PROCESS FILE

Paste Slurries

N32980

APPROVED: SEPTEMBER 25, 1953

CHAMBERS WORKS PROCESS FOR THE SPRAY DRYING OF PASTE SLURRIES IN THE AZO SPRAY DRYER (D-19)

HISTORICAL

This is the first issue of a Chambers Works Process.

Many finished or semi-finished chemical products contain quantities of water as a result of chemical or physical processes which are utilized in their manufacture. Generally this water must be removed to facilitate the grinding, standardization or other finishing steps. Spray drying is one of several methods which may be utilized to remove this water.

The spray dryer installed in the Azo D plant (D-19) is a relatively small experimental unit installed to study the removal of water from Chambers Works products. It is of sufficient size to operate also as a small production unit. It is a venturi-atomizer type developed at the du Pont Experimental Station.

The product slurry is sprayed into a chamber containing hot gases which vaporize the water. The water vapor and gases are separated from the product by a wool felt bag filter and the product is packaged. Normally this dry product is ground and standardized.

PATENT STATUS

We are free to use the spray-drying procedure of this process provided we operate outside the claims of U.S. 2,297,726, Stephanoff, i.e., provided the velocity of the jet of drying air is sufficiently below sonic velocity to avoid the claims. See AN-4392, G. W. Cable, February 4, 1953.

Specific materials to be dried by this process may require further clearance. We are free, with the above limitation, to use this procedure in processing the following types of products:

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"Celanthrene" Color Crudes Solubilized. See AN-4134, J. W. Lotz, April 9, 1952.

Du Pont Anthraquinone Wool Colors. See AN-4517, J. W. Lotz, May 30, 1953.

"Ponsol" D Powders. See AN-4516, J. W. Lotz, May 30, 1953.

PRINCIPLE

Water slurries, preferably of as low a water content as will still allow flow, are pumped through an atomizing nozzle consisting of four concentric tubes. The slurry is pumped through an innermost tube which is jacketed by another tube through which air or water is passing to prevent caking in the tube due to hot gases passing around it.

A third tube jackets the inner two tubes. Hot air (from an electric air heater) is passing through this tube and by means of a nozzle at the bottom, breaks the slurry into small droplets much the same as a spray from a water hose.

The drying air from a gas burner is in a fourth tube surrounding the other three tubes. This drying air removes the water from the atomized droplets.

All tubes discharge into the top of a 2-ft. diameter, 10 ft. long vertical chamber which promotes intimate mixing of the air and atomized slurry. The dry product, water vapor and gases pass from the bottom of the drying chamber into a wool or "Orlon" felt bag dust collector. Gases and water vapor pass through the bag and into the atmosphere. The dry product passes through an air lock into a barrel.

To move the water vapor and introduced gases, the chamber and collector system are under slight vacuum induced by an evacuating fan.

To prevent overheating of the wool or "Orlon" felt bag, air is bled in directly below the drying chamber, as needed, to maintain a temperature no higher than 120°C.

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EQUIPMENT

Azo Area - Building No. 183-D

One barrel or slurry tank for slurry which feeds to the atomizing head of the dryer and partly returns if not needed. For dimensions of slurry tank, see process for HF milling of pastes at Azo.

One Moyno pump. This is a positive displacement pump consisting of a specially compounded rubber tubular housing and a stainless steel rotor (fabricated in the form of an elongated screwed thread). It is used to pump the slurry into the spray dryer. The pump must never be operated without liquid feed since it depends on the liquid pumped for lubrication. A few seconds dry operation will damage the pump extensively.

