March 29, 1949. O. A. BATTISTA 2,465,343

PRODUCTION OF GLOBULAR REGENERATED CELLULOSE PARTICLES

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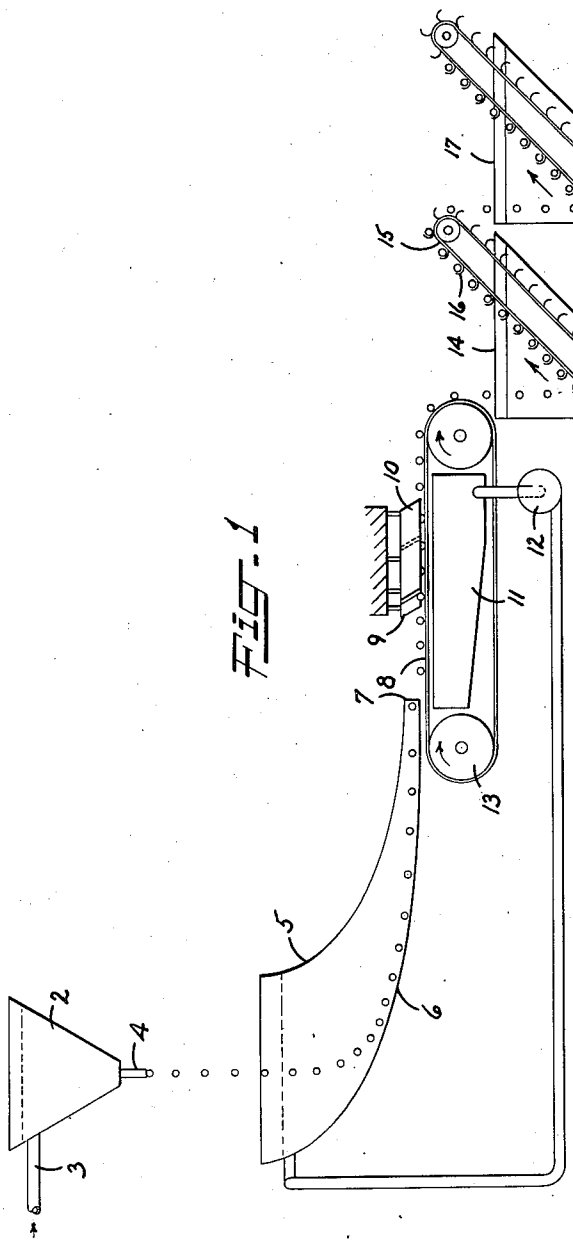

FIG. 1

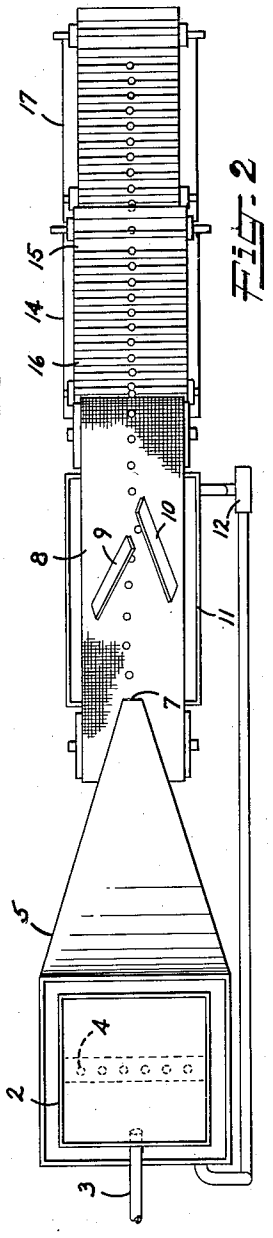

FIG. 2

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PRODUCTION OF GLOBULAR REGENERATED CELLULOSE PARTICLES

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Application February 15, 1947, Serial No. 728,902

10 Claims. (Cl. 18—48)

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This invention relates to the production of globular particles of regenerated cellulose, particularly from viscose.

Methods that have heretofore been employed for making globular particles from viscose resulted generally in products which not only were far from sphericity but were marred by numerous checks and fissures resulting from the inability to prevent the development of gases within the globules during regeneration and the inability to prevent irregular shrinkage during regeneration, subsequent wet process, and drying of the globules.

