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PROVISIONAL SPECIFICATION

IMPROVEMENTS IN OR RELATING TO SPRAY DRYING TECHNIQUE WITH SPECIAL APPLICATION TO ELECTROLYTIC RECOVERY OF MANGANESE, MISCH METAL AND THEIR LIKE.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RASHI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTRERED BY INCORPORATION UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860).

The following specification describes the nature of this invention.

THIS IS AN INVENTION BY GOPALRAM JANAKRAMAN, Poonamalal Sridhar Deshram and Verkaragamaya Aravamudhan of the Central Electrochemical Research Institute, Karikudi-3, S. RAILWAY, MADRAS 95, STATE, INDIA, ALL INDIAN CITIZENS.

This invention relates to improvements in or relating to spray drying technique with special application to electrolytic recovery of magnesium, misch metal and the like and has reference to preparation of anhydrous salts by spray drying technique with special application to extractive metallurgy of magnesium, misch metal or the like.

Hitherto it has been proposed to have atomisers with solid shafts, a sealed shaft entry and plain cup atomizing wheels in conventional spray driers with large heights of the drier chamber.

This is open to the objection that at high temperatures, say 500°C and above, they develop mechanical troubles and economy is affected.

The object of this invention is to obviate these disadvantages. This has been achieved according to the present invention by internal cooling of the atomizer shaft, a free gasless entry of the shaft into the drier chamber which has a large 0 ratio and a 'dispersion' feature in the atomizer wheel.

The success of fused salt electrolysis for the production of metals like magnesium, misch metal and the like depends to a great extent on the production of anhydrous chlorides by a simple technique. A number of procedures have been evolved from time to time in which a protective atmosphere is formed while the chlorides are dehydrated such as through the use of hydrochloric acid gas, ammonium chloride or the like. Dehydration of inorganic chlorides in thin layers preferably in vacuum and drying chloride solutions by sublimed combustion upto a certain stage have also been reported. Most of these procedures involve the use of costly equipment and/or cost consuming. The cost of the fuel to dehydrate the chlorides is also very high and these are usually batch processes.

The idea of spray drying the inorganic chlorides of proper concentration in the presence of hot combustion gases is not new. The novelty in the spray drier designed by us comprises of:

1. The internal cooling of the shaft of the atomizer which is capable of high speeds (well above 3,000 r.p.m. and in fact we have used 15,000 r.p.m. and temperatures of 500°C and above). This simplifies the design of the atomizer and its drive. The shaft is cooled internally by a bore in which the coolant pipe is held stationary. The discharge from the coolant pipe fills up the shaft bore and then after abstracting the heat from the shaft escapes out by sliding over a small dispenser arrangement screwed on to the top end of the rotating shaft itself. The stationary block accommodating the rotating shaft houses a sump in which the coolant dispelled by the dispenser is collected and drained through at the bottom. By virtue of the shaft cooling, bearings are not heated up even in prolonged runs.

2. Elimination of a stuffing box arrangement or gland or back for introducing the shaft of the atomiser into the spray chamber. The said arrangement is source of trouble especially in temperature well above 200°C. The design as described herein includes a small clearance around the shaft. A vane arrangement fitted on to the shaft itself just above the clearance, spins also at the same speed as that of the shaft itself, is so placed that an air cushion is created under the fan and the leakage or loss of dried product is avoided. The vane arrangement is suited to the pressure of the drying chamber.

3. The special design of the atomizer: Though atomizer wheels (inverted saucer-like cups are commonly employed this invention covers the incorporation of a 'dispersion' feature in the atomizer wheel as shown in Fig. 5 of the accompanying drawing. One or more angular cuts ranging from 30° to 120° and best measured 60° creates a sudden blockade to the centrifugal spreading layer of fluids and the layer is dispersed and flung to the top cup from where the final stages of atomisation occurs.

