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G. A. KRAUSE
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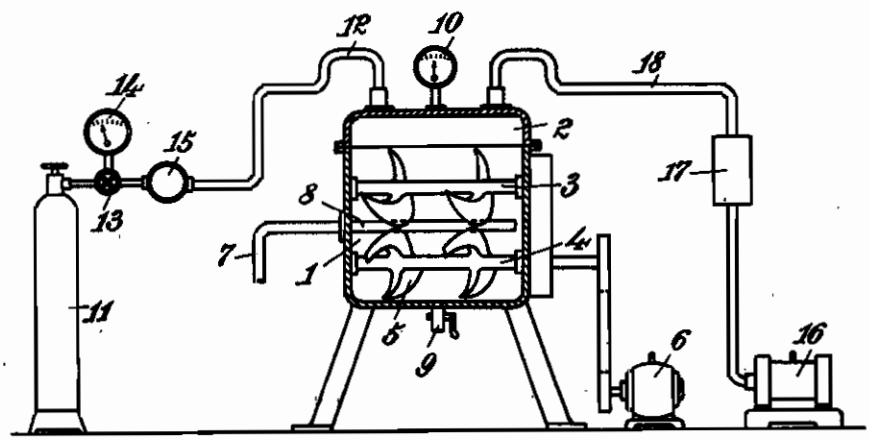

Inventor:
Geo. A. Krause
By
Beam, Brooks, Buckley & Beam
ATTORNEYS

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PROCESS OF RENDERING CASEIN SOLUBLE

Georg A. Krause, Munich 2 No, Germany; vested in the Alien Property Custodian

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The present invention refers to a process of rendering casein soluble by means of gaseous ammonia.

This method of making casein soluble implies a number of advantages, which to a part are due to the fact that the casein is only to a low degree charged with chemical substances. Whereas, for instance, in the manufacture of caseinate of sodium about 2 to 4 g. of sodium-hydroxide are required per 100 g. of dry casein, and even greater quantities in the case of hydrolysable sodium salts (carbonate, bicarbonate), about 0.6 g. of NH_3 are sufficient for the same quantity of casein, in order to prepare ammonia casein. NH_3 and the compounds resulting therefrom, respectively, are, moreover, not unrelated to casein, as are, on the contrary, the sodium compounds. When rendering the casein soluble by means of gaseous agents, it is furthermore unnecessary, to increase the quantity of liquids, which thereafter must again be expelled from the treated material, as would, contrary thereto, be necessary, if sodium salts are used for this treatment.

In spite of these preferences, the hitherto known processes of rendering casein soluble by means of gaseous ammonia, in which processes only dry casein was, moreover, used as initial material, failed due to the extremely long period of treatment, which was detrimental to the material treated, and also due to the difficulty of expelling the excess of gas retained by the casein of adsorption, in particular, however, due to the unequalities in gas absorption. Experiments of charging alcohol or ether with casein, and of subsequently treating the suspension or paste with gaseous agents, did not result in any improvement of the process, but made it more expensive due to higher costs of investment and manufacture. The technical difficulties implied in practising the process, were considered to be so enormous that since the time of the first proposals no serious effort or success has, hitherto, become known in this field in spite of repeated suggestions in literature.

The subject matter of the present invention is a process which offers a possibility of eliminating the difficulties hitherto experienced, when rendering casein soluble by means of gaseous ammonia. Tests furnished proof of the fact that the former unfavorable results are due to particular qualities adapted by the casein in the course of its treatment, because casein, if it is brought into contact with gaseous NH_3, after a longer or shorter period corresponding to the humidity of the casein forms on its surface a viscous layer of jelly, representing a saturated or super-saturated solution of ammonia in casein. This layer of jelly represents a stop preventing the direct absorption of the gaseous ammonia by the non-dissolved casein placed underneath it. Therefore this casein can only be supplied with dissolved ammonia from the layer of jelly enveloping it.

The diffusion of ammonia from the viscous jelly into the layer not yet influenced will, however, take place very slowly and in a perfectly uncontrolled manner. Supersaturated and saturated layers will adjoin others which have been left untreated, and the resulting finished material will consequently not be homogeneous.