One Atomizing head. This head consists of four concentric tubes. The inner or slurry tube is of stainless steel, 18-3/8 in. in length, 3/16 in. in inside diameter and 1/4 in. in outside diameter. Slurry is pumped through this tube into the dryer. The slurry tube is surrounded by a cooling air (water optional) tube which is of stainless steel and is 15-13/16 in. long and 3/8 in. in interior diameter. This tube prevents "caking" in the slurry tube. Air or water is controlled by rotameters on the control panel. The cooling air tube is surrounded by an atomizing air tube. This tube is of stainless steel pipe, 1-1/2 in. in diameter and 11-7/16 in. in length. The tube is fitted at the bottom with a cone-shaped reducing nozzle which extends the pipe an additional 1-3/16 in. The reduced part of the cone is 0.52 in. in diameter. (See Note 1) This constriction atomizes the slurry coming from the slurry tube and forces it down into the drying chamber in the form of a long cone.

The atomizing air tube is surrounded by a stainless steel drying air tube which is 8 in. long and 6 in. in interior diameter. The bottom outlet is constricted with an orifice 4-1/4 in. in diameter. Above, the orifice is fitted a baffle consisting of a stainless steel plate with 1/2- and 1/4-in. holes. This baffle is designed to produce an even, downward flow of hot air without a directional flow towards one side or the other. The atomizing air nozzle projects about an eighth of an inch below the bottom of the drying air orifice, the slurry tube projecting about 1/8 in. below

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the atomizing air tube. The jacket tube is slightly above the bottom of the atomizing air nozzle.

One three-way valve receiving slurry from the Moyno pump and allowing it to enter into the drying chamber through the slurry tube and also returning it to the slurry tank. The amount of feed through the slurry tube is controlled by the temperature within the drying chamber.

One gas burner. (BU-1) This is a Thermal Research and Engineering Corporation's gas burner installation. It consists of a gas burner with stainless steel mixing chamber fitted with a stack leading outside the building for entry of secondary air. A damper on the secondary air is fully open as present belief is that this setting gives best dryer operation. The burner is supplied with producer gas from the Chambers Works gas mains at a pressure of about 39 in. of water. The air supply is from a Spencer blower driven by a 1/2-hp, 550-volt motor. A safety shutoff is installed in the air supply for shutdown in case of blower failure. The air and gas are mixed in a venturi mixer and combustion takes place in a ceramic burner chamber before entry to the mixing chamber. The mixing chamber is equipped with a "Fyr Eye" system which shuts down the burner in case of flame failure to prevent unburned gas being drawn into the dryer chamber. Air and gas supply are automatically mixed in the proper ratio over the required temperature ranges. This is effected by built-in nozzles and a connecting diaphragm valve system. Ignition is by spark plug using a Webster Ignition Transformer. The hot gases resulting from combustion and the dilution air are passed through 5 in. in diameter steel lagged ducting to the drying air tube in the atomizing head. The dryer is located in a cinder block ventilated enclosure with a sheet steel cover. There is an electrical interlock system which will not allow operation of the gas burner, electric Fintube Heater or Moyno Pump until the exhaust blower has been operating for 3-4 minutes.

One Brown Fintube Company's electric air heater (H-10). This heater is used to supply hot air to the atomizing air tube in the atomizing head. Temperature of hot air supplied is registered at the control panel. Air flow is controlled and measured by a rotameter on the control panel.

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One Dryer Chamber. This is a stainless steel cylinder, 2 ft. in diameter and 10 ft. in height. The bottom is closed by a stainless steel nozzle tapering and leading to a 5-in. in diameter iron duct leading to the collector. The top of the chamber is closed by the atomizing assembly. Thermocouples are installed at the hot gas intake and bottom of the drying chamber. Temperatures are recorded on the control panel. The shell is shaken by a vibrator during drying to shake down material adhering to the dryer walls.

One Mikro-Collector. (DC-2) This is a Pulverizing Machinery Company's product and consists of an upper chamber containing a wool felt bag, 36 in. in diameter and 54 in. high. (No. 36-1-54) The bag is attached to the bottom of the dryer chamber and at the bottom to a cone which is closed by an air lock. The gases and dried product enter the bag. A blower driven by a 5-hp. motor creates a partial vacuum (approximately 12-17 in. of water) thereby drawing the air and water vapors through the bag, leaving the product on the inside surface of the bag. It should be noted that the entire system operates under a negative pressure. The air and vapors are blown to the atmosphere and the product is discharged from the bottom of the collector through a motor-driven valve. A manometer shows pressure differential between the interior and exterior of the bag. Lack of pressure indicates bag failure.