In accordance with the present invention these difficulties are substantially completely avoided. This is accomplished by limiting the contact of the freshly formed globules with the acid regenerating bath (into which they fall in droplets) to that which merely forms a thin skin upon the outer surface of the globule and which is inadequate to develop any gas bubbles within the globules. After the limited contact in the acid bath, the globules having the thin skin which maintains the spherical structure are introduced into a heated liquid of non-solvent non-precipitating character in which coagulation and regeneration of the globules are completed. Suitable liquids for this purpose which are incapable of dissolving the granules are hot water or hot mineral oil at a temperature below the boiling point of water.

After the globules are taken from the heated liquid, the by-products of regeneration, such as alkaline and sulfur compounds, are in a water-soluble state and are merely leached out by introducing the globules into water for the desired period of time. Thereafter, the globules may be dried or, before drying, they may be soaked in a plasticizer, such as glycerine, when a softened product is desired. Shrinkage of any substantial extent can only occur during the drying step of this procedure since the contact of the globules with acid is restricted to a minimum and the globules are maintained in water-swelled condition thereafter until the drying stage. It has been found that the shrinkage that occurs during the drying of the globules formed in accordance with the procedure of this invention is remarkably uniform in character so that they undergo little change in their original shape other than a diminution in size during the drying.

The original shape of the globules formed depends upon the height from which the viscose droplets are allowed to fall into the acid bath. If a substantially spherical shape of definite diameter is desired, the viscose should be dropped from a height between four and six feet above the level of the acid bath, the less viscous the viscose, the lower the height that should be used.

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If greater dropping heights are used, the size of the droplet falling from the dropping orifice or jet does not determine the ultimate size of the product as, in such case, they tend to disrupt into numerous smaller particles which may have various sizes and, in the extreme of conditions, a powdered product results.

The drawing illustrates one arrangement for executing the invention, in which

Figure 1 is an elevation of the arrangement,

Figure 2 is a plan view thereof.

As shown in the drawing, the viscose solution is fed into a hopper 2 by a suitable feed pipe 3. The hopper is provided with dropping tubes 4. These tubes have orifices of any conventional character adapted to the formation of droplets under the hydrostatic head within the hopper, or under the application of any other hydraulic force. A receptacle 5 for the acid bath is arranged beneath the hopper to receive the droplets and has a sloped bottom 6 terminating in an opening 7 of small diameter to allow passage of the "skinned" globules onto the top surface of a foraminous belt 8, such as of wire screen. A pair of inclined deflectors 9 and 10 may be arranged in advance of the opening 7 to disrupt any liquid stream flowing therefrom before it goes beyond the belt. A drainbox 11 is arranged beneath the upper course of the belt to catch the acid passing through the belt and this acid may be recirculated to the receptacle 5 by means of pump 12. Rolls 13 drive the belt 8 which dumps the globules thereon into a receptacle 14 containing heated liquid such as mineral oil or water below 99° C. A chain conveyor 15 having projecting transverse slots 16 is arranged within the receptacle at an inclination so that it serves to carry the globules therefrom to the next receptacle 17 containing water in which the globules are leached. A similar inclined conveyor may be arranged in this receptacle for carrying the globules therefrom to the next stage of treatment which may be that of drying or that of soaking in a plasticizer, such as glycerine, after which drying may be effected.

The depth of the acid bath in the receptacle 5 may vary from 3 to 12 inches and the slope of the bottom 6 is so selected with relation to the liquid depth that the time required for the globules to pass through the acid bath is only that which forms a skin without forming gas bubbles within the globules. The maximum permissible time of this passage depends mainly on the concentration of the acid. Where a bath having a sulfuric acid concentration of 10% is used, a passage of one minute is satisfactory, while a longer time of passage is permissible with a weaker acid bath, and vice versa. Sulfuric acid baths of 7 to 12% concentration may be used.

The final products which may be of any size up to the order of ⅓ inch in diameter approach true sphericity and are substantially free of checks, fissures, bubbles, creases and wrinkles. The unplasticized products are hard and have a high dielectric constant. Because of this characteristic and the fact that the products are highly resistant to hydrocarbon oils they may be used in conjunction with such oils for cooling electrical equipment, such as transformers. They may also be used in lightning arresters and as line protective means, for example, fuse elements, particularly in circuit breakers. The unplasticized products may be coated with moisture-proofing and chemically resistant compositions where their use would involve prolonged contact with moisture, acids, alkalies, or other corrosive fluids. The plasticized products are of use where flexibility is desirable.

It is to be understood that changes and variations may be made without departing from the spirit and scope of the invention as defined by the appended claims.