To give a typical example, an atomizer comprising the above features was used in spray drying a magnesium chloride solution of above 40 per cent concentration in admixture with small amounts of sodium and potassium chlorides into a spray chamber which was heated internally to a temperature well above 500°C by a continuous stream of combustion gases. This spray chamber itself is different from the conventional spray chamber in that it has a conical bottom, the ratio of D1/D2 (diameter of the spray chamber to the height of the spray chamber is large) and above, thereby introducing the fuel economy in evaporation.

A mechanical sweeping arrangement provided at the bottom of the spray chamber does not allow the sprayed dried particles to settle, thus enabling their entry into the cyclonic catcher (refer sketch).

The atomizer and the drive incorporating the features in conjunction with the spray chamber mentioned in this patent worked continuously for several days, without trouble.

Drawings:

1. Atomizer Wheel
2. Spray Chamber
3. Atomizer Assembly

The following typical examples are given to illustrate the invention using magnesium chloride solution:

**Example 1**

<table>
<thead>
<tr>
<th>Inlet temperature</th>
<th>500°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outlet temperature</td>
<td>200°C</td>
</tr>
<tr>
<td>Chamber pressure</td>
<td>60 mm. of water</td>
</tr>
<tr>
<td>Deyhydrated product</td>
<td>MgCl2·6H2O</td>
</tr>
</tbody>
</table>

Price: TWO RUPEES.
The following are among the noteworthy features of this invention:

1. The design of a simple atomiser with belt driving for spraying corrosive liquids like magnesium chloride at high temperatures to get dehydrated products suitable for electrolysis.

2. A spray chamber in which the ratio of diameter to height is greater than two and which is also provided with a bottom raking arrangement, which has no conical bottom, to collect the spray dried product in the cyclone chamber.

R. BHASKAR PAL,
Patent Officer,
Council of Scientific and Industrial Research
Date 6th day of November 1964.

COMPLETE SPECIFICATION.

IMPROVEMENTS IN OR RELATING TO SPRAY DRYING TECHNIQUE WITH SPECIAL APPLICATION TO ELECTROLYTIC RECOVERY OF MAGNESIUM, MIESCH METAL AND THE LIKE.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, BAYI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860).

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.

This is an invention by Gopalram Janaikraman, Founannyali Sinivasra Dusenil and Venkatachala Arunachal of the Central Electrochemical Research Institute, Koramangla-3, S. Bangalore, Madras State, India, all Indian citizens.

This invention relates to improvements in or relating to spray drying technique with special application electrolytic recovery of magnesium, miesch metal and the like and has reference to preparation of anhydrous salts by spray drying technique with special application to extractive metallurgy of magnesium, miesch metal and the like.

Hitherto it has been proposed to have atomisers with solid shafts, a sealed shaft entry and plain cup atomising wheels in conventional spray dryers with large heights of the drier chamber.

This is open to the objection that at high temperatures, say 500°C and above, they develop mechanical troubles and economy is affected.

The object of this invention is to obviate these disadvantages. This has been achieved according to the present invention by internal cooling of the atomiser shaft, a free gasketless entry of the shaft into the drier chamber which has a large H/D ratio and a 'dispersion' feature in the atomiser wheel.

The invention includes within its scope an atomiser comprising an atomiser, a drive and a spray drier chamber wherein are provided an internally cooled atomiser shaft, a free gasketless entry of the shaft into the spray drier chamber which has a large H/D ratio and a 'dispersion' feature in the atomiser wheel.

The success of used salt electrolysis for the production of metals like magnesium, miesch metal and the like depends to a great extent on the production of anhydrous chlorides by a simple technique. A number of procedures have been evolved from time to time in which a protective atmosphere is formed while the chlorides are dehydrated such as through the use of hydrochloric acid gas, ammonium chloride or chlorine. Dehydration of inorganic chlorides in thin layers preferably in vacuum and drying chloride solutions by submerged combustion up to a certain stage have also been reported. Most of these procedures involve the use of costly equipment and/or time consuming. The cost of the fuel to dehydrate the chlorides is also very high and these are usually batch processes.

