MEASUREMENT OF THE RESIDENCE TIME DISTRIBUTION OF FCC CATALYST IN ROTARY KILNS

By

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Continuous rotary kilns are among the most widely used solid handling equipment for industrial applications such as drying, incineration, mixing, pre-heating, calcining, and gas-solid reactions. Longer residence times prove beneficial in ensuring that all particles have been treated sufficiently, but this can lead to large material and energy costs. As a result, efficient calcination will require shorter residence time and lower axial dispersion. The purpose of this research is to contribute towards a better understanding of these mass transfer mechanisms in rotary kilns, using industrially relevant equipment and operating conditions directed towards large-scale catalyst manufacturing. In this work, the residence time distribution and axial dispersion coefficient for a free flowing fluid cracking catalyst (FCC) powder is measured in pilot-scale kilns using a tracer study developed by Danckwerts (1952). The tracer study was used to determine residence time distributions for different sets of operating parameters, which were successfully matched to the Taylor fit of the axial dispersion model and the Sullivan prediction for mean residence time. It was found that the mean residence time and axial dispersion coefficient varied inversely with rotary speed of the kiln. The bed depths corresponding to respective flow regimes in such a calcination system were not previously reported. The predictive models showed accuracy for the system considered, even at fill levels <1% which was not the case in previously reported studies.
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Chapter 1

Introduction

Rotary kilns have been established as extremely valuable devices for catalyst manufacturing industries. Numerous industries utilize rotary kilns to process particulate systems such as the cement, ceramics, chemical, metallurgical and pharmaceutical industries. With applications in a wide range of solids manufacturing processes including blending, drying, and calcining, the rotary kiln has been established as an essential device in chemical and metallurgical industries [1]. Rotary kilns gained popularity in these industrial sectors because they offer special possibilities in the continuous processing of a wide range of materials with little or no labor to operate, especially when they are under automatic control. However, characterization of the mass and heat transfer mechanisms that take place in the process are still not completely known and are a challenge to properly predict. Thus, developing the fundamental understanding of rotary kilns will greatly improve their scale-up from laboratory and pilot plant scales to a commercial manufacturing scale.

Significant focus has been attributed towards understanding the heat transfer as it is the limiting factor in many rotary kilns [2]. However, it is important to understand all the processes that occur in rotary kilns on a fundamental level before optimal design and operation of can be achieved. Particularly, in the preparation of chemical catalysts, an improved understanding of the fundamental processes in rotary kilns will improve continuous calcination processes. Figure 1 offers a visual representation of the rotary kiln where the particle bed exchanges heat with sweep gas and the interior surfaces as it rotates and moves axially along the length of the kiln. Normally, longer residence times are more beneficial more ensuring that all particles
have been sufficiently and uniformly treated [3], but this can burden the operations with large energy and material costs. Therefore, efficient calcination in rotary kilns requires low residence time and axial dispersion. The objective is to minimize both the residence time and axial dispersion while understanding their relationship to the kiln’s operating parameters.

Figure 1. Schematic of Rotary Kiln

Residence time and the time for calcination are influenced by both, radial and axial mixing. The first published experimental studies on rotary kilns recorded the relationship of rotation speed and kiln inclination, with bed depth and solids residence time [4, 5]. Lifters and dams of various shapes and sizes are commonly used in industrial scale kilns in order to improve mixing. The degree of particle mixing depends on the characteristics of the bed motion through the kiln. The mode of bed motion in the kiln influences the degree of particle mixing. These modes of motion depend upon rheological properties, fill level and rotation rate. [1]. A model to compute the mean residence time based on material properties and kiln geometry was developed by Saeman [3], based on the assumption that particles in a rolling bed move in a circular motion with the rotation of the kiln, and then fall down the surface of the bed in a thin layer. Analytical solutions that agree well with the model have since been developed [6, 7].
Empirical formulas were presented by Sullivan [5] which enabled the calculation of kiln factors such as volume of material in the kiln, depth of bed, capacity and the time of passage of solid particles through rotary kilns. Studies using such models have shown that feed rate have a low impact on mean residence time; however, an inverse relation was observed with respect to speed of rotation and angle of incline [3, 8, 9, 10, 11, 12].