It was considered almost impossible to expel the excess in NH_3 from the viscous jelly with ordinary means.

Under the present invention such phenomena are prevented by saturating under pressure the humid casein with the acting gas. In this manner the layer of material to be treated, which may have any desired height, appropriate to the individual requirements of the case concerned, will be permeated by the gas in a perfectly uniform manner, so as to be saturated with said gas within a comparatively short time, and in such a way that it is possible to increase and control the velocity of reaction in due correspondence to the pressure employed.

The saturation under pressure of the casein can be carried through in different ways. First of all it is possible to cause the gas, when in its compressed condition, to bring its influence to bear on the material to be treated, for instance in such a way that liquefied ammonia displays its influence by detention within a pressure-proof vessel. It is, however, also possible to proceed in such a way that the gas superimposed upon the material to be treated is exposed to pressure by means of mechanical influences, for instance in such a way that it is compressed by a piston, or that the gas, when exposed to normal pressure, is incorporated with the casein under pressure by means of a kneading or beating operation, or the like.

An embodiment of the invention, which is appropriate to the purpose in question, would also be represented by a combination of the aforedescribed methods, i. e. by mechanically working the gas under pre-tension into the material to be treated, e. g. by equipping a pressure-proof vessel with a kneading device or an agitator, thus promoting the gas pressure by the mechanical process. In this manner it will be possible to par-

ticularly restrict the dimensions of the equipment necessary for treatment.

The solution of ammonia in casein will most probably commence to take place by way of the water contained in the material to be treated, even in case of these water contents being very small; as, on the other hand, the solubility of ammonia in water is a function of both pressure and temperature, different ways will be offered of controlling the process under the invention so, as to regulate it. It is, in particular, possible to control the concentration of the gas in the treated material by an alteration of the pressure, which latter, on its part, can be controlled by a change in temperature. On the other hand it would, however, also be possible, for instance to cool down the treated material also during the process of saturation; as otherwise the treated material would adopt a higher temperature during the course of the reaction process, it is thus possible to increase the concentration of the ammonia during the process of solution by cooling it, and by maintaining the pressure at the same height.

Finally it would also be possible to control the concentration of the gas by altering the water contents of the material to be treated, it also being possible, in doing so to combine the various aforementioned measures in any desired manner, if necessary.

In all of the embodiments it must be kept in mind that the process of rendering the casein soluble will take place in a solution of agents used for treatment, which is saturated in due correspondence to the pressure of the agent used for treatment and to the temperature existing, at the time, because the pressure above a liquid is always equal to the steam pressure, and to the aggregate amount of the partial pressures, respectively.

When availing oneself of the process under the present invention for practical operation, it is either possible to successively add the gas in stages up to the moment when solubility of the casein is attained, or contrary thereto at first to use an excess in gas and to again compensate such excess later on by an addition of untreated casein. It is preferable for this purpose to provide the equipment for treatment with a device making it possible to draw test quantities, in order to be able at any time to ascertain the condition of the treated material, and the completion of the treatment, respectively, it, however, being necessary to saturate the casein with the agent used for treatment in strict correspondence to the water contents, and in such a way that the pH figure can be controlled.

In case of an excess quantity of ammonia being present in the treated material after saturation, such excess can be expelled in an appropriate manner, and under certain circumstances can again be used for circulation. It is preferable for this purpose to heat the casein, e. g. in vacuo, and at the same time, or subsequent thereto, to suck off such gas as has been retained by adsorption. The ammonia excess can be reclaimed by having it adsorbed by cold water, from which it is then expelled and again re-introduced into the process under the present invention e. g. after its previous liquefaction.

A particular advantage will result in the treatment under the present invention of such casein as will be so treated after precipitation, washing, and after having been partly freed of water by mechanical means.

When proceeding in this manner, the equipment for drying the casein can be dispensed with, and the heat used for expelling the gas will expedite the drying process.