The idea of spray drying the inorganic chlorides of proper concentration in the presence of hot combustion gases is not new. The novelty in the spray drier designed by the inventors comprises of:

1. The internal cooling of the shaft of the atomiser which is capable of high speeds (wall above 3,000 r.p.m. and in fact we have used 12,000 r.p.m. and temperature of 500°C and above). This simplifies the design of the atomiser and its drive. The shaft is cooled internally by a bore in which the coolant pipe is held stationary. The discharge from the coolant pipe fills up the shaft bore and then after absobing the heat from the shaft escapes out by sliding over a small dispenser arrangement screwed on to the top end of the rotating shaft itself.

The stationary block accommodating the rotating shaft houses a pump in which the coolant dispelled by the dispenser is collected and drained through at the bottom. By virtue of the shaft cooling, bearings are not heated up even in prolonged runs. An arrangement such as the one mentioned in this invention has not so far been published.

2. Elimination of a stuffing box arrangement or gland or bush for introducing the shaft of the atomiser into the spray chamber. The said arrangements are sources of troubles especially in temperatures well above 200°C. The design, as described in this patent, includes a small clearance around the shaft. A wave arrangement fitted on to the shaft itself just above the clearance, spins also at the same speed so that the shaft itself, is so placed that an air cushion is created under the fan and the leakage or loss of dried product is avoided. The wave arrangement is suited to the pressure of the drying chamber.

3. The special design of the atomiser: Though atomiser wheels (invented saucer like cups) are commonly employed, this invention covers the incorporation of a 'dispersion' feature as shown in the accompanying drawing. One or more angular cuts ranging from 30° to 120° and best near 60° creates a sudden blockage to the centrifugal spreading layer of fluids and the layer is dispersed and flung to the top cup from where the final stages of atomisation occur.

The invention has been illustrated with the help of drawings accompanying the provisional specification and the accompanying drawings wherein the description of the figures is as follows:

Provisional Specification:
Fig. 1: a plan view of the atomiser wheel;
Fig. 2: a sectional front elevation of the atomiser wheel;
Fig. 3: a spray drying chamber—sectional front elevation;
Fig. 4: sectional plan of the spray drying chamber.

Complete Specification:
Fig. 1: a plan view of the atomiser wheel;
Fig. 2: sectional front elevation of the atomiser wheel;
Fig. 3: revised version of spray drying chamber—sectional front elevation;
Fig. 4: sectional plan of revised version of spray drying chamber;
Fig. 5: atomiser assembly—sectional front elevation.
(ii) The parts represented by numerals in the drawings accompanying the complete specification are as follows:

1. Atomizer outer disc
2. Atomizer inner disc
3. Atomizer set screws
4. Spray drier inner chamber
5. Spray drier outer casing
6. Sweeper arm
7. Sweeper arm bearing housing
8. Sweeper arm shaft
9. Flexible coupling

(see Fig. 3 of drawings accompanying the Provincial Specification.)

10. Reaction gear unit
11. Spray drier intake pipe
12. Spray drier outlet pipe
13. Atomizer cooling pipe
14. Grip nut
15. Water sump lid
16. Coolant diffuser
17. Sump block
18. Atomizer shaft
19. Top pillar nuts
20. A, B and D.
21. Top pillar
22. Top bearing case
23. A and B. Pulley check nuts
24. Pulley
25. Bearing case cover screw
26. Bottom bearing
27. Bottom bearing house
28. Top bearing
29. Bottom pulser
30. A, B and D. Bottom pillar check nuts
31. Bottom bearing case top plate
32. Atomizer base plate
33. Cushion impeller
34. Cushion impeller check nuts
35. Atomizer disc
36. Atomizer disc nuts
37. Feed pipe casing
38. Feed pipe

(iii) Description of the spray drier assembly:

An outline of the spray drier assembly can be seen in Fig. 3 and 4. The spray drier unit consists of a drying chamber (4), a centrifugal atomizer assembly (Fig. 5) mounted on the top of the chamber and a sweeping device (6, 7, 8) at the bottom of the chamber. The spray chamber is a cylindrical metallic chamber with comparatively large diameter and low height and is provided with a conical inner casing (5). Between the outer wall (5) and inner wall (4) of the spray chamber the spray chamber heat insulation material is filled in. Two pipes (11 and 12) for the entry and exit respectively of hot combustion gases or hot air are provided tangentially at the top and bottom of the spray chamber.