To avoid burning of the wool felt bag, it is necessary to bleed cool air into the duct between the dryer and the collector. A temperature controller varies the amount of air bled in as required through a rubber balloon diaphragm valve and sounds an alarm as the temperature rises to a dangerous level. The bag should be treated to prevent static build-up.

The product is loosened from the inside surface of the bag by a slotted ring which travels up and down on the outside of the bag. The ring is slightly smaller than the bag and exerts a slight squeezing action. Air is also forced through slots on the inner side of the ring by a blower thereby blowing the product off the bag into the conical opening at the bottom of the collector. The spray dryer should never be operated when the blow ring drive is not energized. The build-up of material inside the bag prevents flexure of the bag. When the blow ring drive is energized, probable damage to the blow ring mechanism may result.

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The bottom of the collector cone is closed by a motor driven rotary valve with "Teflon" blades. This allows solid material to drop from the cone to the shipping package in the drumming booth below without reducing vacuum in the collector and drying system above.

One Control Panel. This panel contains the instruments for control of operation for the spray dryer. A recording instrument records and controls the temperature of the entering drying air. Another recording instrument records and controls the temperature of the atomizing air. Another recording instrument records the temperature near the bottom of the drying chamber. This instrument controls the rate of slurry feed automatically to maintain temperature within the dryer at the temperature set point. A fourth instrument measures the temperature within the bag and air will be bled in through the duct above the felt bag to maintain this temperature. A danger temperature setting is installed so as to sound an alarm system in case of excessive temperature within the bag. The entire system will be shut down if too high a temperature in the bag is obtained. The alarm will also sound if no pressure or too high a pressure is exerted on the bag. An emergency button is located on the control panel to shut down the entire system. In this case, the exhauster fan will run for one minute to clear gases from the system.

The gas line for introduction of producer gas to the system is closed by two valves which may be closed in an emergency or for repairs or adjustment to the gas system. A spool piece in the line can be removed and a steam line can be introduced for steaming out to clean the gas line.

A flow meter to record rate of flow of gases through the system is installed on the north side of the collector.

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PROCEDURE

Be sure the dryer is clean or if starting up after a shutdown during a run that the dryer is sufficiently clean, especially around the orifice plate at the atomizing head that no fire will start from burner gases igniting color before slurry is fed. The controlling instruments are assigned letters on the operating chart for clarity. Before starting the dryer, if drying from T-50, open the line from T-50 to the Moyno pump (slurry pump) to allow slurry to feed to the pump and close the two valves on the return line from the three-way valve M leading back to the barrel (at barrel and on third floor). If drying from a barrel, open the two valves from the barrel to the Moyno pump, open the valve from the three-way valve M back to the barrel and shut both valves on the return line leading to T-50. Shut the feed line from T-50 tub (two valves). The Moyno pump (P-20) must never be allowed to run dry as it requires slurry for lubrication. When using a barrel for feeding slurry, the union at the barrel should be tight to get proper suction. P-20 should be full of cleanup water on the start-up. If there is any doubt, fill the pump and lines with water from the attached water line. Bleed the air trap on the catch tank supplying air for the system.

Shut the bleed valve on the slurry tube at the atomizing head.

1. Change charts on instruments A, B and C.

2. Press the exhauster (start-up) button. A red light will come on for several minutes while gases are being drawn from the system. This acts as a safety measure against producer gas leaks. When the evacuation cycle has been

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completed (2-4 minutes), the red exhauster light will go off and the green exhauster light will come on. Simultaneously red lights will appear over the gas burner, slurry pump and electric heater buttons.

