June 12, 1962 E. W. COMINGS ETAL 3,038,533

DRYING PROCESS AND APPARATUS FOR HEAT-SENSITIVE MATERIALS

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Fig. 1.

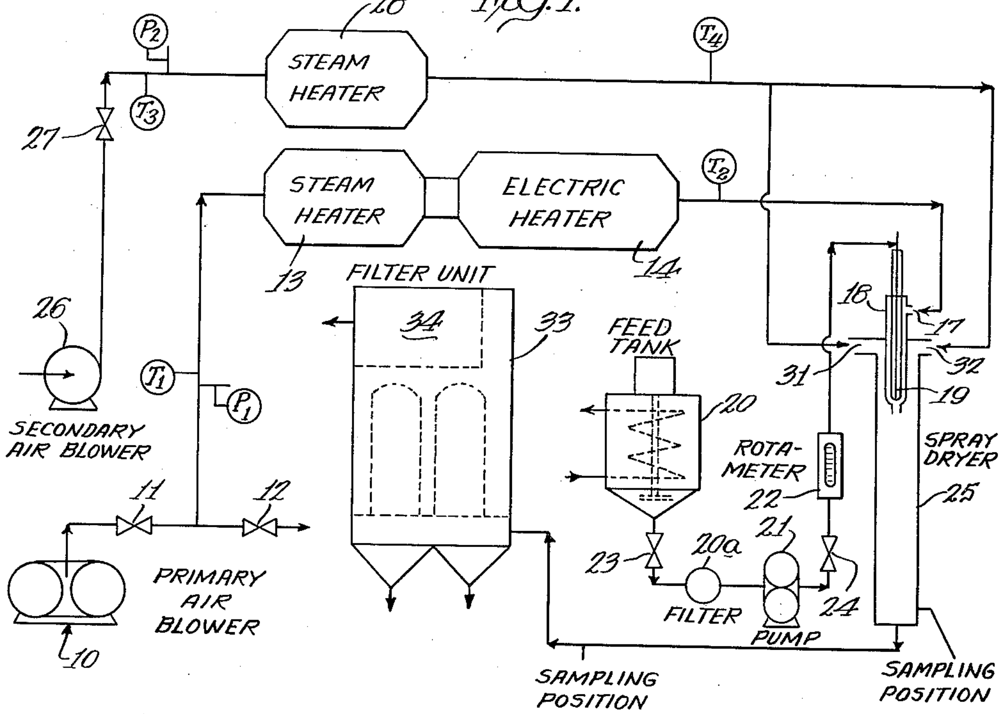

Fig. 2.