While recognizing such trends aid in kiln operation, the mean residence time alone fails to provide insight into the axial dispersion of the particles and therefore the overall quality of the treated feed. For this, the residence time distribution, a probability distribution characterizing the flow profile of a material, must be measured [13, 14]. The width of the distribution depends on material flow determined by material properties and operating conditions. Narrower distributions are indicative of yielding a more uniform product. This can be measured experimentally by conducting a tracer study where tracer particles are injected at pre-determined location, and observed at the discharge end of the rotary kiln [15, 16]. This study has been previously conducted for millimeter-sized extrudates, broken rice grains and sand [3, 8, 9]. For studies involving extrudates, it was observed that higher feed rates and larger angle of repose of the materials led to higher fill levels, and a reduction in axial dispersion. In addition to this Gao et al. [3] found that the axial dispersion coefficient of millimeter-sized extrudates decreased with rotary speed and incline angle. These trends were confirmed for studies conducted using sand and rice grains by Njeng et al. in a pilot plant kiln equipped with square and rectangular lifters [9, 10, 11].

The goal of this research is therefore to extend the material database gained form previous kiln studies through study of a dry free-flowing powder. While powders are widespread in industry, predicting and characterizing their flow is difficult due to several
factors. In rotary kilns, product uniformity depends on kiln geometry and operating conditions [12, 13, 14]. In the experiments discussed in this paper, the tracer study developed by Danckwerts [15] was used to determine residence time distributions for different sets of operating parameters. The studies were conducted in pilot scale rotary kilns, at room temperature, using a free-flowing fluid catalytic cracking powder provided by W.R. Grace & Co. The parameters were chosen based on those from previous experiments and equipment capabilities. Seeking similar trends to those found for extrudates and dry cohesive powders [25], tracer studies following a methodology by Danckwerts [16] were conducted at room temperature using a dry cohesive powder as feedstock seeking similar trends to those found for extrudates.
Chapter 2

Methods

Measurement of Mean Residence Time

The mean residence time represents the average time it takes for particles to pass through the kiln. An effective way to calculate mean residence time is to use the mass holdup, $M$, which was measured after each trial by shutting off the feed and rotation followed by collecting all the powder in the kiln, and then dividing the holdup value by the mass flow rate, $\dot{m}$.

$$\tau_{\text{holdup}} = \frac{M}{\dot{m}}$$  \hspace{1cm} (1)

where $\tau_{\text{holdup}}$ is the mean residence time, $M$ is the mass hold up, and $\dot{m}$ is the mass flow rate of the material. The measured $\tau_{\text{holdup}}$ was then used to validate the prediction of mean residence time proposed by Sullivan (Equation 2).

$$\tau_{\text{Sullivan}} = \frac{1.77(\phi)^{0.5}Lf}{\beta D\omega}$$  \hspace{1cm} (2)

where $\phi$ the static angle of repose of the material, $L$ is the length of the kiln, $\beta$ is the angle of incline of the kiln, $D$ is the internal diameter of the kiln, and $\omega$ is the speed of rotation. A flow factor, $f$, is included to account for obstructions in flow due to modifications such as lifters. As the kiln used in these experiments contained no lifters, a value of $f=1$ was assumed to determine the accuracy of the model during each experiment. The flow factor can be defined as the ratio of $\tau_{\text{holdup}}$ to $\tau_{\text{Sullivan}}$.

$$f = \frac{\tau_{\text{holdup}}}{\tau_{\text{Sullivan}}}$$  \hspace{1cm} (3)
Residence Time Distribution and Axial Dispersion Coefficient

The residence time distribution was calculated using the following equation [17]:

\[ E(t) = \frac{C(t)}{\int C(t)} \] (4)

where \( C(t) \) is the concentration of tracer particles at time \( t \) and \( E(t) \) is the residence time distribution.