3. Check to see that the air valve on the third floor to the electric heater is open. Open the rotameter valve E to the electric heater and regulate air pressure to 9 ± 1 pounds per square inch. (See Note 1) Open valve F to supply air to the tube jacketing the slurry tube. Adjust pressure to 10 ± 1 p.s.i. (pounds per square inch). Press the start button for the electric heater, the red light for the electric heater should go out and the green light should go on. Set the electric heater set needle to 450°C. Allow the heater to warm the atomizing air flow to at least 150°C. before proceeding further. This temperature is shown on instrument B. A light will show on instrument B indicating that the instrument is functioning. Maximum temperature obtainable with this electric heater is only about 350°C.

4. Start the ring drive, the ring blower and the rotary air lock by pressing butterfly switches H, I and J. Check their operation.

5. Place a crude dye barrel under the rotary air lock and weigh for tare weight. Place a lid over the barrel for collection of the first dried product. This lid is removed after the first of the dried product is collected and found to be satisfactory. No lumps or wet material should be obtained. Observe the pressure differential as shown on manometer K. This should be at least 2 in. (1 in. up one side and 1 in. down the other side of the column) even on start-up. (The column is filled with water and a little

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red ink.) Less pressure differential indicates a hole in the wool felt collector bag or that the bag is not securely bound by the retaining straps around the top or bottom of the collector. Check this manometer frequently during operation to be sure the bag remains in good condition.

6. Adjust the green needle on instrument C to 250°C. This will prevent the three-way valve M feeding slurry into a cold dryer. Press the start button on the slurry pump. The red light on the control panel should go off and the green light should go on indicating that the pump is running. Slurry must start returning back to the barrel or T-50 whichever is used within less than a minute. During the operation of the dryer, check this return flow frequently to be sure the slurry pump continues to function.

7. Set the orange pointer on instrument D to 150°C. If the temperature within the felt bag rises to 150°C., the entire system will shut down and the horn on the collector will blow. Set the other needle pointer at 110°C. The temperature within the bag is shown by the bulb-shaped pointer. As the temperature reaches 110°C., air will be bled in above the bag to cool the temperature to 110°C., the set point. This horn will also blow if there is a low pressure differential (about 1/2-1 in.) or a high pressure differential between the inside of the bag and the outside of the bag.

8. When atomizing air temperature is over 150°C., set the burner temperature on instrument A to 150°C., press the start button on the gas burner and hold in the button until the red light goes off and the green light goes on. The green light indicates that the producer gas has been ignited and the burner is operating. The temperature of the

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incoming gases from the burner will be shown on recording thermometer A. At temperatures lower than about 450°C., the temperature of the gases may be somewhat over the set point. Increase the set point temperature every 2-3 minutes by 50°C. so as to gradually heat up the dryer shell. The present final operating temperature on instrument A is 450°C.

9. The temperature at the bottom of the dryer is recorded by the pen of instrument C. When this temperature rises to about 90°C., slowly reduce the green needle on instrument C until three-way valve M just starts to open. This can be determined by observing the pressure of the air gauge at the bottom west side of instrument C. Initially this pressure is at 0 but should be increased to 3-4 pounds after the dryer has heated to 90°C. and the green needle has been partly lowered. Gradually decrease the green needle at such a rate that the temperature within the dryer will not drop back but will increase to 110°C. and meet the green needle at this point. The pressure on the air gauge controlling the three-way valve M will increase to approximately 10 pounds. The temperature will hold at 110°C. and should draw a straight line on the chart. If the dryer temperature rises much above the set point of 110°C. (say 135°C.-140°C.), this indicates a plugged slurry needle and a shutdown will probably have to be made and the needle cleaned out with a rod. This rise of temperature is due to failure to deliver slurry because of the plugged slurry needle, the absorption of heat required to evaporate the water in the slurry having a cooling effect. If the recorder pen shows a fluctuating temperature, this indicates a partially plugged slurry needle. Occasionally this plug can be removed by moving the green needle down to 50°C. for a few seconds and then returning it to 110°C. This must be

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done only for a few seconds as this manipulation tends to throw too much wet slurry into the dryer. When the temperature within the dryer is steady, place the red needle at the same temperature as the pen indicates and activate the horn alarm system on the operating panel and the third floor. This alarm system will sound if the temperature within the dryer varies more than 5°C. from the red needle set point, either up or down. Increasing temperature of the gas burner will cause fluctuations and this instrument will have to be watched closely until the system is in equilibrium. Start the vibrator. At present, we run the vibrator the entire period of drying but this may not be necessary.

