

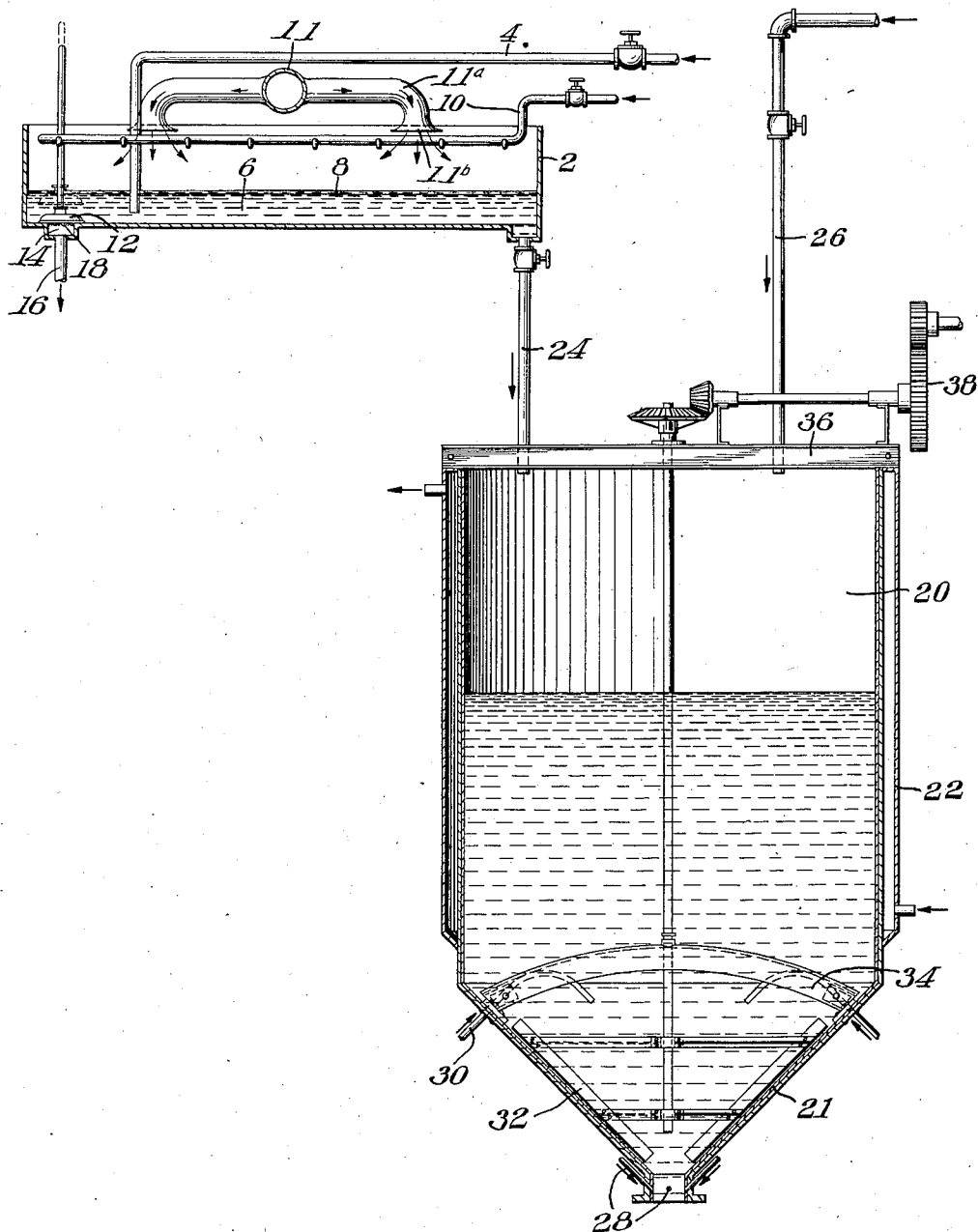
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CRYSTALLIZING PROCESS

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CRYSTALLIZING PROCESS

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This invention relates to the production of substantially uniform needle crystals of salts by initially preparing desirable seeding crystals which are thereafter permitted to grow to the required size in a solution of the salt. The invention is particularly applicable to the production of magnesium sulfate needle crystals.