Though this is not absolutely necessary, the drying of the finished material, which has been treated, is preferably carried out in such a way that the casein is already dried in the course of expulsion of the superfluous treatment agent, this drying process in a preferable embodiment of the present invention taking place in such a way that saturation, removal of gas, and drying, are continuously taking place in one common vessel. For this purpose a vacuum-boiler can, for instance, be used, which is also resistant to pressure, and equipped with an agitator or a kneading device, and with an appropriate heating apparatus. Particular care must, however, be taken to carry out the expulsion of gas and the drying action in such a way that a large surface is exposed to the treatment, in particular, where casein with lower water contents than 70% is concerned, because the viscous jelly offers a high resistance both to the passage of heat and to the vacuo. If a great-surface thin-layer process on vacuum rollers or sheets in a vacuum drying cabinet, which could also be used for treatment, is not selected, and if it is intended to render the installation particularly small and efficient, it is preferable to use the above mentioned vacuum kneading device, which can be heated and is able to resist pressure, and by which always other surface of the treated material are feed to the heating surface, and again conveyed away, wherefore it is possible to expel and suck off the gas used for treatment, or the water.

The present invention finally also covers devices for practising the new process. As already explained, a treatment vessel which can be sealed so as to be gas-tight, possessing conduits for gas supply and evacuation, and provided with a heating and cooling device, is particularly suitable for the inventive purpose. Such installations can also be equipped with controlling devices, e. g. pressure gauges, thermometers, hygrometers, or the like, by which the supply of gas, the adjustment of the operation-temperature, the water contents etc., are, corresponding to the condition of operation prevailing at the time, automatically controlled.

The drawing diagrammatically illustrates a device for carrying into practice the process under the present invention.

Kneaders **5** on two shafts **3** and **4**, which are coupled in an appropriate manner, rotate within the treatment vessel **1** which by means of a lid **2** can be closed so as to be airtight, and are driven by a motor **6**. **7** designates the conduit provided for a heating and cooling device **6**, whereas **9** is a device for drawing test quantities, **10** being a pressure gauge.

Container **1** can be supplied with liquefied ammonia from bottle **11** by means of a moveable conduit **12** and a pressure reduction vaive **13** with pressure gauge **14**, and through a gas meter **15**.

On the other hand a vacuum pump **18** is, through a cooling pocket **17**, which is intended to store the condensed product, connected with the treatment vessel **1** by a conduit **16**.

The manner in which the described installation is used, can be seen from the following description of operation, given by way of example:

50 kg. of casein, which has been precipitated with muriatic acid and repeatedly washed, its

water contents being kept at 50% by mechanical means (pressing or centrifugation), are placed into the kneader 1. After lid 2 has been closed so as to be gas-tight, the kneading device is started by means of motor 6, whereupon, controlled by the measuring device 14, 15 10 about 150 g. of ammonia under a pressure of 6 atmosphere, are feed to vessel 1. At the same time by means of device 8, the material to be treated, which has increased its temperature in consequence of the reaction process taking place, is cooled down to about 15° Celsius. After a kneading action of about 30 minutes, during which the pressure will become lower in the course of progress of the reaction, several grams of the treated material are taken out from the vessel by means of device 9. By measuring the pH figure and, if necessary, by subjecting the drawn material to a solution test, it is ascertained whether the treatment is completed. The pH figure will naturally be dependent upon the desired qualities of the finished material, and in the present example of operation will probably be about 6.0 to 6.3

During the following stage, in which the gas is expelled and drying takes place, the material to be treated is heated up to 58° Celsius and kept at this temperature by means of device 8, which, for instance, can be provided with a hot-water circulation. By starting vacuum pump 16, a vacuum of about 100 mm. of mercury column is brought about in the interior of vessel 1. The quantity of gas, which has been retained by adsorption, if any, is sucked off and the treated material then dried.

The casein which now still contains about 5 to 6% of water, is then withdrawn from the installation, and can be subjected in the customary manner to a further process of disintegration.

The material prepared under this process is a semi-translucent, hard and splintery substance which in the course of the drying operation will spontaneously detach from the drying surface, which, further on, can be easily ground, and is without any residues readily soluble in cold and hot water. In accordance with its previous treatment and the quality of the primary material it will contain about 85–90% of high-quality protein which is highly digestible, i. e. an article which can be used both in the food industry, and for technical purposes.

GEORG A. KRAUSE.