I claim:

1. The method of making granular particles comprising regenerated cellulose comprising the steps of allowing droplets of viscose to fall into an acid bath, removing the formed granules of viscose from the bath after a thin skin has been formed thereon as a result of the precipitating action of the acid bath but before gas bubbles are formed within the granules, and regenerating the superficially precipitated granules in a hot substantially non-acid bath of a liquid incapable of dissolving the granules.

2. The method of making granular particles comprising regenerated cellulose comprising the steps of allowing droplets of viscose to fall into an acid bath, removing the formed granules of viscose from the bath after a thin skin has been formed thereon as a result of the precipitating action of the acid bath but before gas bubbles are formed within the granules, regenerating the superficially precipitated granules in a hot substantially non-acid bath of a liquid incapable of dissolving the granules and then leaching the granules.

3. The method of making granular particles comprising regenerated cellulose comprising the steps of allowing droplets of viscose to fall from a height of about 4 to 6 feet into a sulfuric acid regenerating bath, removing the formed granules of viscose from the bath after a thin skin has been formed thereon as a result of the precipitating action of the acid bath but before gas bubbles are formed with the granules, regenerating the superficially precipitated granules in a hot substantially non-acid bath of a liquid incapable of dissolving the granules.

4. The method of making granular particles comprising regenerated cellulose comprising the steps of allowing droplets of viscose from a height of about 4 to 6 feet into a sulfuric acid regenerating bath, removing the formed granules of viscose from the bath after a thin skin has been formed thereon as a result of the precipitating action of the acid bath but before gas bubbles are formed with the granules, and regenerating the superficially precipitated granules in a hot bath of mineral oil.

5. The method of making regenerated cellulose globules approaching sphericity in shape comprising the steps of allowing viscose to fall in

the form of individual drops from a height of about 4 to 6 feet into a sulfuric acid regenerating bath of about 7 to 12% concentration, removing the formed globules from the bath after a thin skin has been formed thereon but before gas bubbles are developed within the globules, and regenerating the superficially precipitated globules in a hot substantially non-acid bath of a liquid incapable of dissolving the globules.

6. The method of making regenerated cellulose globules approaching sphericity in shape comprising the steps of allowing viscose to fall in the form of individual drops from a height of about 4 to 6 feet into a sulfuric acid regenerating bath of about 7 to 12% concentration, removing the formed globules from the bath after a thin skin has been formed thereon but before gas bubbles are developed with the globules, and regenerating the superficially precipitated globules in a hot mineral oil bath.

7. The method of making regenerated cellulose globules approaching sphericity in shape comprising the steps of allowing viscose to fall in the form of individual drops from a height of about 4 to 6 feet into a sulfuric acid regenerating bath of about 10% concentration, removing the formed globules from the bath after about one minute, regenerating the superficially precipitated globules in a hot substantially non-acid bath of a liquid incapable of dissolving the globules.

8. The method of making regenerated cellulose globules approaching sphericity in shape comprising the steps of allowing viscose to fall in the form of individual drops from a height of about 4 to 6 feet into a sulfuric acid regenerating bath of about 10% concentration, removing the formed globules from the bath after about one minute, and regenerating the superficially precipitated globules in a hot mineral oil bath.

9. The method of making regenerated cellulose globules approaching sphericity in shape comprising the steps of allowing viscose to fall in the form of individual drops from a height of about 4 to 6 feet into a sulfuric acid regenerating bath of about 10% concentration, removing the formed globules from the bath after about one minute, regenerating the superficially precipitated globules in a hot mineral oil bath, leaching the globules in water, and subsequentially drying them.

10. The method of making regenerated cellulose globules approaching sphericity in shape comprising the steps of allowing viscose to fall in the form of individual drops from a height of about 4 to 6 feet into a sulfuric acid regenerating bath of about 10% concentration, removing the formed globules from the bath after about one minute, regenerating the superficially precipitated globules in a hot mineral oil bath, leaching the globules in water, introducing the globules while wet into a bath of plasticizer, and drying them.

ORLANDO A. BATTISTA.

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The following references are of record in the file of this patent:

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Certificate of Correction

Patent No. 2,465,343. March 29, 1949.

ORLANDO A. BATTISTA

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows:

Column 3, line 62, after the word "viscose" insert *to fall*;

and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 11th day of October, A. D. 1949.

[SEAL]

THOMAS F. MURPHY,
Assistant Commissioner of Patents.