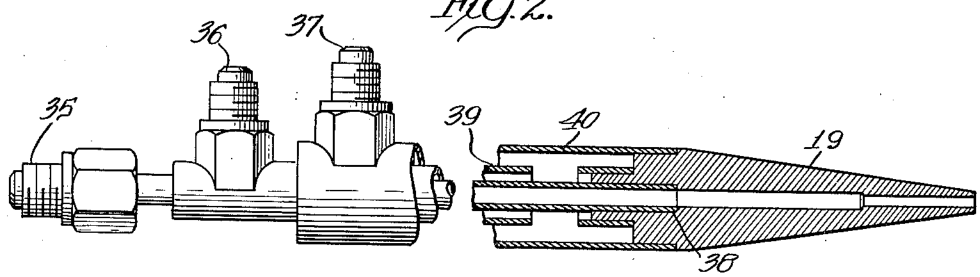

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Fig. 3

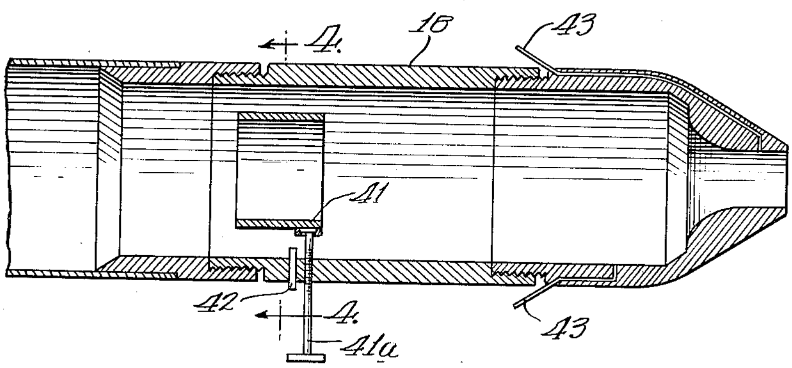


Fig. 4

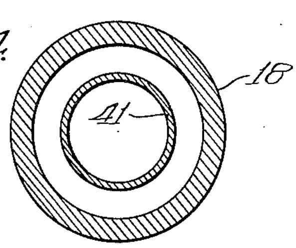
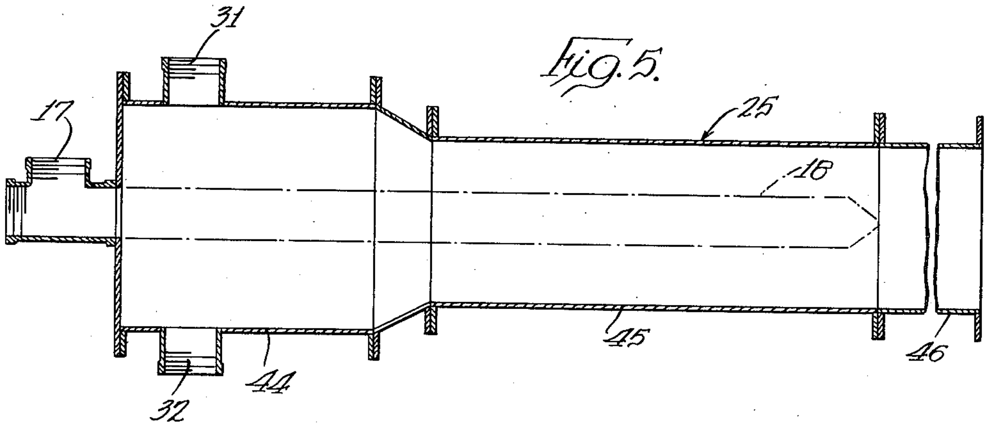


Fig. 5



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DRYING PROCESS AND APPARATUS FOR HEAT-SENSITIVE MATERIALS

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3 Claims. (Cl. 159—4)

This invention relates to a method of removing materials from an aqueous medium or vehicle and more particularly relates to a method of separating heat-sensitive materials in dry particulate form from an aqueous suspension or solution of the materials.

The present invention is an adaptation of the known techniques of spray drying which render that process suitable for handling extremely heat-sensitive materials through a complete drying period with no deterioration of the materials. Atomization to produce a minimum particle size is achieved with a two-fluid nozzle in which the aqueous medium and entrained solids move through an orifice into a stream of air which in an area of extreme turbulence converts the medium and entrained material into minute particles. From the zone of turbulence, where dispersion and initial volatilization of the aqueous medium have occurred, the dispersed particles move into a second cocurrent stream of air where final drying, if any, is completed at a lower temperature. The effect of the foregoing is to achieve a substantial drying of the dispersed particles while minimizing the residence time of the medium in the higher temperatures of the primary or dispersing air. The high ratio of surface to mass in the dispersion makes for high evaporation rates at an elevated temperature which would be destructive of the material, if residence time were of longer duration.

Further description of the invention can be had by reference to the accompanying drawings in which:

FIGURE 1 is a schematic showing of a suitable arrangement of equipment adapted to the practice of the process of this invention;

FIGURE 2 is a partial cross-sectioned view of the liquid injector for handling the aqueous medium to be converted into a dry particulate form;

FIGURE 3 is an elevational view of the nozzle that surrounds the liquid injector of FIGURE 2 and handles the stream of primary air employed as a dispersant or atomizer of the liquid medium;

FIGURE 4 is a cross-section of the nozzle of FIGURE 3 taken at line 4—4 in FIGURE 3; and

FIGURE 5 is a lengthwise cross-section of the housing through which a second stream of air is introduced behind the nozzle to serve as a stabilizer for the cocurrent primary air stream and solids issuing from the nozzle.

