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(54) A PROCESS FOR OBTAINING ANHYDROUS SODIUM SULPHATE FROM GLAUBER'S SALT

(71) We, ESCHER WYSS LIMITED, a Swiss Body Corporate of Hardstrasse 319, 8023 Zurich, Switzerland, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to a process for obtaining anhydrous sodium sulphate (Na_2SO_4) from Glauber's salt ($Na_2SO_4 \cdot 10\ H_2O$).

Sodium sulphate in its anhydrous form is produced as a chemical mass product and is used e.g. admixed with washing powder in the washing agent industry or as a chemical in the cellulose, dyestuffs and glass industries.

Glauber's salt is obtained in accordance with conventional industrial technology generally from its aqueous solution by cooling crystallization below 32°C.

A very large number of different techniques are used for converting Glauber's salt into the form of sodium sulphate free of water of crystallisation. Probably the commonest technique at the present time consists of melting and dissolving the Glauber's salt crystals above 32°C in their water of crystallisation or in sub-saturated solution, and then subjecting this solution to an evaporation crystallisation. The sodium sulphate crystals free of water of crystallisation which are obtained are then separated from the saturated solution, for example, in centrifuges, and dried in a drying apparatus. This process involves very considerable investment costs.

It is also known that anhydrous sodium sulphate can be produced from Glauber's salt by pure thermal treatment, for example, in directly fired long rotary tubular furnaces. In that case the melting and the dewatering are carried out in a single stage. But this procedure has the great disadvantage of resulting not only in a non-homogeneous kind of product but also in large lumps which have to be broken down subsequently, giving non-uniform particle forms.

It has been attempted to melt the Glauber's salt in its own water of crystallisation and then to spray this liquid into or on to a fluidised bed of dried sodium sulphate. Owing to various technical difficulties, such as blocking of nozzles etc., this method has not become widely adopted to a successful extent, although in principle granulation with a fairly uniform particle form is achieved. The considerable amount of dust produced is also a drawback.

A so-called "add-back" process has also been investigated for converting Glauber's salt to the anhydrous form of sodium sulphate. In that process, the already dried sodium sulphate is returned to a granulating apparatus preceding the actual drier, and in the said apparatus the water of crystallisation of the Glauber's salt produced is so combined by the returned dry sodium sulphate that flowable granulates of sodium sulphate are obtained. This is a practicable process, but it has the disadvantage that 7 to 12t of dried sodium sulphate per tonne of Glauber's salt have to be returned. In addition the end product obtained in this way exhibits a heterogeneous particle spectrum, and also contains a relatively large proportion of dust.

The aim of the present invention is to provide a process for the production of anhydrous sodium sulphate from Glauber's salt whereby a dust-free sodium sulphate with a homogeneous grain spectrum is obtained in an economical manner in only a single process stage.

Accordingly the present invention provides a process for obtaining anhydrous sodium sulphate from Glauber's salt which comprises mixing anhydrous sodium sulphate fines with unmelted Glauber's salt crystals without thereby causing release of the water of crystallisation, uniformly distributing the resulting mixture onto the surface of a bed of anhydrous sodium sulphate which is so turbulently fluidised by hot gas that the introduced material is immediately and intensively mixed with the material of the bed, whereby anhydrous sodium sulphate agglomerates are produced by dehydrating the Glauber's salt and withdrawing the anhydrous sodium sulphate agglomerates from the fluidised bed.

This technique is economical, involves only a single process stage, and yields a dust-free sodium sulphate product having a homogeneous grain spectrum and a very uniform grain form.

It will be noted that the Glauber's salt is not separately melted, but is mixed with anhydrous sodium sulphate fines, and the mixture is supplied to the fluidised bed in solid form. The bed must be given considerable turbulence so that the introduced mixture is at once mixed intensively with the sodium sulphate of the bed. The heating in the fluidised bed frees the water of crystallisation of the Glauber's salt, and the substance which has thus become liquid becomes attached to the anhydrous sodium sulphate agglomerates present in the bed, and forms thereon a further sodium sulphate layer, with simultaneous evaporation of the water of crystallisation. The anhydrous, usually spherical sodium sulphate grains produced with the dehydrating and drying issue as end product from the fluidised bed.

The grain range can be controlled by modifying the proportion of anhydrous sodium sulphate fines in the mixture which is to be introduced into the fluidised bed drier.

The invention will be described in detail and explained hereinafter with reference to the accompanying drawings, which shows an example of the installation for carrying out the process according to the present invention in diagrammatic manner.

The Glauber's salt crystals which are to be processed pass from a container 1 into a mixer 6, where a quantity of anhydrous sodium sulphate fines is added thereto. The mixture is applied by means of a feed device 2 to a bed 3 fluidised by means of hot gases, of anhydrous sodium sulphate agglomerates in a fluidised bed drier 4. The feed device 2 ensures uniform distribution of the applied mixture over the entire surface of the fluidised bed 3. The bed 3 is fluidised with great turbulence and a high degree of thorough mixing.

A suitably high standard of mixing in the bed 3 is obtained for example if for 700 to 1000kg of Glauber's salt and sodium sulphate mixture per hour and per square metre of incident surface at the fluidised bed drier 4, the bed 3 in the fluidised bed drier 4 is given a minimum height of 400 to 600mm. Bed heights up to 1000mm are considered practical. In order to permit use of fluidizing gas having a suitably high temperature, without any harmful effects on the product, so that the process can be carried out in an economical manner, the gas entry floor 10 is constructed in a manner known *per se* as an insulated floor with nozzles 11. In this way the surface temperature of the floor 10 is lower than the temperature of the fluidising gases in a gas distributing casing 9 of the fluidised bed drier 4. In this manner the product is prevented from burning on to the floor.