The axial dispersion model was used to represent the residence time distribution under the following assumptions [8, 18, 19]:

(i) the kiln reached steady state;

(ii) a delta-dirac tracer pulse was a function of axial position and time;

(iii) the axial convective velocity and axial dispersion coefficient of tracer particles was constant for stable operating conditions.

The model considers a convective and a diffusive component to the mass transfer. These components are resultant of the bulk motion of the material and random motion of the particles, respectively. This can be represented by the Fokker-Planck equation, which describes the in continuous systems Gao, 2012 [18, 20, 21].

\[ \frac{\partial C}{\partial t} = \frac{1}{Pe} \frac{\partial^2 C}{\partial x^2} - \frac{\partial C}{\partial x} \] (5)

Here,

\[ Pe = \frac{v_x l}{D_{ax}} \] (6)

where \( Pe \) is the Peclet number, \( l \) is the length traveled by the tracer, \( D \) is the axial dispersion
coefficient, and \( v_x \) is the axial velocity of the particles. In literature, several analytical solutions have been presented for granular materials [18, 20, 22, 23]. The Taylor [20, 22, 24, 25] dispersion solution to the partial differential equation was considered (Equation 7)

\[
E(\varepsilon, \theta) = \frac{Pe^{0.5}}{(4\pi \theta)^{0.5}} e^{-\frac{Pe(\varepsilon-\theta)^2}{4\theta}}
\]

\[
\theta = \frac{t}{\tau_{Taylor}}; \quad \varepsilon = \frac{x}{l}
\]

where \( \theta \) and \( \varepsilon \) represent the dimensionless time and location of the material in the kiln, respectively; and \( \tau_{Taylor} \) is the mean residence time which is implicitly generated from the Taylor fit, which can be compared to the other mean residence times that are calculated from the mass holdup and Sullivan’s model.

**Experimental Setup**

Experiments were conducted in a pilot plant rotary kiln at atmospheric temperature and pressure. In each experiment, a tracer study developed by Danckwerts [15] was used to determine the residence time distribution for different operating parameters and the implementation was similar to a previous study by Sudah et al. [9]. The operating parameters were chosen based on those from previous experiments [3, 25] and equipment capabilities. At a chosen speed of rotation and angle of incline, fluid catalytic cracking (FCC) powder was fed into the kiln at a constant feed rate until steady state is reached. The kiln was then shut off and cobalt impregnated FCC tracer particles were inserted into the kiln from the discharge end using a scoop mounted on a metal rod. Since a pulse injection is assumed, the dimensions of the spoon were neglected in the data analysis. A mixture of tracer and undyed FCC powder would exit the kiln, and timed samples were collected until the tracer was invisible to the eye. As the tracer
was distinguishable from the undyed powder by its black color, concentration $C(t)$ was determined as a function of time based on the % reflectance of each sample measured using a spectrophotometer. The collection time for the samples was determined by how much powder would be needed to pack the sample holder for the spectrophotometer (Spex cap, # 3167, 38 mm OD x 10 mm high). This was done to maximize the number of tracer samples collected, which in turn would provide sufficient concentration data points for the residence time distribution curves. Once all the visible tracer particles had exited the kiln, the feeder was turned off and the remaining particles in the kiln were collected for measuring the mass holdup. For each set of operating parameters, experiments were repeated three times in order to test reproducibility and consistency of the residence time distributions.

**Setup: 6.5 inch rotary kiln**

The kiln had an internal diameter of 6.5 inches and a length of 131 inches, giving an L/D ratio of 20.15. The kiln had no lifters installed. A schematic representation of the kiln is shown in figure 2.

![Figure 2. Rotary Kiln with Fixed Dam on the Feeder Section](image)

The matrix of experiments conducted is summarized below in Table 1. A screw feeder (Accu-Rate) fed the FCC feed material into the kiln at constant rates of 6.6 lb/hr. A control panel was used to set the kiln at various rotary speeds. A jack mechanism at the inlet of the kiln was used to adjust the incline angle to 1.1° or 2.3°. There was a fixed circular dam at the inlet, fitted
with helical lifters, as shown in figure 2, which prevented backward leakage of materials while ensuring the feed was deposited uniformly throughout the continuous feeding process. The maximum operation fill level without backward leakage was about 15%. Operating fill levels during experiments ranged from 0.7% to 15.86%. At higher fill levels, the breach, pictured in figure 2, was charged with FCC so as to prevent backflow of material. This material was removed, after each run, in order to prevent it from being collected when measuring holdup.

