted of brush screening approximately 5 grams of sample on a 140-mesh screen. A 0.75 gram portion of the minus fraction was then dispersed in 10 grams of acrylic lacquer and the mix drawn down against the NF-103-D standard.

The drawdown was evaluated against the NF-103-D standard for color and against a previously prepared laboratory calcination ladder series for correlation of color to kiln temperature. A temperature rating was assigned to each sample. Thus, a rating of 970°C. indicated that the color developed in the kiln for the sample in question was equivalent to the 970°C. drawdown in the ladder series. This assigned temperature was used as the basis for selecting the kiln operating temperature. For the kiln discharge to agree with the laboratory calcined product it was necessary to operate the kiln at 1020°C. measured one foot from the discharge end. At this temperature the kiln product was equivalent to the laboratory product calcined at 970°C.

Generally, there was color variability from drum to drum even though the kiln temperature control setting was maintained constant. The color differences, however, were slight but discernible.

All but five drums were acceptable NF-103-D quality. The five drums were undercalcined and were returned to the kiln. The undercalcination resulted from a flameout in the kiln when a welder's cable struck the control panel which upset a mercury switch in the kiln instrumentation.

B. Process Observations

1. Kiln Temperature--To develop satisfactory color in the kiln discharge it was necessary to operate the kiln at a temperature of 1020°C. at the 1-foot thermocouple. At this setting the temperature cycled within the kiln between 1000°C. to a maximum of 1030°C., depending upon whether the thermowell was in the kiln bed or out of it as the kiln rotated.

B. Process Observations (Contd)

2. Feed Rate--The presscake charged to the feed hopper was 140 lbs. per 15 minutes. The charging was not uniform over the 15-minute interval but was completed in a period of approximately 5 minutes. The remaining time until the next charge was used by the operator to perform other duties.

The feed to the kiln appeared to enter at a non-uniform rate. This was suggested by the depression in the temperature measured in the stack at the feed end. The depression occurred at the start of each 15-minute feed addition followed by a gradual temperature recovery before the next feed increment was made.

A screw feeder was used. It was operated at constant speed since it was not equipped with a variable-speed drive.

3. Feed Moisture--Five samples from five different drums were analyzed for percent solids in the presscake. The average for the five samples was 61% solids, however, the range was from 53% to 66%. This contributed to further nonuniformity in feed rate since the presscake was charged on a gross weight basis with no allowance for moisture content.
4. Kiln Discharge Rate--The discharge rate from the kiln averaged 287 lbs. per hour. The rate, however, was nonuniform ranging generally from about 190 lbs. per hour to 460 lbs. per hour. This wide variation contributed to the drum-to-drum color difference.

It was observed that the bed depth in the kiln varied during the course of the run. When the same location in the kiln interior was viewed at periodic intervals a difference in bed depth was noted. At times the difference was slight while at other times it was appreciable. No pattern was found that would allow prediction of when a buildup would occur. However, it is suspected that the major contributor was the nonuniform feed rate.

5. Stack Discharge--Dust released to the atmosphere from the stack averaged 3.98 lbs. per hour or 370 lbs. total for the campaign. Dust recovered by the dust collector was recycled continuously to the feed hopper.

B. Process Observations (Contd)

6. Breechings

Once each shift the kiln breechings were unloaded from the breechings' collection chamber. This was done by raking the breechings into a hopper that discharged into a drum on a lower floor. While raking the breechings a significant amount was lost through spillage and entrainment in the air within the building.

The breechings that were recovered were checked for calcined quality and were found to be undercalcined. Normally, breechings are returned to the kiln; however, they were withheld during the campaign. In retrospect the decision to withhold them was arbitrary and after evaluating the campaign it was concluded that recycling the breechings would not contribute to any detectable quality variation and, therefore, they should be returned to the kiln.

7. Material Balance

A material balance for the kiln operation is detailed below:

- a. Presscake charged to kiln: 48,121 lbs.
- b. Percent solids in presscake: 61% (average of five determinations)
- c. Solids in presscake: $(48,121)(0.61) = 29,354$ lbs.
- d. Weight loss in calcination: 9.1%
- e. Calcined product to account for:
 $(29,354)(.909) = 26,683$ lbs.
- f. Product drummed: 22,888 lbs.
- g. Losses:

1) Breechings	1,207 lbs.*
2) Stack	370
3) Kiln holdup	123
4) Dust Collector	10
5) Feeder Spillage	10
	<u>1,720 lbs.</u>

* 907 Lbs. collected in drums and weighed plus an estimated 300 lbs. due to spillage and entrainment into the building room air.