The anhydrous sodium sulphate grains obtained by the fluidisation in the bed 3 are guided as end product from the fluidised bed drier to a screening device 12 which contains a coarse sieve 16′ and a fine sieve 20′, where any fines and oversize particles are screened out of the end product which is removed via aperture 20.

With this installation it is proposed to add the Glauber's salt crystals to be processed the anhydrous sodium sulphate fines which are discharged from the fluidised bed 3 out of the fluidised bed drier 4 with the discharge air through a discharge air conduit 7. These fine particles are separated from the outgoing air in a separator 8 and, advantageously, before introduction through a conduit 5 into the mixer 6, they are cooled in a cooler 21 to a temperature below 32°C. This is very important because the admixing of hot sodium sulphate fines must not cause premature release, not even a partial one, of the water of crystallisation of the Glauber's salt already in the mixer 6; that must not take place until in the fluidised bed 3 in the drier 4.

With this installation it is also possible to add to the Glauber's salt crystals the sodium sulphate fines screened out of the end product, by means of fine sieve 20′. These fines fall out of the screening device 12 at an aperture 13, and are taken by means of a fan 14 and a conveying conduit 15 by way of the discharge air conduit 7 to the separator 8 etc.

This installation also allows oversize particles screened out of the end product in the screening device 12 by means of coarse sieve 16′ to be admixed with the Glauber's salt crystals. The oversize particles leave by way of an aperture 16 of the screening device 12, are comminuted to a desired size in a grinding device 17, and transported by means of a fan 18, a conveying conduit 19 and the discharge air conduit 7 to the separator 8 etc.

Finally, in the event of further requirements it is also possible to add a portion of the end product to the Glauber's salt crystals. However, in that case a portion of the end product must be comminuted and passed into the mixer 6 after cooling in the cooler 21.

The mean grain size of the end product obtained in the fluidised bed 3 can be controlled, as already mentioned, by modifying the proportion of anhydrous sodium sulphate fines in the mixture with Glauber's salt crystals. For example, in order to obtain an end product with an average granulation of d' = 0.3 to 0.8mm on the Rosin/Rammlen/Sperling (RRS) scale, 5 to 20 parts by weight of anhydrous sodium sulphate fines are added to 100 parts by weight of Glauber's salt depending on grain size.

WHAT WE CLAIM IS:

1. A process for obtaining anhydrous sodium sulphate from Glauber's salt which comprises mixing anhydrous sodium sulphate fines with unmelted Glauber's salt crystals without thereby causing release of the water of crystallisation, uniformly distributing the resulting mixture onto the surface of a bed of anhydrous sodium sulphate which is so turbulently fluidised by hot gas that the introduced material is immediately and intensively mixed with the material of the bed, whereby anhydrous sodium sulphate agglomerates are produced by dehydrating the Glauber's salt and withdrawing the anhydrous sodium sulphate agglomerates from the fluidised bed.

2. A process according to Claim 1, wherein anhydrous sodium sulphate fines discharged from the fluidised bed with the discharging crystals are mixed with the Glauber's salt crystals in the mixing step as at least part of the fines to be admixed.

3. A process according to Claim 1, which includes the steps of screening the product withdrawn from the fluidised bed to separate fine sodium sulphate particles and admixing the separated particles with the Glauber's salt crystals in the mixing step to separate undersize anhydrous sodium sulphate fines which are then used as at least a part of the fines to be mixed with the Glauber's salt crystals in the mixing step.

4. A process according to Claim 1, which includes the steps of screening the product withdrawn from the fluidised bed to separate oversize particles of sodium sulphate, comminuting the oversize particles, and admixing the comminuted particles with the Glauber's salt crystals in the mixing step as at least a part of the fines to be admixed.

5. A process according to any one of Claims 1 to 4, wherein the temperature of the anhydrous sodium sulphate fines which are added to the Glauber's salt crystals is below the melting point of the Glauber's salt, i.e. below 32°C.

6. A process according to any one of Claims 1 to 5, wherein the mixture of Glauber's salt and sodium sulphate is introduced into the fluidised bed in a quantity of 700 to 1000 kg/h per m^2 of incident flow bed surface area.

7. A process according to any one of Claims 1 to 6, wherein the fluidised bed is kept to a height of 400 to 1000mm.

8. A process according to any one of Claims 1 to 7, wherein the mixture contains 100 parts by weight of Glauber's salt and 5 to 20 parts by weight of anhydrous sodium sulphate fines, and the product withdrawn from the fluidised bed has an average granulation of d' = 0.3 to 0.8mm on the Rosin/Rammlen/Sperling (RRS) scale.

9. A process according to any one of Claims 1 to 8, wherein the hot gases are supplied to the fluidised bed through nozzles in a bed floor which is insulated from an underlying gas distribution chamber.

10. A process for obtaining anhydrous sodium sulphate (Na_2SO_4) from Glauber's salt ($Na_2So_4 . 10 H_2O$) substantially according to claim 1 substantially as herein described.

11. Anhydrous sodium sulphate when obtained by the method according to any one of the preceding claims.

12. An apparatus when used for the manufacture of anhydrous sodium sulphate substantially as herein described with reference to and as shown in the accompanying drawing.

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1562824 COMPLETE SPECIFICATION

1 SHEET *This drawing is a reproduction of the Original on a reduced scale*