<table>
<thead>
<tr>
<th>Operation Variables</th>
<th>Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Incline angle $\beta$ ($^\circ$)</td>
<td>1.1, 2.3</td>
</tr>
<tr>
<td>Rotary speed $\omega$ (rpm)</td>
<td>1, 4, 12</td>
</tr>
<tr>
<td>Feed Rate (lb/hr)</td>
<td>6.6</td>
</tr>
</tbody>
</table>

**Table 1. Matrix of Operating Conditions for Setup**

**Materials**

The feed material used was a fluid catalytic cracking (FCC) powder manufactured by W.R. Grace & Co. The catalyst powder chosen because it is industrially relevant and it will help build upon the previously conducted residence time distribution studies [11, 25]. The measured material properties can be found below in Table 2. The loose bulk density was measured using the mass of material required to fill a 100 mL graduated cylinder. The static angle of repose was measured using a fixed funnel method. Each measurement was repeated three times and the average angle of repose was considered.
Incipient wetness impregnation method was used to prepare the tracer material using cobalt (II) nitrate hexahydrate as a precursor. The resultant tracer contained 10 wt% cobalt oxide following oven drying at 120°C for 8 hours and calcination at 538°C for 6 hours. Table 2 also shows the loose bulk density and the static angle of repose of the tracer material.

<table>
<thead>
<tr>
<th>Material</th>
<th>Diameter (mm)</th>
<th>Bulk density (g/cm³)</th>
<th>Static angle of repose (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FCC Catalyst (W.R. Grace)</td>
<td>0.071</td>
<td>0.73</td>
<td>24.9</td>
</tr>
<tr>
<td>FCC Tracer (10wt% Co)</td>
<td>0.076</td>
<td>0.86</td>
<td>25.8</td>
</tr>
</tbody>
</table>

Sample Analysis

Figure 3 shows an X-rite 939 spectrophotometer that was used to measure the concentration of tracer particles in the samples obtained from the experiments. Each sample was tested for % reflectance values based on the L-a-b color space (Figure 4). The L-dimension, based on the lightness of the sample, was the primary value used to determine tracer concentrations. The data shown in figure 4 was taken at an incline of 2.3°, 4 rpm and a feed rate of 6.6 lb/hr.
A calibration curve that was developed using % reflectance data of FCC samples containing pre-determined amounts of tracer concentrations ranging from 0 wt% to 50 wt%. The resulting concentration curve shown in Figure 5 was then used to generate the residence time distribution curve fitted using the Taylor fit (Equation 4), this is shown if figure 6. The Taylor fit also outputs values for experimental mean residence time, axial dispersion coefficient, and Peclet number.

Figure 4. % Reflectance vs. Time.
**Figure 5. Example of Spectrophotometer Calibration Curve**

![Spectrophotometer Calibration Curve](image1)

The calibration curve is given by the equation:

\[ y = 0.0064x^2 - 0.9668x + 87.715 \]

with an \( R^2 \) value of 0.9978.

**Figure 6. Tracer Concentration vs. Time**

![Tracer Concentration vs. Time](image2)

The tracer concentration over time for three trials is shown in the graph.

- **Trial 1** (green line)
- **Trial 2** (red line)
- **Trial 3** (blue line)
Chapter 3

Results and Discussion

Mean Residence Time for 6.5 inch rotary kiln

Figure 7 shows the comparison among the experimental data and Sullivan’s model at both 1.1° and 2.3° angles of incline. Generally, Sullivan’s model agrees well with the experimental mean residence time, calculated from holdup. The Sullivan model thus predicts mean residence time more accurately for free-flowing powder in kilns without lifters as there is no back mixing of the particles as they travel along the powder bed. This could also serve to confirm such back mixing as the reason for underestimation of the residence time by Sullivan’s model, in the presence of lifters regardless of shape and size [32, 33], as observed by Njeng et al. [8, 9] and Paredes et al. [25]. An increase in incline was found to decrease the mean residence time, as predicted by Sullivan. Since the feed is kept constant, this decrease in mean residence time is due to the axial velocity gained by the particles with an increase in incline.

