CONTINUUM APPROACH
TO OPTIMIZING DOWNSTREAM FINAL DRYING WITH UPSTREAM SOLID-LIQUID FILTRATION, CAKE WASHING & DEWATERING

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ABSTRACT

This article will review the optimization of the selection of the final drying technology in conjunction with the upstream solid-liquid separation for filtration, centrifugation, cake washing, and dewatering technology selection. Most often when analyzing a new process development approach, engineers take a "silo" approach and look at each step independently. This article illustrates that by taking an integrated and holistic approach and looking at each step not individually but as a continuum, the process solution becomes much more efficient.

Three examples are discussed illustrating an integrated and continuum approach. The standard approach would be to first look at the solid-liquid filtration or centrifugation step and optimize this step for the maximum washing and drying efficiency and then with this information optimize the downstream drying. However, a new and different approach is to look at the process as a continuum from solid-liquid filtration/centrifugation through cake washing and dewatering to final drying. The "Integrated and Continuum Approach" results in operational energy and nitrogen savings as well as lower capital and installation costs for a more efficient and reliable process.

1. INTRODUCTION

Process optimization takes time and effort to analyze the problem, not the symptoms but the causes, talk to the plant operating staff and actually "walk around" the process to understand all of the details. Each step of the process is interrelated and to be effective in problem solving, the approach must also be integrated. For example, when troubleshooting a dryer, improved solid-liquid separation maybe the key to improving the dryer.

The following examples demonstrate this integrated and continuum approach to process optimization. The solid-liquid separation technologies include continuous pressure filtration with either a "single-drum" or "drum with individual cells" and centrifuges either vertical peeler or horizontal inverting [1]. The final dryer technologies include either conical vacuum or "Nauta-type" [2].

2. EXAMPLE 1: CONTINUOUS PRESSURE FILTRATION TO BATCH CONICAL DRYER

In this first example for manufacturing of this specialty chemical, the crystals coming from the reactor in a methanol slurry must be filtered, washed, and dewatered and then dried to a final moisture of less than 1.0 (<1.0 %). The operating company had several objectives in mind for this process:

- Maximum solid-liquid filtration performance
- Low wash ratios for minimum wash media consumption
- Lowest possible residual moisture in discharged filter cake
- Final moisture of <1.0%

The analysis of the process always begins in the laboratory with slurry testing.

2.1 ON-SITE PROCESS LAB TEST WORK

Process testing can be conducted at the site's laboratory or at an outside or vendor lab. It is important that if the testing is conducted outside of the plant, that the slurry is representative and does not change during shipping, storage, etc. A typical test set-up is shown in Figure 1.

The objectives for this lab testing are as follows:

- Filtration time vs. cake thickness
- Filtrate quality vs. filter media
- Cake solids wash time and quality
- Cake solids drying time and final moisture

2.2 CONCLUSION FROM THE LAB & PILOT TESTING AND SCALE-UP

The tests demonstrated that acceptable filtration and solids wash rates could be obtained for this product and acceptable solids levels were observed for the mother liquor filtrate. Washing results and drying quality were also achieved.

The pilot tests confirmed the lab testing such that with a 14 micron cloth and a cake thickness of 25 mm, filtration times and filtrate quality were achieved. The wash ratios were very efficient, ranging from 0.7 to 1.2 kg Methanol/kg dry solids.

The moisture content of the cake varied between 11 – 30% depending upon the amount of nitrogen for blowing of the cake for drying. Based upon the initial thinking, the sizing of the Continuous Pressure Filter for moisture of 11% resulted in a filtration area of 2.88 m^2 including a nitrogen solvent recovery package to reduce the nitrogen usage. This is the important point for the dryer optimization.

2.3 PROCESS DRYING TESTING

The wet cake from the Continuous Pressure Filter was tested on a vertical batch dryer. This is where the benefits of the “continuum” approach really shine. The drying testing started with three initial moistures at 11.6%, 17.5% and 30%. The results are as follows:

Initial moisture	Max. product temp.	Vacuum (mbar)	Drying time	Final moisture	Dryer Size
17.5%	65°C	100 - 300	45 min	<1.0%	2.4 m3
11.6%	95°C	5-120	35 min	<1.0%	1.9 m3
30 %	65°C	100 - 300	63 min	<1.0%	3 m3

The conclusion from the testing shows a small increase in the drying time and dryer sizing from a cake moisture of 30% moisture as compared with 11.6%. Figure 2 shows the existing approach as well as the benefits from an integrated and continuum approach.

