April 14, 1970

G. JACQUELIN

3,506,536

METHOD OF TREATING SUSPENSIONS OF FIBRES TO FORM AGGREGATES

Filed June 13, 1966

2 Sheets-Sheet 1



FIG. 1

FIG. 2





April 14, 1970

G. JACQUELIN

3,506,536

METHOD OF TREATING SUSPENSIONS OF FIBRES TO FORM AGGREGATES Filed June 13, 1966 2 Sheets-Sheet 2



United States Patent Office

1

3,506,536 METHOD OF TREATING SUSPENSIONS OF FIBRES TO FORM AGGREGATES Guy Jacquelin, Grenoble, Isere, France, assignor, by mesne

- assignments, to Societe d'Exploitation des Brevets 5 Granofibre, Paris, France
 - Continuation-in-part of application Ser. No. 427,040, Jan. 21, 1965. This application June 13, 1966, Ser.

No. 557,036 Claims priority, application France, Jan. 21, 1964,

960,962; June 14, 1965, 20,695 Int. Cl. D21b; D01c; D01d 3/00 U.S. Cl. 162-1

13 Claims

ABSTRACT OF THE DISCLOSURE

A method of treating a suspension of fibres to form aggregates which comprises subjecting a suspension of the fibres in a liquid to rotational agitation to establish a speed gradient between points in the suspension caus-20 ing the formation of eddys, which result in the fibres moving together into groups. The grouped fibres cling to one another to form aggregates, the concentration by weight of the fibres in the suspension at the beginning of the treatment being relatively low. 25

- This application is a continuation-in-part of my earlier application Ser. No. 427,040 filed Jan. 21, 1965 and now abandoned.
- 30The present invention relates to a method of treating suspensions of natural, regenerated or synthetic fibres in a liquid medium.

The purpose of the method of the invention is to enable the formation within a suspension of aggregates of $_{35}$ regular fibres which are individualized and relatively consistent, said aggregates generally being of regular shape such as, for example, spheroidal, and of a size which may be of the order of the length of the fibres in suspension (0.2 to 15 mm.). 40

It is an object of the invention to provide a method of treating suspensions of fibres comprising causing a general agitation in a liquid bath containing fibres in suspension, said agitation being carried out under conditions which are produced by the rotating and circu- 45 lating movements of the said fibres in a weak shearing or cutting field so that said fibres cling to one another and form aggregates, which are roughly grouped or sized and of regular shape.

According to a feature of the invention, the agitation 50 of the bath is followed by filtration enabling the aggregates of the liquid medium to be separated from the nonfibrous elements and if desired from the non-aggregated fibres

In this way, the fibres of the other solid components 55 of a bath may be separated, this being particularly advantageous and especially applicable to the shearing of a paper pulp. At least two categories of fibres may be separated, thus providing for manufacturing from the same suspension at least two paper pulps having different 60 properties. To this end, the aggregates separated by filtration are disintegrated and returned to homogeneous suspension by agitation in a liquid medium while the other components are removed with a filter and subjected to further treatment as desired.

The fibrous aggregates separated by filtration can also be drained and then dried in a current of hot air, which technique has the advantage of being more economical than the technique, which is currently used, of draining and drying the paper pulp in sheets. Moreover, by this 70 technique, independent aggregates are obtained, which are roughly grouped or sized, light, easy to stock, and

2

readily manipulated and treated, and which may be used for many purposes.

According to another feature of the invention, the fibrous aggregates can be agglomerated either by using the other components of the bath as bonds, or after impregnation with a deposited bond, said operation being followed by a drying of the porous agglomerate thus obtained, and if desired also by a thermal treatment. Such agglomerates thus produced can be used, depending upon the nature and amount of the bonds, as linings for walls,

10 floors or other partitions, or for thermal or sound insulation.

According to another feature of the invention, the fibrous aggregates can be impregnated with a resin or with a prepolymer in order to constitute fibrous granules 15 for compression by heat in order to form molded or laminated parts. In this way, a fibrous structure forming a reinforcement can be given to molded or laminated parts by heat and under pressure.

- The invention also has as an object the provision of apparatus for carrying out the aforesaid method, such apparatus comprising a tank which is to be partially filled from the bath to be treated, and means for gently agitating said bath.
- The agitation means may comprise means for rotating the tank which may be cylindrical and mounted so as to rotate about an axis, or by a rotatable member arranged in a fixed tank, or by at least one gas injecting member.
- The apparatus may be cyclically or continuously operated. In the latter case, the apparatus may comprise a tank mounted on a horizontal axis and in which an Archimedean screw is arranged, the latter constituting the agitation means, if desired.
- The invention also relates to the new industrial products obtained by carrying out the method and which may be constituted by:
- fibrous spheroids, the diameter of which may vary from 0.2 to 15 mm. and which may have a low apparent density of between 0.05 and 0.70, after draining and drying.
- the sheared paper pulp,
- a porous conglomerate having an apparent density between 0.02 and 1 according to the field of use,
- molded or laminated parts having a fibrous structure made by compression and thermal treatment of granules formed from fibrous spheroids impregnated with a resin or other products.