An increase in incline from 1.1° to 2.3° led to an average of 52% decrease in mean residence time at 4rpm and 1rpm; however the decrease was only 8% at 12rpm. At low speeds of rotation, the motions serves its purpose to improve mixing; however, at higher speeds of rotation the bed motion in the kiln was observed to be in the slipping regime. As a result, at 12rpm the axial velocity of the bed was high enough to approach plug-flow like behavior, and the impact of incline on the mean residence time was subsequently diminished. The mean residence times were measured for the set of parameters listed in table 3, at a constant feed rate of 6.6 lb/hr. Sullivan’s model was found to be sufficiently accurate at a wide range of fill levels, ranging from 0.77% to 15.86%.
Table 3. Operating Conditions for Mean Residence Time Measurement

<table>
<thead>
<tr>
<th>6.5” Rotary Kiln</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Angle of incline $\beta$ ($^\circ$)</strong></td>
</tr>
<tr>
<td><strong>Rotation Speed (rpm)</strong></td>
</tr>
</tbody>
</table>

Figure 7. Mean Residence Time Data for 6.5 inch Kiln at Various Speeds of Rotation and Angles of Incline

\[
\text{MRT} = \frac{178}{\omega \cdot \beta}
\]
Residence Time Distribution and Axial Dispersion Coefficient

The residence time distribution $E(t)$ was obtained for each run conducted at various operating conditions. For each set of runs, three residence time distribution curves were averaged together to make an average residence time distribution curve for a given set of operating parameters. This is done by computing the average mean residence time and axial dispersion coefficient from each of the three trials and plugging those values into the Taylor fit equation. Figure 8 shows the residence time distribution curves generated for the same operating conditions for Figures 4 and 6. Though minor variations were seen among trials, a good agreement with the Taylor Dispersion model was observed.

A specific set of criteria was established to reduce the amount of experimental error and adjust the residence time distribution curves. Any data sets that contained negative tracer concentration values were adjusted upwards by adding the absolute value of the most negative value to all the data points. Any outliers at the beginning or the tail ends of the curves were set to 0 wt%, if they were more than 25% of the maximum concentration value in a specific data set. When comparing the normalized residence time distribution curves, before and after these adjustments were made, there were no significant changes in mean residence time or axial dispersion coefficient.
Effect of Rotary Speed and Mean Residence Time

Residence time distribution curves were obtained for 1, 4 and 12 rpm at 2.3° incline. As the speed of rotation of the kiln was increased, the residence time distribution curves got narrower and there was a decrease in the mean residence time, as shown in figure 9. Good agreement with the Taylor Dispersion model was found. This can be explained by the increase in the axial velocity of the particles with rotary speed. From the Taylor Dispersion model, the axial velocity of the particles was found to be 5.31 cm/min, 22.23 cm/min and 57.07 cm/min for rotary speeds of 1, 4 and 12 rpm, respectively. Thus, more particles would move into the active layer at higher speeds of rotation, subsequently causing them to spend less time in the kiln.

The sensitivity of the axial dispersion coefficient, $D_{ax}$, is shown in figure 10. At 4rpm the value of $D_{ax}$ increased by nearly seven times the value of $D_{ax}$ at 1rpm, while it increase about thirteen times from 4rpm to 12rpm.
A narrow RTD curve was expected to point towards a lower value of $D_{ax}$. However, trends from figures 9 and 10 suggest an inverse relationship of the axial dispersion coefficient to the mean residence time, and consequently an inverse relationship to the speed of rotation.
Bed Depth in 6.5 inch Rotary Kiln:

The depth of the powder bed was measured from the discharge end at regular intervals after each run was completed, but before the holdup was measured. Two methods for measuring bed depth were considered for these measurements - the first method involved using a depth gauge to measure bed depth, while the second method utilized a divider as a drafting tool to accurately measure the width of the powder bed which was used to calculate the bed depth.