3. EXAMPLE 2: BATCH CENTRIFUGATION TO BATCH CONICAL DRYER

In this pharma example, the process engineers of this multipurpose plant had an objective to optimize the centrifuges and conical dryers. The questions facing the engineers included (1) should the centrifuges be operated with or without predrying and (2) how would the centrifuge operation impact the downstream dryers.

3.1 ANALYSIS AND DECISIONS

As discussed previously, laboratory testing is critical to decision making. The process is as follows:

Product C: Required capacity, dry solids: 1200 kg in 6 days (20 h day) ➔ 10 kg/h

Option 1 with No Pre-drying:
Capacity for 600 mm centrifuge, Residual Moisture after centrifugation is approximately 10 % w/w.

Option 2 with Pre- drying:
Capacity for 600 mm centrifuge with Pressure Gas at 49°C, Residual Moisture is approximately 4 % w/w

Final Drying: < 1% hexane

Product M: Required capacity dry solids: 1000 kg in 4 days (20 h day) ➔ 12.5 kg/h

Option 1- Centrifugation with No Pre-drying
Capacity for 600 mm centrifuge, Residual Moisture after centrifugation is approximately 35 % w/w

Option 2- Centrifugation + Pre- drying
Capacity for 600 mm centrifuge with Pressure Gas at 25°C, Residual Moisture is approximately 17 % w/w

Final Drying: < 100 ppm heptane

The final design resulted in one centrifuge, 800 mm, without pre-drying (to save the costs of nitrogen, etc.) and one 1.5 m^3 Conical Vacuum Dryer.

4. **EXAMPLE 3: BATCH CENTRIFUGATION TO BATCH "NAUTA-TYPE" CONICAL SCREW DRYER**

4.1 ANALYSIS AND DECISIONS

This multipurpose active pharmaceutical ingredients (APIs) plant produces two products (1) Product A at 400 tons/year and (2) Product B at 100 tons/year. The engineers had several options for centrifuges:

- One (1) x 1000 mm or Two (2) x 800 mm
- With or Without Pre-Drying

In addition, for Product A, the initial moisture after centrifugation is 42% LOD and with pressure assisted gas predrying, the LOD reduced to 23%. Similarly, for Product B, the LODs were 23% and 18%, respectively.

For the dryer choices, the decisions for the Conical Screw Dryers are as follows:

- Two (2) x 200-liter dryer or Two (2) x 250-liter dryer
- One (1) x 400-liter dryer

The final design is shown in Figure 3.

5. CONTINUUM APPROACH & SUMMARY

In summary, the "Continuum and Integrated Approach" results in savings of energy and nitrogen plus capital savings. As operating companies develop new and unique chemical processes, there are many choices for filtration, centrifugation, cake washing, dewatering, and drying. These steps are a complex process where there is considerable amount of engineering and science involved. Engineers must evaluate all outcomes to make an informed and successful decision. Technical evaluation and, as shown, laboratory and pilot testing are critical for a successful decision and project. The take-away is that close collaboration between the operating company and the vendor will allow for creative problem-solving and process solutions to achieve the desired quality and production requirements for a reliable operation.

REFERENCES

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FIGURE 1: PRESSURE FILTRATION TESTING (COURTESY OF BHS-SONTHOFEN INC.)

EXAMPLE 1: Continuous Pressure Filtration to Batch Conical Dryer

- Initial Design
  - Filter Size: 2.88 m^2 with 11% moisture using 260 m^3/hr N_2 + Vacuum
  - Dryer Size: 1.93 m^3
  - Dryer Cycle Time: 35 minutes
  - Total System Budget Price: $2 million
- Optimized Design
  - Filter Size: 1.44 m^2 with 30% moisture using 200 m^3/hr N_2 + Vacuum
  - Dryer Size: 3.0 m^3
  - Dryer Cycle Time: 60 minutes
  - Total System Budget Price: $1.5 million

FIGURE 2: BENEFITS OF AN INTEGRATED AND OPTIMIZED APPROACH FOR NITROGEN USAGE AND LOWER CAPITAL COSTS

EXAMPLE 3: Batch Centrifugation to Batch "Nauta-Type" Dryer

FINAL DESIGN

- Multipurpose Pharma Plant
  - Product A: 400 tons/year
  - Product B: 100 tons/year
- Centrifuge Decision
  - **One (1) x 1000 mm**
  - **Without Pre-Drying**
- Nauta Conical Screw Dryer Decision
  - **One (1) x 400-liter dryer**

FIGURE 3: BENEFITS OF AN INTEGRATED AND OPTIMIZED APPROACH FOR NITROGEN SAVINGS AND LOWER CAPITAL COSTS