By specific reference to FIGURE 1, it can be seen that a blower **10**, capable of delivering air at 15 p.s.i.g. and 300 s.c.f.m., moves primary air through valve **11** with a portion of the air being intermittently vented at valve **12**, if desired, for humidity measurements. The primary air then passes through a heat exchanger **13** and through an electric air heater **14**. Suitable pressure and temperature measuring stations are located at P_1 and T_1, respectively, with a temperature control station being located at T_2. Using the heat exchanger alone, primary air temperature of 300° F. is obtained. This temperature may be elevated to substantially 600°–700° F. through the added effects of heater **14**. Air passing through nozzle inlet **17** on nozzle **18** constitutes the primary dispersing or atomizing gas for the aqueous medium entering injector **19** from liquid feed tank **20** through filter **20a**, metering pump **21** and rotameter **22**. Shut-off valves **23** and **24** are also provided.

As the primary air and liquid meet and pass rapidly through the choke orifice forming the outlet of nozzle **18**, the liquid is finely dispersed to form droplets having entrained or dissolved solids. Such droplets are discharged from nozzle **18** into the interior of a tubular dryer **25** where they enter a stabilizing cocurrent stream of secondary air having a temperature considerably less than the temperature of the primary air.

Secondary air is supplied from a separate blower **26**, through valve **27** and a steam heater **28** capable of elevating its temperature to substantially 300° F. The blower **26** may be of an order capable of delivering air up to 1400 s.c.f.m. at substantially 2 p.s.i.g. Temperature and pressure measuring stations T_3 and P_2 are employed along with a temperature control station T_4, as indicated. Secondary air is discharged into the dryer **25** via two inlets **31** and **32** disposed on opposite sides thereof adjacent nozzle **18**. The travel of the gas and entrained material is substantially unidirectional longitudinally of the drier chamber **25**, and so is the flow of secondary air which envelops it. The secondary air serves to cool the dry and dispersed particles effectively. As a commingled body, the moist atmosphere and dried solids move into a tank or vessel **33** from which separated solids are removed at the bottom by bag filter means, while gases and moisture are discharged at the top through an air cleaning deep-bed filter **34**. A cyclone may also be used in place of this vessel.

Injector **19**, as shown in FIGURE 2, comprises a feed inlet **35**, cooling water inlet **36** and cooling water outlet **37**. Feed inlet **35** is connected by tubing **38** to the discharge end of the injector, tubing **38** being jacketed by a concentric pipe **39** leading cool water from inlet **36** to be discharged through concentric pipe **40** returning to cooling water outlet **37**. The purpose of the cooling water is to preclude over heating of the liquid to be treated during the period that it is within the body of the nozzle **18** which transports the highly heated primary air for atomization. In practice, feed water conveniently obtainable at 50° to 60° F. will leave the cooling jacket at temperatures on the order of 120° F. depending on various operating conditions. While preventing over-heating that would destroy or cause deterioration of the entrained solids, the liquid receives enough heat as it is injected into the primary air to raise its temperature to the wet-bulb temperature of such atomizing air so that evaporation proceeds immediately.

FIGURE 3 shows nozzle **18** with the injector **19** of FIGURE 2 removed, the injector **19** being inserted through a packing gland at the rear of the nozzle and supported by a centering ring **41** adjustable by means of an adjusting screw **41a**. The centering ring is also shown in the end view, FIGURE 4. A thermocouple **42** and pressure taps **43**, **43** are illustrated in FIGURE 3 for measuring temperature and pressure. Nozzles used in the practice of this invention are extremely sensitive to proper centering of the injector to produce uniform flow through the nozzle and avoid build-up of particulate material on the hot interior surfaces.

Preferably, drying chamber **25**, as shown in FIGURE 5, has a uniformly smooth inner surface which can be fabricated from sheet metal by rolling and butt welding of the seams. Air at a temperature in the range of 100° to 200° F. enters through the secondary air inlets **31** and **32**, moves through the entrance section **44** and approaches section **45**, passes the tip of nozzle **18** shown in broken lines, and enters the drying section **46** where commingling of the hotter primary air, dispersed medium and suspended materials occurs.