Using the depth gauge caused the powder bed to be disturbed and as a result, the subsequent repetitions of depth measurements were inconsistent. Moreover, using the depth gauge heavily relied on maintaining line of sight and the graduations on the gauge were harder to discern beyond a certain length from the discharge. Using the divider was much easier as the bed width could be accurately transferred to a scale on which the measured value was read. As the bed was left undisturbed during bed width measurements, multiple reading could be taken in order for confirmation. Figure 11 shows a visual representation of the bed depth calculation.

Figure 11. (a) Powder bed in 6.5 inch rotary kiln at 4rpm, 2.3° incline and a feed rate of 6.6 lb/hr. (b) Calculation of bed depth using width measurements of powder in kiln

\[ d = r - \sqrt{r^2 - l^2} \]
Figure 12 shows the comparison between bed depth values obtained using each of the two aforementioned methods. The depth values calculated from the bed width measurements consistency and hence were considered reliable for future measurements. Figure 13 shows variation of bed depth with respect to change in speed of rotation and angle of incline for the rotary kiln studied.

![Figure 12. Comparison of Measured Bed Depth and Calculated Bed Depth at 2.3° and 4rpm](image1)

![Figure 13. Bed Depths Calculated at 1.1° and 2.3° for Various Rotary Speeds](image2)

As expected, the bed depth showed an inverse relation with the angle of incline and rotary
speed. However, the readings were taken until the bed appeared to be uniform, which was found to be 18 inches from the discharge for 1rpm and 4rpm. At 12 rpm however, the bed reached uniformity around 12 inches from the discharge end. The shell of the rotary kiln was assumed to be perfectly cylindrical for these calculations. The velocity profiles of the particles in the active layer can be symmetric or asymmetric due to variations bed depth and rotary speed.

![Figure 14. The various zones of the powder bed in a rotating kiln](image1)

![Figure 15. Schematic representation on modes of transverse bed motion. [21]](image2)
The motion of the particle beds at these speeds of rotation were also observed. The mode or regime in which the kiln operates can be expressed in terms of bed behavior at given conditions – slipping, slumping, rolling, cascading, contracting, centrifuging. As shown in figure 14, the layer of particles moving down the face of the bed leads to improved mass transfer [21], therefore the surface layer is often referred to as the “active layer”. Figure 15 shows a schematic representation of the aforementioned regimes [21] based on the observed transverse bed motion.

At 1rpm, the particle bed resembled a cascading behavior along the length of the kiln while at 4rpm, the bed motion appeared to exit the cascading regime well within the heating zone and exit at the discharge in a rolling regime. At 12rpm however, the bed behavior was predominantly in the slipping regime. In comparison to previous work done [3], this analysis is insufficient to understand the effect of fill level and bed depth on the possible transverse bed behavior in rotary kilns.
Conclusions and Future Work

The mean residence time for dry free-flowing powder was measured and the residence time distribution was studied in this work, using the Taylor dispersion fit to the axial dispersion model. Previously, this model was used to study residence times and determine the value of $D_{ax}$ for free-flowing materials like extrudates and dry cohesive powders. Overall, it was observed that for increasing speed of rotation and angle of incline, there was a general decrease in mean residence time and residence time distribution curves shifted towards lower time values and became narrower. These results are consistent with previous results for different calcination systems where the incline and rotary speed of rotary kilns were found to have a more significant effect on the residence time distribution and the mean residence time. Values for axial dispersion coefficient were also within similar ranges with some variations in trends for increasing certain variables [25, 19, 30, 33]. The experimental residence time agreed well with the residence time predicted by the Sullivan model. The contribution of incline angles to the mean residence time may be dominant, but that may only be true at lower speeds of rotation as Sullivan’s model was found to be more accurate at higher rotary speeds and a higher incline, which was not seen in previous experiments.