10. Check flow meter L on the side of the collector frequently during operation of the dryer. This indicates rate of flow of gases through the system and is measured at an orifice plate in the duct from the gas burner just before it reaches the atomizing head. A flow rate of 1.5 ± 0.1 is normal. If the flow drops to 1.0 or lower, an abnormality is indicated, probably a dryer stoppage and the system probably will shortly stop operating. If such a low reading is obtained, check with supervision as to whether to shut down and clean out the dryer.

11. Tap the cone of the collector frequently with a wooden mallet to knock down dye so that it will pass through the rotary air lock valve to the crude dye barrel. The product is in the form of a fine dust and tends to stick to the collector cone.

12. A red button on the control panel is for emergency shutdown of the entire system. If this button is pressed, every part of the dryer will shut down except the exhauster fan

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which will run for several minutes to purge the system. The collector will continue to run.

13. Record on log sheets the dryer performance and the record of occupancy of the dryer.

14. Check exit gases going through the barrel on the roof with a wet spot paper. No color should be observed.

15. When crude dye barrels are full, weigh and take a sample in a bottle. Take the same size sample from each barrel and composite. Head the barrels, affix paper labels showing name, charge numbers, gross, tare and net weights and move barrels to first floor for shipment. Most materials dried will be shipped to "Ponsol" Standardizing No. 2 East Platform.

When drying is completed, drain the lines to a barrel, head the barrel, label with name and charge number and store for future production. Do not mix with future production until authorized by supervision. Shut down the dryer in the following manner.

16. If the run has been completed, add 50-100 pounds of water to the barrel or T-50 to flush out lines and "dry" this water in order to flush slurry out of the lines and avoid loss. If midway in the run, this flushing out is disregarded.

17. Press the stop button on the slurry pump. The pump red light should come on and the green light go off. Slurry or water should stop recycling.

18. Press the stop buttons on the electric heater and the gas burner. The corresponding green lights should go out and the red lights should come on.

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19. Shut off the alarm system of instrument C. Increase the green pointer of instrument C to 250°C. This will stop slurry feed to the dryer. Open the bleed valve below three-way valve M on the slurry tube so that slurry will be pulled down through the slurry tube and not dry out and pack the tube. This bleed valve must be shut after a few minutes to avoid possible oversight later on start-up with resultant loss.

20. Shut off the atomizing air at the valves on the control panel at E. Pressure should drop to 0.

21. Allow the exhauster fan to run until the temperature within the dryer (pen of instrument C) has dropped to 75°C. or lower.

22. Knock dry color from inside the collector cone and when all color has discharged, shut off the rotary air lock, ring blower and ring drive (H, I and J). Shut down the vibrator.

23. Shut down the exhauster fan. The green light should go off and the red light should come on.

24. Observe temperature within the dryer (pen of instrument C) for excessive temperature rise which would indicate a fire. A slight rise is normal due to the hot dryer walls, but this should subside in a few minutes.

25. Shut off the jacket air at valve F on the control panel. Pressure should drop to 0.

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Cleanup

In case dryer cleanup is necessary prior to completion of a run due to excessive material on the walls (over 1/4-1/2 in. in thickness or around the orifice plate), this may be done by opening the port on the side of the dryer and scraping down the walls. When doing this, run the exhauster to pull away dust. The opening in the collector leading to the exhauster will have to be partially constricted with a reducing orifice so as to avoid exhauster failure due to overloading of the exhauster.

Scrapings are reserved, sampled and shipped separately from the charge.

When a run is completed, the lines to the dryer should be drained free of color and this slurry saved. Wash the lines and T-50 free of color with warm water.