An apparatus has been constructed to spray dry heat-sensitive materials in accordance with the aforementioned characteristics and conditions. This apparatus has been used to run the representative examples set forth below. The drying chamber **25** is of substantially uniform circular cross-section having an internal diameter of 8 inches. Axially positioned within the drying chamber is a nozzle **18** having an external diameter of about 2.5 inches and tapering, at its forward end, to an orifice diameter of 0.65 inch. The drying chamber has an effective length of about 6 feet downstream of the nozzle orifice wherein the particles become dry and the air streams commingle.

Materials to which the present technique can be applied are exemplified by such products as enzyme extracts, hormone extracts and beverages such as orange juice, coffee, milk and like materials. In the successful application of the described equipment to the separation of suspended heat-sensitive biologicals, it is essential that the material be in contact with the highly heated atomizing air for an extremely short period of time on the order of 0.001 to 0.01 second. Atomizing air at temperatures up to 700° F. moves past the tip of the liquid injector at average velocities up to 1300 ft./sec. The liquid feed for such operations is under pressures ranging from 0 to 60 p.s.i. with the dispersing gas being under pressures of 3 to 15 p.s.i. It can be readily appreciated that operation occurs under a relatively low pressure system which finally exhausts directly to the atmosphere so that pressures in the drying section are only slightly above atmospheric.

Further detailed description of the invention can be found in the following representative examples dealing with the separation of suspensions of *Serratia marcescens*, a gram-negative, facultative anaerobe and skim milk:

EXAMPLE I

Cell History

Organism: *Serratia marcescens.*
Culture medium: Tryptose broth.
Subsequent treatment: Centrifugation with culture placed in Sharples centrifuge and cells subsequently resuspended in diluent after storage at 4° C. for 48 hours.

Feed Stock

Diluent—
 Dextrin: 20 g./l.
 Ascorbic acid: 10 g./l.
Total cells/ml.: 242×10^9.
Viable cells/ml.: 5.5×10^9.
Percent viability: 2.3%.
pH: 6.8.
Total solids: 6.0%.

Inlet Dryer Conditions

Feed rate: 2.48 gal./hour. Feed temp.: 69° F.
Primary air temperature at dryer inlet: 400° F.
Primary air rate: 121 cu. ft./min. (60° F., 1 atm.).
Secondary air temperature at dryer inlet: 175° F.
Secondary air rate: 1225 to 1082 cu. ft./min. (60° F., 1 atm.).
Primary air velocity at nozzle throat: 1304 ft./sec.
Humidity of inlet air: 0.01145 lb. water/lb. air.

Outlet Dryer Conditions

Air temperature: 178° F.
Humidity of outlet air: 0.01474 to 0.01509 lb. water/lb. air.

Product Analysis

Total cells/ml. reconstituted product: 16.5×10^9.
Viable cells/ml. reconstituted product: 0.22×10^9.
Percent viability: 1.3%.
Viable recovery: 57%.

EXAMPLE II

Cell History

Organism: *Serratia marcescens.*
Culture medium: Tryptose broth.
Subsequent treatment: Centrifugation with Sharples centrifuge and immediate resuspension of mud in diluent.

Feed Stock

Diluent—
 Dextrin: 20 g./l.
 Ascorbic acid: 10 g./l.
Total cells/ml.: 55.0×10^9.
Viable cells/ml. 0.20×10^9.
Percent viability: 0.36%.
pH: 6.7.
Total solids: 4.5%.

Inlet Dryer Conditions

Feed rate: 2.48 gal./hour. Feed temp.: 87° F.
Primary air temperature at dryer inlet: 400° F.
Primary air rate: 117 cu. ft./min. (60° F., 1 atm.).
Secondary air temperature at dryer inlet: 150° F.
Secondary air rate: 1234 to 1162 cu. ft./min. (60° F., 1 atm.).
Primary air velocity at nozzle throat: 1301 ft./sec.
Humidity of inlet air: 0.00749 lb. water/lb. air.

Outlet Dryer Conditions

Air temperature: 154° F.
Humidity of outlet air: 0.01075 to 0.01093 lb. Water/lb. air.

Product Analysis

Total cells/ml. reconstituted product: 41.8×10^9.
Viable cells/ml. reconstituted product: 0.13×10^9.
Percent viability: 0.31%.
Viable recovery: 85%.