The preferred magnesium sulfate crystals at the present time are relatively large and uniform needle crystals which pack sufficiently densely that an unduly large bulk is avoided for a given weight of the material. It has been found difficult to produce crystals of this type in one operation due to the fact that ordinarily undesirable salt crystals or other irregular crystals are present and are necessarily utilized as nuclei or seeding crystals.

In accordance with the present invention, seeding crystals are produced in one vessel and thereafter these crystals are preferably transferred to a different vessel particularly constructed for further crystal growth. During the formation of the seeding crystals the solution may desirably be maintained in shallow pans and under relatively quiescent and protected conditions which will produce substantial supersaturation before precipitation occurs. On breaking down the supersaturation, many fine needle crystals are produced, the crystals growing to only a slight extent in this solution due to the relatively small quantity of solution present.

It has been found that substantial supersaturation can be induced by spraying a thin layer of water gently over the surface of the Epsom solution in the precipitating pan. Supersaturation can then be broken in any desired way as by agitating the solution, introducing seeding crystals, spraying with water, or otherwise.

After seeding crystals are formed they may be transferred to a relatively deep crystallizing vessel more adapted to crystal growth than the shallow precipitating pans. A substantial quantity of the seeding crystals is mixed in the crystallizing tanks with a large body of slightly undersaturated Epsom solution, the solution and crystals being agitated, preferably by compressed air as the solution cools to the saturation point and the seeding crystals or nuclei grow at the expense of the solution.

The several steps of the process will be more fully understood in connection with the description of the construction and operation of the apparatus illustrated in the drawing. It will be understood that the apparatus disclosed is merely

illustrative and that it may assume various forms for carrying out the present invention.

In the drawing the shallow evaporating pan 2 is designed to receive a supply of magnesium sulfate or other liquor, supplied, for example, through the valved pipe 4. When used for precipitating magnesium sulfate crystals the solution, when introduced, should have a specific gravity of from 1.35 to 1.40 at 130° F. and the temperature of the solution should be somewhat above the saturation temperature of 120° F., for example, 122° to 130° F. The specific gravity may be as high as 1.40 and the temperature 170° F. The solution is fed into the vessel to a depth of from 6 to 10 inches.

Immediately thereafter the solution is sprayed with a light film of water 8. It has been found that this liquid remains as a separate water layer or blanket on the surface of the Epsom solution for a number of hours and that there is little dilution of the Epsom solution by reason of the thin layer of water thereon. The water may be applied as a thin film or mist by means of a hand sprinkler with nozzle attachment or, if desired, pipes and spray nozzles may be supplied as indicated at 10 for discharging a thin mist of water which falls gently upon and covers the Epsom solution without agitation. The contents of the tank are thereafter permitted to cool gradually for a few hours and until substantial supersaturation has appeared in the solution. This supersaturation can be broken in any preferred way, as stated above, for inducing the precipitation of magnesium sulfate crystals. It is usually found necessary to repeatedly break down supersaturation, which condition redevelops when the protected solution is permitted to remain quiescent for a period even after precipitation has begun.

Since in the shallow evaporating pans cooling is preferably accomplished without the use of cooling coils, or the like, it is desirable to produce a moderate draft of air over the surface of the liquid as the solution temperature declines. This can be accomplished by means of separate fans positioned adjacent the pans, or, as shown, the supply pipe 11 with branch outlets 11a may be utilized for this purpose. In case a plurality of pans 2 is utilized, the main conduit 11 can thus supply sufficient cooling air for the entire bank of pans. In order to reduce the disturbance of the solution to a minimum, the ends of the branch pipe 11a are preferably flared, as shown at 11b. In extremely hot weather the cooling air supplied through the pipe 11 may be refrigerated,

although for most normal temperatures this has been found unnecessary. Suction instead of pressure, can be supplied to the pipe 11 for causing a flow of air over the surface of the solution with fairly satisfactory results.

At the end of from 10 to 15 hours there will have developed a layer of thin needles from 2 to 4 inches deep in the bottom of the pan. The temperature of the solution at this time may have reached 100° F. and a specific gravity of 1.36. The solution is then removed from the precipitated crystals by a siphon or pump or, if desired, by removing the plug 12 from the opening 14 leading to a discharge pipe 16, provided with a screen 18. The withdrawn solution may then be further evaporated or fortified with additional crystals ready for further use in connection with the process.