The atomizer assembly is mounted centrally at the top of the spray chamber. The atomizer unit consists of an atomizer wheel which can be a plain disc or one of a design as indicated in Figs. 1 and 3. This particular atomizer consists of an outer piece (1) and an inner piece (2) in the shape indicated in Figures 1 and 2 screwed together by four nuts screws (3). The atomizer wheel is screwed to the atomizer shaft (18) which is mounted through two ball bearings (25, 27) and is connected to a driving mechanism through a V-groove pulley (22) fixed between the two ball bearings (25, 27). The atomizer shaft (18) has a hollow portion extending from the top to the position of bottom bearing (25) through which a cooling water pipe (13) is introduced and fixed by means of a grip nut (14). During the running of the atomizer, water is introduced through this pipe (13) which comes out and gets diffused by the diffuser (16) and falls into the sump (17) and gets out through an outlet pipe (not shown in the figure). The atomizer assembly is mounted on a bottom plate (31) which is screwed to the top of the spray chamber (4). The plate (30) holding the bottom bearing (25) and its casing (29) is mounted above the bottom plate (31) and is held in position by four pillars (28). Similarly the top bearing (27) and the cooling water sump (17) is held in position above the top plate (30) by means of four pillars (28). At the vicinity where the shaft (18) passes into the atomizer chamber (4) a cushion impeller (32) is fixed by means of two screws (33 A and 33 B) in order to prevent the hot flue gases leaking through the hole through with the shaft passes. The feed solution (34) to the spray drier is introduced in the centre of the atomizer wheel (34) by means of a tube (37) rigidly fixed by check nuts (35 A, B, C) in an outer pipe (36).

A sweeping mechanism consisting of a blade (6) mounted on a shaft (8) and driven (see Fig. 3 of drawings accompanying the provisional specification) by a suitable motor and reduction gear unit 10, Fig. 3, provisional specification at a very low revolution rate is provided at the bottom of the spray chamber (4).

(iv) Operation:

During the operation, the generator of hot flue gases (not shown) is switched on and introduced into the chamber through the inlet pipe (11) and the outlet temperature is allowed to increase to the desired limit. Simultaneously the atomizer driving mechanism is started and allowed to rotate at high speeds. When the desired temperature is reached the feed solution is introduced through the tube (37) at predetermined rates at the centre of the atomizer wheel (34). The sweeping device (6) is also allowed to rotate at a low revolution rate. The solution gets diffused into a fine spray and gets immediately dried due to the hot gases which passes at high velocity. The dried particles and resultant water vapour is carried along with the exit gases through the outlet (33) and reaches a cyclone chamber of conventional type (not shown) where the dried particles get separated from the gases.

To give typical example, an atomizer comprising the above features was used in spraying a magnesium chloride solution of above 40 per cent concentration in admixture with small amounts of sodium and potassium chlorides into a spray chamber which was heated internally to a temperature well above 500°C by a continuous stream of combustion gases. This spray chamber itself is different from the conventional spray chamber in that,

(1) it has no conical bottom;
(2) the ratio of D (diameter of the spray chamber to the height of the spray chamber) is large, say 2: 4 above, thereby introducing the fuel economy in evaporation;
(3) a mechanical sweeping arrangement provided at the bottom of the spray chamber does not allow the spray drier particles to settle, thus enabling their entry into the cyclone cutcher (refer sketch).

The atomizer and the drive incorporating the features in conjunction with the spray chamber mentioned in this invention worked continuously for several days, without trouble.

The following typical examples are given to illustrate the invention using magnesium chloride solution:

**Example 1.**

| Inlet temperature | 550°C |
| Outlet temperature | 250°C |
| Chamber pressure | 60 mm of water |
| Dehydrated product | MgCl₂·6H₂O |

**Example 2.**

| Inlet temperature | 525°C |
| Outlet temperature | 225°C |
| Chamber pressure | 60 mm of water |
| Dehydrated product | MgCl₂·6H₂O |

The following are among the noteworthy features of this invention:

(1) The design of a simple atomizer with belt driving for spraying corrosive liquids like magnesium chloride at high temperatures to get dehydrated products suitable for electrolysis.