EXAMPLE III

Cell History

Organism: *Serratia marcescens.*
Culture medium: Tryptose broth.
Subsequent treatment: Centrifugation with Sharples and immediate resuspension of mud in diluent.

Feed Stock

Diluent—
 Dextrin: 20 g./l.
 Ascorbic acid: 10 g./l.
Total cells/ml.: 27.5×10^9.
Viable cells/ml.: 1.40×10^9.
Percent viability: 5.1%.
pH: 7.2.
Total solids: 5.0%.

Inlet Dryer Conditions

Feed rate: 2.49 gal./hour. Feed temp.: 65° F.
Primary air temperature at dryer inlet: 399° F.
Primary air rate: 121 cu. ft./min. (60° F., 1 atm.).
Secondary air temperature at dryer inlet: 123° F.
Secondary air rate: 1250 to 1152 cu. ft./min. (60° F., 1 atm.).
Primary air velocity at nozzle throat: 1310 ft./sec.
Humidity of inlet air: 0.00638 lb. water/lb. air.

Outlet Dryer Conditions

Air temperature: 129° F.
Humidity of outlet air: 0.00961 to 0.00985 lb. water/lb. air.

Product Analysis

Total cells/ml. reconstituted product: 49.2×10^9.
Viable cells/ml. reconstituted product: 2.50×10^9.
Percent viability: 5.1%.
Viable recovery: 100%.

EXAMPLE IV

Cell History

Organism: *Serratia marcescens.*
Culture medium: Tryptose broth.
Subsequent treatment: Centrifugation of 20 hr. culture.

Feed Stock

Diluent—
 Ascorbic acid: 10 g./l.
 Dextrin: 20 g./l.
Total cells/ml.: 6.6×10^9.
pH: 7.0.
Total solids: 2.9%.

Inlet Dryer Conditions

Feed rate: 2.49 gal./hour. Feed temp.: 124° F.
Primary air temperature at dryer inlet: 504° F.
Primary air rate: 114 cu. ft./min. (60° F., 1 atm.).
Secondary air temperature at dryer inlet: 123° F.
Secondary air rate: 1097 to 951 cu. ft./min. (60° F., 1 atm.).
Primary air velocity at nozzle throat: 1385 ft./sec.
Humidity of inlet air: 0.00427 lb. water/lb. air.

Outlet Dryer Conditions

Air temperature: 143° F.
Humidity of outlet air: 0.00801 to 0.00840 lb. water/lb. air.

Product Analysis

	Cyclone I	Cyclone II
Total cells/gm. dry product	$1,360 \times 10^9$	$1,409 \times 10^9$
Viable cells/gm. dry product	982×10^9	$1,419 \times 10^9$
Viable recovery, percent	72.1	100.5
Final moisture content Wt. percent	4.5	4.8

EXAMPLE V

Feed Stock

Skim milk.
Total solids: 9.28%.

Inlet Dryer Conditions

Feed rate: 3.0 gal./hr. Feed temp.: 84° F.
Primary air temperature at dryer inlet: 500° F.
Primary air rate: 99 cu. ft./min.. (60° F., 1 atm.).
Secondary air temperature at dryer inlet: 125° F.
Secondary air rate: 1199 to 1095 cu. ft./min. (60° F., 1 atm.).
Primary air velocity at nozzle throat: 1190 ft./sec.
Humidity of inlet air: 0.00985 lb. water/lb. air.

Outlet Dryer Conditions

Air temperature: 131° F.
Humidity of outlet air: 0.01386 to 0.01421 lb. water/lb. air.

Product Analysis

	Cyclone I	Cyclone II
Final Moisture Content, Wt. percent	3.75	3.8

EXAMPLE VI

Feed Stock

Skim milk.
Total solids 9.28%.

Inlet Dryer Conditions

Feed rate: 3.5 gal./hr. Feed temp.: 76° F.
Primary air temperature at dryer inlet: 502° F.
Primary air rate: 99 cu. ft./min. (60° F., 1 atm.).
Secondary air temperature at dryer inlet: 140° F.