Various other features of the invention will become apparent from the detailed description which is to follow. taken in conjunction with the accompanying drawings which show various embodiments of apparatus according to the invention, by way of example, and in which:

FIGURE 1 diagrammatically illustrates cyclically-operated apparatus employed in accordance with a first embodiment of the invention;

FIGURE 2 diagrammatically illustrates cyclically-operated apparatus employed in accordance with a second embodiment of the invention;

FIGURE 3 diagrammatically illustrates cyclically-operated apparatus employed in accordance with a third embodiment of the invention;

FIGURE 4 is a diagrammatic view illustrating a con-65 tinuously operated apparatus;

FIGURE 5 shows the paths and speed distributions of the fibres in the suspension produced in the above apparatus: and

FIGURE 6 shows the variations in production and yield of the particles as a function of the concentration of the fibres in the suspension.

According to the invention, a fibrous suspension to be

treated is subjected to agitation produced under conditions which cause:

(1) Rotating movements of the fibres enabling them to be hooked when they come into contact, either with each other, or with aggregates in the course of formation, independent fibrous assemblies, relatively consistent, being developed by the addition of fibres to one another.

(2) A circulation in the mass of the suspension, so that a large number of fibres and aggregates in the course of formation passes into adjacent areas having solid surfaces, 10 where the shearing or cutting power, depending on the speed gradients of the fluid, causes the rotation of the fibrous elements and the formation of eddies in the fluid propagating the above mentioned phenomenon. Moreover, the agitation of the suspension must be less turbulent for 15 the sole reason that the shearing or cutting power caused by the relative movements of the suspension and of the solid surfaces is sufficiently low so as not to destroy the fibrous aggregates formed in the suspension. Moreover, the surface of the solids in contact with the suspension is 20 preferably smooth and less adherent to the fibres so that they do not cling to them nor destroy the aggregates in the course of their movement. When these conditions are combined, the changes in the structure of the suspensions also depend on the morphological and physiochemical 25properties of the fibres. Thus, certain fibres refuse to cling together and others, on the contrary, rapidly form regular aggregates. This aptitude of the fibres may moreover be modified by physical or chemical treatments and by the action upon them by the properties of the suspension 30 medium.

In the case of paper fibres of substantially similar length, if the axis of the rotation torques applied to the fibres by reason of the agitation of the suspension does not have a mandatory direction, the aggregates appear in 35 the form of separate regular pellets which are of relatively uniform dimensions which may be of the order of the mean length of the fibres. They are easy to separate by means of a rough filtration and may represent more than 80% of the total quantity of fibres. The dimensions 40 of said spherical pellets depend not only on the sizes of the fibres, but also on their concentration and the characteristics of the agitation (form and strength).

Thus, the invention enables a new industrial product to be manufactured which is constituted by fibrous spheroids, $_{45}$ the diameter of which may vary from 0.2 mm. to 15 mm. After draining and drying, they have a low apparent density of between 0.05 and 0.70.

The drainability of the suspension of fibrous aggregates (after treatment) is clearly improved compared with that 50of the initial homogenous suspension. Moreover, according to an important characteristic phase of the method, the drying of the drained fibrous pulp (in the form of independent aggregates) may be effected in a current of hot gas according to well known methods which could not be 55applied, up to the present, directly to paper pulp, which, in fact, had to be drained in sheets (or layers) and dried in this state or even broken into pieces before drying. It will be apparent that the drying of the pulp directly obtained in the form of small and very permeable aggre-60 gates, in a current of hot gas, is much more economical and advantageous.

The method of the invention also enables the fibres of the other elements in suspension (fillers, fine elements, vessels and accessory elements of annual plants in particular) to be separated. These non-fibrous elements are incorporated very slightly with the aggregates and consequently the majority remains in suspension. They may thus be easily separated by a rough filtration retaining the aggregates. Moreover, certain fibrous elements, the properties (strength, surface condition, length) of which do not allow their linking to other fibres, are also separable from the rest of the other fibres, by means of this method. This is, for example, of use for modifying the properties of a paper pulp. In order to shear a paper pulp, according to a particularly advantageous way of carrying out the method, the aggregates or fibrous spheroids are separated by filtration, then they are disintegrated and replaced in homogeneous suspension by agitation in a liquid medium of suitable nature and strength, the invention extending to the new industrial product constituted by the paper pulp thus sheared.