Example 1.

Inlet temperature . . . 500°C
Outlet temperature . . . 250°C
Chamber pressure . . . 60 mm of water
Dehydrated product . . . MgCl₂·6H₂O

Example 2.

Inlet temperature . . . 525°C
Outlet temperature . . . 225°C
Chamber pressure . . . 60 mm of water
Dehydrated product . . . MgCl₂·6H₂O

The following are among the noteworthy features of this invention:

(1) The design of a simple atomizer with belt driving for spraying corrosive liquids like magnesium chloride at high temperatures to get dehydrated products suitable for electrolysis.
(2) A spray chamber in which the ratio of diameter to height is greater than two and which is also provided with a low or bottom arrangement, which has no conical bottom, to collect the spray dried product in the cyclone chamber.

We claim:

1. An improved spray drying process to obtain products suitable for use in the electrolytic recovery of magnesium, much metal and the like, characterized by internal cooling of the atomizer shaft, a free gasless entry of the shaft into the dryer chamber which has a large D: H ratio and a 'dispersion' means in the atomiser wheel.

2. A process as claimed in Claim 1, wherein is used an atomizer shaft capable of high speed (well above 3,000 r.p.m., e.g., 12,000 r.p.m. and temperatures of 500°C and above).

3. A process as claimed in Claim 1 or 2 wherein the shaft is cooled internally by a bore in which the coolant pipe is held stationary, the discharge from the coolant pipe fills up the shaft bore and thus after abstracting the heat from the shaft escapes out by sliding over a small disperser arrangement screwed on to the top end of the rotating shaft itself.

4. A process as claimed in any of the preceding claims wherein the stationary block accommodating the rotating shaft houses a ramp in which the coolant dispelled by the disperser is collected and drained through at the bottom.

5. A process as claimed in any of the preceding claims wherein a small clearance around the shaft and a vane arrangement fitted on to the shaft itself just above the clearance, spins also at the same speed as that of the shaft itself, is so placed that an air-cushion is created under the fan and the leakage or loss of dried product is avoided.

6. A process as claimed in any of the preceding claims wherein a 'dispersion' means is incorporated, as shown in Figure 3, sheet No. 3 of the provisional specification, and one or more angular cuts ranging from 30-120°C and best near 90° creating a sudden blockage to the centrifugal spreading layer of fluids and the layer being dispersed and flung to the top nip from where the final stages of atomization occur.

7. A process as claimed in any of the preceding claims wherein a magnesium chloride solution of 30 per cent concentration and above in admixture with small amounts of sodium and potassium chlorides is sprayed into a spray chamber heated internally to a temperature well above 500°C by a continuous stream of combustion gases.

8. A process as claimed in any of the preceding claims wherein the spray chamber—
(i) has no conical bottom;
(ii) the ratio of D: H (diameter of the spray chamber to the height of the spray chamber) is large, say 2 and above;
(iii) a mechanical sweeping arrangement provided at the bottom of the spray chamber (Fig. 6, sheet No. 3 of the provisional specification) does not allow the spray dried particles to settle, thus enabling their entry into the cyclone catcher.

9. Spray drying process to obtain products suitable for use in the electrolytic recovery of magnesium, much metal and the like substantially as herebefore described in the examples.

10. An assembly for carrying out a spray drying process as claimed in any of the preceding claims comprising an atomiser, a drive and a spray dryer chamber wherein are provided an internally cooled atomiser shaft, a free gasless entry of the shaft into the spray dryer chamber which has a large D: H ratio and a 'dispersion' feature in the atomiser wheel.

11. An assembly comprising an atomiser, a drive and a spray dryer chamber substantially as herebefore described.

R. Bhaskar Pai,
Patent Officer,
Council of Scientific and Industrial Research.

Dated the 6th day of September 1965.
SECTION AT 'AA'

**Fig. 2**

**Fig. 1**

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