The crystals are then preferably transferred to an empty clean crystallizer of the type indicated at 20. This vessel has a substantial height, greater than that of the pan 2, and is formed with cooling means shown as a jacket 22 through which cooling water or the like may be circulated for reducing the temperature of the solution. The crystals may be transferred from the pan 2 to the vessel 20 in any desired manner, for example, by hand or bucket conveyor, or, if desired, the tank may be connected with the crystallizer by means of a valved pipe 24, as shown, and substantially saturated Epsom solution introduced through the pipe 4 and utilized for washing the collected crystals into the tank 20.

An additional supply of Epsom solution is introduced into the tank 20 through the pipe 26 for producing growth of the seed crystals in the solution. While the nuclei may be added to the solution in the tank 20, it is found that more uniform results are attained by introducing the nuclei first and thereafter adding the solution. The Epsom solution introduced into the crystallizer should have a fairly high concentration, for example, about 68% magnesium sulfate, a specific gravity of 1.380 at 130° F. and a temperature, such that the solution is slightly undersaturated, for example, 124° to 130° F. for a solution of the above concentration. Immediately on introducing the seeding crystals and solution, agitation and cooling of the contents of the tank are begun. Agitation may be accomplished by introducing compressed air through the series of four or more pipes 28 positioned at the vortex of the cone-shaped bottom 21 of the tank and additional pipes 30 discharging at about the junction of the cone-shaped bottom with the vertical side walls of the crystallizer. While the air agitation is ordinarily sufficient to maintain the crystals in suspension during crystal growth, channeling may occur along the cone-shaped bottom 21 of the vessel. It is therefore desirable to provide some means for breaking up the channeling at this point. This may be accomplished by means of the mechanical agitator 32 supported on the cross-beams 34 and 36 and rotated slowly by means of gearing or the like as indicated at 38.

Agitation in the solution is continued for a number of hours as the solution is gradually cooled to a temperature of approximately 80° to 90° F., the solution at this temperature having a specific gravity of approximately 1.30 to 1.32. The crystals may be inspected periodically and when they have attained the proper size the agitation and cooling are discontinued and the crystals

are allowed to settle to the bottom of the vessel. The mother liquor is then siphoned or drawn off and mixed with the mother liquor from the pans for further treatment. The crystals are then drained, washed, dried, screened and packaged for use. The entire harvest is marketable except for a small quantity which adheres to the cooled side wall of the upper portion of the vessel, these latter being dissolved and added to the Epsom solution for undergoing further treatment.

As to the proportions of seeding crystals to the quantity of solution introduced into the crystallizers, it is found satisfactory to transfer 800 to 1000 pounds of crystals into the tanks which are then charged with from 40 to 50 barrels of the above Epsom solution. The final yield varies from 2 to 3 tons of marketable needle crystals.

It has been found that the yield of needle crystals in accordance with the present invention is very high, the needles being substantially uniform in size and appearance. It is believed that this uniformity is accounted for by reason of the production of particularly desirable seeding crystals in the shallow pools, protected to secure a high degree of supersaturation. Uniformity is further enhanced by the contact of the relatively fine seeding crystals with the undersaturated solution in the crystallizers. It is believed that the solution melts or dissolves any irregular formations from the seeding crystals as the solution temperature drops to saturation so that during this short interim the seeding crystals become substantially uniform and ready for further growth. During crystal growth imperfections, such as nicks in the edges, tend to heal due to the fact that such points are active centers and growth will therefore proceed more rapidly at points of imperfection than at regular surfaces. Furthermore, during this period nuclei become acclimated to the solution and crystal growth begins immediately after saturation of the solution is reached.

It is necessary to avoid too great an undersaturation, either by reason of too low a concentration or too high a temperature in the solution mixed with the crystals as this might destroy a large number of seeding crystals and result in the precipitation of undesirable salt crystals from the solution. If the solution is more than 5°, for example, above saturation at the time of the introduction into the crystallizer, it is preferred to introduce a portion of the solution into the body of crystals in the crystallizer and permit the solution to reach the saturation point before the introduction of the remainder of the solution.