According to another way of carrying out the method of the invention, the fibrous spheroids formed in the bath may be agglomerated together either in their humid state, the non-aggregated elements of the suspension, if desired, serving as a bond or a bonding agent, or after impregnation by a bond. Moreover, at the beginning of the treatment, the bath may contain deposited matter, serving particularly as a bond such as, for example resins, gums, polymerizable or cross-linkable products . . . this matter being adapted for being put into solution or emulsion in order to be integrated in the fibrous aggregates during their formation and/or to be connected to the aggregates formed. A new industrial product is thus obtained, after drving and, if desired, thermal treatment, said product being constituted by a conglomerate, which is more or less porous, the apparent density of which may be still lower than that of the spheroids. It varies for example between 0.02 and 1. These spherules, associated or not with other matter, polymeric bonds for example, enable panels and other products to be produced, the properties of which may be varied to a large extent from the point of view of density, strength, porosity, and thermal or sound insulation.

In another embodiment of apparatus for carrying out the method of the invention, the fibrous spheroids, formed and dried, may be impregnated with a resin or with a prepolymer and may thus constitute fibrous granules. By compression of said latter and thermal treatment if desired or found necessary, molded or laminated parts may be produced, the fibres of which serve as a reinforcement. The invernion thus also povides new industrial semi-finished products constituted by the granules and the new industrial finished product formed by parts having a fibrous structure.

Apparatus for carrying out the method can be cyclically (FIGURES 1 to 3) or continuously (FIGURE 4) operated.

In the case of the apparatus of FIGURES 1 to 3, the equipment comprises a tank 1 containing the bath, i.e. the liquid, to be treated, which preferably only partially fills the said tank. This bath has in these circumstances a free surface 2 and is in contact with solid surfaces constituted by the internal wall of the tank and possibly by internal rigid elements. These solid surfaces may advantageously be smooth, i.e. without roughness, and their structure must be evolutive, i.e. without any abrupt variation of curvature, in order to prevent the fibres linking or hooking together and violent shocks on the aggregates in suspension.

The relative movemet of the suspension and of the solid surfaces causing the agitation necessary for carrying out the method is produced either by the movement of the tank itself (FIGURES 1 and 2) or by the movement of a member 3 internally located (FIGURE 3) or by the application of other forces (for example, by injecting gas) or by various combiniations of these means.

In a particularly advantageous embodiment, the tank 1 is cylindrical and the agitation is produced by rotating the member provided for agitating the bath preferably about the axis of the member, namely either the tank 1 itself (FIGURES 1 and 2) or the rotatable member 3 (FIGURE 3). The axis of the tank may be horizontal (FIGURE 1), inclined (FIGURE 2) or vertical (FIGURE 3).

This arrangement enables a suitable circulation of the fibres to be obtained and a sufficient speed gradient on 75 the walls to be maintained in order to produce the rota-

5

tion of said fibres in different planes by compounding the action of gravity and the action of rotation.

In the embodiment illustrated in FIGURE 3, the rotatable member 3 has a highly developed movable surface which is, for example, a simple spiral. It could also have other shapes.

Whatever the embodiment envisaged (FIGURES 1, 2 and 3) may be, the cyclically operated apparatus is associated with means for filling and emptying the tank 1, put into operation at the beginning and end of the treat-10 ment.

The continuously operated apparatus comprises a tank 1 and agitation means set up and adapted to operate in such a way that the circulation of the suspension is orientated to effect a continuous progression of the sus-15 pension from the inlet to the outlet of the tank where permanent filling and emptying means are put into operation.

In the embodiment shown in FIGURE 4, the tank 1 is cylindrical and an Archimedean screw 4 is arranged $_{20}$ therewithin along its substantially horizontal axis. This screw can moreover constitute the agitation means.

The tank 1 may be rotatable and the screw 4 fixed, or the tank may be fixed and the screw rotatable, or the screw and the tank can even be secured and rotatable to-25 gether.

In any case, the rotation of the agitation member (tank and/or screw) causes the progression of the suspension from the inlet 5 of the tank 1 to the outlet 6. The suspension to be treated is continuously fed in through a $_{30}$ coaxial supply pipe 7 in the inlet 5, is subjected to the treatment during its progression through the tank 1, and the treated suspension flows out through an emptying opening 8 to a utilization circuit 9 (for filtration and drying or agglomeration or diluting. etc.). 35

According to another embodiment, the continuous progression of the bath to be treated is effected by the application of a difference in pressure between the inlet and outlet ends.

The paths and distributions of speed in the fibrous sus- 40 pension subjected to the above mentioned slight turbulent agitation, in the case where the tank 1 is cylindrical and is rotated in the direction of the arrow F about its horizontal axis is shown in FIG. 6.