- h. Kiln discharge accounted for:

22,888 lbs.
<u>1,720</u>
24,608 lbs.
- i. Kiln discharge unaccounted for:

26,683 lbs.
<u>-24,608</u>
2,075 lbs.

B. Process Observations (Contd)

7. Material Balance (Contd)

j. Percent accounted for: $\frac{24,608}{26,683} \times 100 = 92.2\%$

k. Breechings loss as percent: $\frac{1,207}{26,683} \times 100 = 4.5\%$

l. Stack loss as percent: $\frac{370}{26,683} \times 100 = 1.4\%$

8. Kiln Cleanout

To minimize the turnaround time when changing from one Afflair® grade to another, a new cleanout procedure was attempted. Previously, a complete shutdown was required to clean the interior of the kiln. Since entry into the kiln was necessary, a cooling period was required. The complete changeover normally took approximately 2-1/2 days.

The new procedure called for a reduction in operating temperature from 1020°C. to 600°C., while rolling the kiln until the kiln no longer discharged. At this point, 600 lbs. of flint type gravel about 1/2 to 1-inch in major dimension was introduced at the feed end and allowed to tumble to the discharge end where they were removed. Midway through, the combustion chamber was pulled and the kiln interior was inspected. Only an insignificant quantity of pigment was found. Following reassembly of the combustion chamber, the kiln was rolled an additional three hours for a total time of six hours. At this point the kiln was heated to temperature and a flushout of pearl Afflair® put through before starting the pearl (NF-104-D) presscake feed. The total cleanout time, measured from when the last gold product drum was removed to the start of the pearl presscake feed, was approximately 18 hours.

While the new procedure successfully cleaned the kiln interior, a problem developed in getting all the stones to discharge. It was not until the tenth drum of pearl product had been removed that it was reasonably certain that all of the stone was out of the kiln. Since Newport is not equipped to handle contaminated product, Edge Moor hand screened the 10 drums.

B. Process Observations (Contd)

8. Kiln Cleanout (Contd)

The large reduction in changeover time makes this cleanout attractive; however, further work is indicated to determine the correct stone size, shapes and quantity to use to insure rapid and complete discharge with safeguards to prevent contamination of the Afflair® product.

II. RECOMMENDATIONS

The Afflair® quality obtained during the May 1968 NF-103-D run is a measure of the success of the campaign. The quality was "OK Straight." This can be attributed to the efforts of those involved in the campaign and further emphasizes the high degree of cooperation and attentiveness this product requires for it to meet this demanding quality requirement. For continued successful production the following are recommended:

1. The calcination ladder series furnished by Newport should be standardized to include the following temperatures: 800°C., 900, 925, 950, 975, 1000.
2. Present practice is to compare the plant sample to the NF-103-D shipping standard. Since the shipping standard is finished classified product while the plant sample is only brush screened, there is a particle size distribution difference that makes it difficult to compare the two. It is recommended that an in-process standard be established and used in place of the shipping standard.
3. The pH of the kiln discharge is an important quality specification. Low pH contributes to poor lightfastness. Each drum sample of kiln discharge should be analyzed, and all drums with a pH of 7.0 or lower should be recalcined.
4. Since the kiln is used both as a dryer and calciner, the water variable introduced in the wet presscake feed should be controlled. Wide variations in moisture content in the presscake contributes to nonuniform kiln operation. The filter press procedures should be reviewed to insure that the cake discharged from the presses has a uniform water content both within the press and between the two presses.

II. RECOMMENDATIONS (Contd)

5. Standard product quality can be realized in the kiln by maintaining constant operating conditions. A uniform feed rate is a primary requisite. This was demonstrated during the campaign when emphasis was placed on charging the feed hopper every 15 minutes. In addition, once the kiln temperature was established for a hydro batch, adjustments were made only after careful deliberation. Differences in color between successive drums discharged were considered to be within the normal variability of the process and no changes were made unless subsequent drums continued the trend. It is suggested that in future campaigns this added emphasis on feed rate and temperature control be continued.

III. CONCLUSIONS

The campaign demonstrated that standard quality NF-103-D can be achieved with no changes in current Afflair® technology. However, because of greater sensitivity to process variables than pearl Afflair®, more attentiveness is required to produce this product. An appreciation for maintaining close control of process variables is essential. This appreciation was shown by those involved in the campaign and the end result was a finished product that met the goal of standard quality in every respect.

BWC/mja