Secondary air rate: 1140 to 1092 cu. ft./min. (60° F., 1 atm.).
Primary air velocity at nozzle throat: 1265 ft./sec.
Humidity of inlet air: 0.00933 lb. water/lb. air.

Outlet Dryer Conditions

Air temperature: 141° F.
Humidity of outlet air: 0.01361 to 0.01370 lb. water/lb. air.

Product Analysis

	Cyclone I	Cyclone II
Final Moisture Content, Wt. percent	4.31	4.52

Having thus described the invention, what is claimed is:

1. A method for producing heat-sensitive material in particulate form from a liquid medium in which it is contained which comprises projecting a current of relatively dry secondary air at a temperature not in excess of 200° F. longitudinally through a tubular drying chamber, introducing a jet of said liquid medium axially into said drying chamber in the direction of flow of said secondary air, projecting a co-current of primary air at a temperature of from about 300° F. to 700° F. in contact with said jet at an acoustic velocity up to about 1385 feet per second, said primary air being of sufficient rate to both break up the jet and disperse it into fine droplets and to simultaneously add to said droplets the heat of vaporization of the liquid, said droplets being evaporated to dryness in said tubular drying chamber to form particles of said heat sensitive material and being carried along in the combined primary and secondary air moving through said tubular drying chamber, said secondary air stream having a flow of from at least 8 to 15 times that of the primary air flow sufficient to prevent recirculation of dry particles to the hot primary air stream.

2. A method for producing heat sensitive material in dry particulate form from a liquid medium containing said material which comprises projecting a current of relatively dry secondary air at a temperature not substantially above 200° F. longitudinally through a tubular drying chamber, projecting a jet of said liquid medium initially at a temperature not substantially above 124° F. substantially axially into the said drying chamber into a venturi-like orifice the outlet of which discharges along substantially the longitudinal axis of said tubular chamber, projecting a co-current of primary air at a temperature of from about 300° F. to 700° F. through said orifice in contact with said jet at a velocity of from about 1190 to 1385 feet per second, said primary air being of sufficient rate to both break up the jet into fine droplets and to impart to said droplets the necessary heat of vaporization, said droplets being evaporated to dryness in said tubular drying chamber to form particles carried along in the combined primary and secondary air streams, said secondary air stream flowing in volume of from at least 8 to 15 times the volume of the heated primary air to prevent recirculation of dry particles to the hot primary air stream and at no time becoming heated substantially above 200° F.

3. A spray drying apparatus adapted for drying of heat-sensitive materials at very high temperatures and velocities, said apparatus comprising: an elongated cylindrical drying chamber having an inlet end and an outlet end; a nozzle assembly axially aligned with said chamber and projecting a short distance within the inlet end thereof, said nozzle assembly terminating in an orifice within said chamber and adapted to project a stream of a first gas substantially axially therein; a liquid injector spaced axially within said nozzle assembly so as to inject a stream of liquid containing the heat-sensitive material toward said orifice within an annular stream of said first gas whereby said liquid stream will be broken up into

fine droplets and mixed with said first gas as the latter passes beyond said orifice into said chamber at velocities up to about 1385 feet per second, said liquid injector having a cooling jacket about substantially its full length including an inlet and outlet for recirculating a fluid coolant; an annular passageway about said nozzle assembly extending from said inlet end of said cylindrical chamber to project a second gas axially of said chamber co-current with and enveloping said primary gas and liquid streams as the latter pass from said orifice, said annular passageway being defined by the wall of said chamber and said nozzle assembly therein; a first gas impelling means connected to deliver a flow rate of said first gas to said nozzle assembly; a second gas impelling means connected to deliver a much larger flow rate of said second gas to said annular passageway, said flow rate of said second gas being from 8 to 15 times the flow rate of said first gas at normal atmospheric conditions; a first heat exchanger connected in series with said first impelling means and said nozzle assembly for controllably heating the first gas to within a range of 300° to 700° F.; a second heat exchanger connected in series with said second impelling means and said annular passageway for controllably heating the second gas up

to about 300° F.; a source of liquid containing the heat sensitive material connected to said liquid injector; and means to control the rate of flow of said liquid to said injector.

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