The process described herein is particularly beneficial in that it produces a large yield of uniform crystals in a minimum period of time and allows for adjustments for producing the desired size of crystals. Thus, the cooling and agitation may be stopped at any time and the crystals harvested. If the crystals have not made the necessary growth, additional solution may be added to the crystallizer to produce further growth. The complete time required for the cycle, that is, the production of the nuclei and the growth of the nuclei to the required size, varies from 12 to 24 hours, dependent on various conditions encountered.

Although the invention has been described with particular reference to the manufacture of Epsom salt, it may be utilized in the preparation of other materials which crystallize from solution, such, for example, as Glauber's salt, Rochelle salt, so-

dium carbonate, calcium chloride, and the like. The method disclosed herein for crystal formation is believed applicable to any crystallizing salt having a solubility which decreases appreciably with decreasing temperature.

Instead of using water for protecting the surface of the solution as supersaturation develops, it is contemplated to employ for the protective layer a dilute solution of the material undergoing treatment, of considerably lower concentration than the main body of solution and lower than that necessary to effect precipitation.

It will be understood that the above disclosure is for the purpose of enabling a clear understanding of the invention and that the invention is subject to variation within the scope of the appended claims.

I claim:

1. The method of producing magnesium sulfate crystals comprising forming seeding crystals by precipitation in a shallow pool, transferring the seeding crystals to a crystallizing chamber, mixing said crystals with a slightly undersaturated solution of magnesium sulfate in a relatively deep pool and cooling said solution during agitation to produce growth of said seeding crystals.

2. The method of producing magnesium sulfate needle crystals comprising forming seeding crystals by cooling a solution of the salt in a shallow pool to a point of supersaturation under relatively quiescent conditions, precipitating seeding crystals therefrom, mixing said seeding crystals with a fresh supply of magnesium sulfate solution which is undersaturated by reason of an elevated temperature, agitating said solution and seeding crystals in a deep pool as the solution temperature drops to the saturation point and continuing the agitation during growth of the seeding crystals.

3. The method of producing magnesium sulfate in crystal form comprising forming seeding crystals by precipitation in one vessel, separating the mother liquor from said crystals transferring the precipitated crystals to another vessel, introducing slightly undersaturated solution of magnesium sulfate into said other vessel and agitating the seeding crystals in said solution during crystal growth.

4. The method of producing magnesium sulfate crystals comprising maintaining magnesium sulfate solution in a shallow vessel under quiescent conditions to induce supersaturation of the solution, causing crystals to be precipitated from said supersaturated solution, commingling said crystals with a slightly undersaturated magnesium

sulfate solution in a relatively deep vessel and agitating said crystals and said solution together to cause portions of said crystals to be dissolved in said solution before said solution reaches saturation and to produce crystal growth at the expense of said solution thereafter.

5. The method of producing magnesium sulfate crystals comprising maintaining magnesium sulfate solution in a shallow vessel under quiescent conditions to induce supersaturation of the solution, causing crystals to be precipitated from said supersaturated solution, commingling said crystals with a slightly undersaturated magnesium sulfate solution in a relatively deep vessel and agitating said crystals and said solution together by means of an air flow introduced adjacent the bottom of said last named vessel during the period of undersaturation of said solution, as said crystals are partially dissolved in said solution, and after saturation is reached and said crystals grow at the expense of said solution.

6. The method of producing magnesium sulfate crystals comprising maintaining magnesium sulfate solution in a shallow vessel under quiescent conditions to induce supersaturation of the solution, causing crystals to be precipitated from said supersaturated solution, commingling said crystals with a slightly undersaturated magnesium sulfate solution in a relatively deep vessel and agitating said crystals and said solution together by means of an air flow and mechanical agitation adjacent the bottom of said last named vessel during crystal growth.

7. The method of producing crystals of precipitated salt comprising forming seed crystals by maintaining an undersaturated solution of a salt under quiescent conditions and without boiling in a shallow pool until supersaturation occurs and thereafter precipitating seed crystals from said solution, mixing said seed crystals with a fresh supply of a solution of said salt and agitating said mixture in a relatively deep pool during crystal growth.

8. The method of producing crystals of precipitated salt comprising forming seed crystals by maintaining an undersaturated solution of a salt under quiescent conditions and without boiling in a shallow pool until supersaturation occurs and thereafter precipitating seed crystals from said solution, mixing said seed crystals with a fresh supply of a solution of said salt and agitating said mixture in a relatively deep pool during crystal growth while artificially cooling said deep pool of solution and seed crystals.

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