Previous studies by Paredes et al.[25] showed unexpected effects of feed rate as the velocity profile was discontinuous due to the large fraction of the power bed being consumed by the lifters, and thus, hindering the effect of rotary speed. The mean residence times were measured for a fixed feed rate of 6.6 lb/hr as the absence of lifters implied an absence of such hindrance in the particle flow behavior. Therefore, by comparison to previously used equipment, the experimental setup discussed in this work can be used further to obtain a better understanding of the impact of rotary speed on the residence time in rotary kilns.
In regards to future work, there is more analysis to be done in regards to characterizing trends for residence time distribution curves and axial dispersion coefficients. Work must be done to better account for the effects of feed rate and geometry on the axial dispersion coefficient, $D_{ax}$. Figure 16 shows preliminary work identifying the impact of scaling on mean residence time, which has not been done before.

![Graph](image)

Figure 16. Mean Residence Time Data for 13.5 inch Kiln at a Fixed Angle of Incline (1.1°). Feed Rates Scaled Volumetrically from 6.5 inch Kiln Studies.

The main areas of interest are performing multivariate analysis to quantify the effects of changing multiple independent variables and making more comparisons to previous experiments. The ultimate goal is to combine our understandings of mass transfer and heat transfer to aid scale-up of rotary kilns.
Appendix A

Standard Operating Procedure for Rotary Kiln Tracer Study

Preparation

1. Prior to the start of the experiment, record the time, temperature and humidity data available for the location.
2. Set the kiln incline, rotary speed and feed rate to desired levels.
3. Load the feeder with powder.
4. Allow the kiln to run until the outlet feed rate is constant. This typically takes 1 – 1.5 times the mean residence time of the particles. Sullivan’s model can be used to get a somewhat accurate prediction.
   a. Measure the feed rate every five minutes by collecting and weighing mass samples until they are constant.
   b. Check where the feeder drops the material in the kiln. If it is not directly at the start of the shell tube, measure the distance between the inlet face and the drop off point.
   c. Calculate the holdup of this region to determine if it is significant enough to effect results.
5. Once the flow is steady, take a 1-minute video from the calciner outlet to get a view of particle behavior at that operating condition.

Tracer Study

1. Weigh out the desired amount of tracer required for the study.
2. Shut off the kiln completely, so that is no longer receiving material from the inlet or rotating.
3. Introduce the tracer at a distance 45” from the discharge end of the kiln using a spoon.
4. Turn the equipment back on, allowing tracer to mix and flow with the particle bed.
5. Collect samples every 5 minutes for 30 seconds at a time from the moment the kiln is turned back on.
6. When tracer is visible in the sampling cup, continuously collect samples for time interval of 10 to 30 seconds each, depending on the rate of discharge, as long as each time enough sample is collected to pack the Spex® cap used to conduct %reflectance tests on the spectrophotometer. Collect samples until tracer is no longer visible.
7. Following the tracer study, empty the calciner and mass the holdup.

* For 2 and 4, it may be helpful to have someone else shut down and turn on the kiln to reduce the lag time between steps.
Standard Operating Procedure for Rotary Kiln Tracer Study

1. Transfer 1 kg of FCC catalyst to a Buchner funnel covered with a wetted filter paper and attached to a filtration flask.

2. Prepare an aqueous solution using 534 g of cobalt (II) nitrate hexahydrate and 500.44 mL of water.

3. Begin vacuum filtration and add the aqueous solution to the FCC catalyst slowly to evenly cover the FCC in the Buchner funnel.

4. Turn off the vacuum once the filtration is completed.

5. Dry the resulting filter cake at 250°F (120°C) for 8 hours.

6. Calcine the powder, by introducing heat at 3°F (1.67°C) per minute until 1000°F (538°C) is reached.

7. Once 1000°F (538°C) is reached, leave the powder at that temperature for 30 minutes.

8. Remove the powder from the oven. The final powder should be black in color as shown in figure 17.

Figure 17. Cobalt Impregnated FCC Tracer
References


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