Remove the top of the dryer and scrap down color to a barrel. Sample and ship scrapings separately. Remove bag and hold for the next lot of the same color. The bag may be vacuum cleaned but must not be washed as it will shrink. Wash the dryer well, including the atomizing head. Be sure the atomizing assembly is centered properly before reassembling the dryer. Be sure no dye dust has diffused back into the gas burner duct; if so, it must be cleaned out to avoid fire on start-up.

Remove the clean out port on the duct between the dryer and the collector and wash out. Wash out the line from the rubber diaphragm valve to the bottom of the collector.

Reassemble the dryer using a dry felt bag.

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Precautions

1. Occasionally the entire system will kick out. Immediately press the relay on the collector blower (exhauster) and try to start-up the system again.

2. Watch the manometer K which indicates bag failure. At least 2 in. differential should be obtained. Even 2 in. has been obtained with an 8-in. gap of the bag not held by the retaining ring joining the bag to the collector. On this occasion, color was present on the collector floor outside of the bag, in the barrel on the roof and in the exhaust gases on the roof.

3. Watch the flow meter L. Before feeding, it may be low, even 0.5; but after arriving at proper temperature and feed rate, it will probably be 1.0-1.5.

4. The "Fyr Eye" on the burner mixing chamber has given difficulty. At one time, even tapping the box housing the "Fyr Eye", control instruments would shut down the dryer system. This was due to improper adjustment of a relay.

5. The shutoff instrument N in the line from the Spencer blower supplying air to the gas burner has given difficulty. This instrument measures pressure in the air system and shuts down the dryer if air pressure is below the set point. This instrument is usually set at 0.5 pounds per square inch.

6. If wet or taffy-like material issues from the star valve to the crude dye barrel, it is indicative of improper adjustment of the atomizing head assembly with resultant plastering and build-up of partially wet material on the dryer walls. This

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material will in part then drop off and be observed with the dry crude.

7. Marasperse CB is more flammable than Marasperse B although it has been dried. Never confuse these two products.

HAZARDS

Fire

This usually results from plastering of color on the orifice near the atomizing head. This dye is heated to incandescense by the hot burner gases and glowing dye particles can be observed dropping into the crude dye barrel. If this occurs, remove the crude dye barrel and substitute an empty barrel. Shut off the gas burner. Shut off the electric heater. Shut off the slurry feed and turn water into the Moyno pump from the water line attached to the Moyno pump. Shut return line to slurry tank to avoid dilution of slurry. Turn the green needle down to 25°C. on instrument C. Allow air to continue passing through rotameters E and F. This will spray water into the dryer shell and has put out fires. Allow the exhauster to continue running. It will be necessary to clean up the dryer in case of such a fire. Such a fire may also burn holes in the felt bag necessitating its replacement.

Yield Data

Output from the dryer is influenced by many factors. The heat transfer of the dye particle, the amount of water which must be removed from the slurry, the temperature at which the product can be dried (gases from the burner), the temperature necessary in the dryer chamber to get a dry product and so forth. Maximum output to date has been "Latyl"

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Violet B which has been dried at the rate of 100 pounds of crude per hour using a 40-45 per cent solids slurry.

NOTES

1. Atomizing air at the nozzle should be 9 ± 1 pounds. This corresponds to about 12 pounds at the operating panel. With a 0.52-in. diameter nozzle, this will deliver the same weight of air at subsonic velocity as a 0.48-in. in diameter nozzle at 15 ± 1 pounds pressure on the panel or 12-13 pounds at the nozzle which is sonic velocity. Output is considered the same under the above two conditions. We are restricted from operating at sonic velocities. The patent stituation is as follows:

a. The Legal Department has ruled that U.S. patent 2,297,726, which claims atomizing air at sonic velocity is invalid.

b. Since we believe we can operate with little or no penalty as to output at less than sonic velocity, we have directed no request to the Patent Board to ignore this patent.

c. We consequently at this time are not free to operate at sonic velocities.

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