The solid line 2 represents the shape of the surface which is free from the suspension during this rotation. ⁴⁵ The dotted lines **35** and **36** in particular, show the shape in projection on a straight section of the cylinder of the orbits followed by the suspended particles which for the most part are generally constituted by the fibres. These orbits are not stable for various reasons (beatings due to the drive by the flat walls limiting the ends of the cylindrical curve, variations in the dimensions of the particles in the course of the aggregation, vibrations and so on), but the projection retains the general shape indicated in the drawing. ⁵⁵

Tests have shown that a fibre AB (which is shown considerably enlarged in order to facilitate understanding and which is assumed to be in the plane of the cross section) has at its ends linear speeds VA and VB which are different at any given instant, on account of the shape of the orbits imposed by the shape of the tank and its movement and on account of the sliding of the layers of fluid due to the inertia of the suspension in the course of its drive under the action of the walls.

The gradient of speed thus existing between the different layers of fluid causes a shearing in the center of the suspension and produces a torque determining the rotation of the fibres. Moreover, the speed VR applied to the center of gravity of the rotating fibre AB in question causes the circulation of this fibre along the indicated orbits.

The force M at point C communicated to the suspension by the movement of the wall of the tank is vectored with the action of gravity P at the same point and deter-75 or synthetic fibres, the optimum concentrations may be

mines the shape of the orbit followed by the fibres as shown by the resultant R.

In the course of these rotational and circulatory movements in a shearing field, movements which undergo accelerations (positive or negative) mainly in the areas C and D, there occur shocks between the fibres if the concentration is sufficient and consequently these fibres cling together.

Apart from this experimental illustration of the method of the invention, the present invention has as an object the increasing of the efficiency of the treatment, and improving the output of the products obtained and of varying these latter and their applications.

In accordance with the invention, the concentration by weight of the fibres in the suspension, at the beginning of the treatment is, lower than 15%. In the case of conventional papermaking fibres, this original concentration is preferably between one and four percent. In connection with leafy wood fibers (i.e. fibres from leafy European type trees), the production of aggregates is maximum, based on equal durations of treatment and if the composition is not modified in the course of treatment, when the original concentration of fibres is between 2.8 and 3.4%.

According to another feature, the concentration of the fibres may be modified during the treatment. Similarly, the composition of the fibrous suspension may be modified during this treatment. Moreover, a plurality of successive additions of fibres having different qualities and

30 properties, may be provided during the same treatment. Further features of the invention relate to modifications in the method consisting of well determined aims in incorporating the additional products either in the suspension itself or on or in the aggregates, whether the latter 35 be simply drained or dried. In any case, these particular features will be clearly understood from the detailed description which follows.

It is specified above that the fibrous suspension must remain sufficiently fluid in order to permit the fibres to move with respect to one another in order to be organized into aggregate form under the action of liquid currents.

In order that the fibres have a sufficient degree of freedom, their concentration in the liquid must remain relatively low. The most effective zone of concentration at the beginning of the treatment for the conventional paper-making fibres, as stated above, is between 1 and 4% by weight with respect to the fluid.

For leafy wood fibres treated for ten hours in a rotating cylindrical tank whose axis is inclined by about 45° with respect to the vertical, the peripheral speed of this tank being 35 meters per minute for a diameter of 20 cm., the variations in the production and the yield of the particles as a function of the concentration of the fibres in the suspension are given by the curves illustrated in FIG-URE 6.

In this figure, the x axis shows the percentage concentration (C%) which is equal to the ratio of the weight of the fibres placed originally in the suspension and of the weight of the fluid of this suspension; the solid line of the y axis shown the production (Pr) of the particles formed in four liters of suspension, this production being expressed in grams; the dotted line of the y axis shows the yield (R%) of the treatment which is equal to the ratio of the weight of the particles formed and of the total weight of the fibres placed originally in suspension.

The solid line curve 37 represents the variation in the production Pr and shows that this production is maximum, for a fixed duration of treatment and if the composition is not modified during this treatment when the concentration C% is between 2.8 and 3.4%. The dotted curve 38 shows the variation in the yield R% and shows that this yield is maximum, under the same conditions as previously, when the concentration C% is between 2.4 and 2.8%. Of course, for other natural or synthetic fibres the optimum concentrations may be

higher. They depend upon the length and the suppleness of the fibres. Moreover, if new fibres are added during the development of the aggregate, the optimum concentration may be moved towards denser zones. In any case, the profitable zones of concentration generally remain lower than 15%.

Thus, the concentration of the fibres may be modified during the treatment. Several successive additions of fibres of different qualities and properties may even be provided during the same treatment, for the particular 10 purpose of obtaining a stratification.

Moreover, the fibrous suspension may comprise not only a fluid and fibres but also other products responding to the well-determined aims which are mentioned hereinafter. In any case, the composition of this fibrous 15suspension may be established once and for all at the beginning of the treatment, may be also maintained constant during this treatment, or may even be modified in the course of said treatment.

It is important to note that the particles obtained, $_{20}$ whether they be constituted by simple aggregation without any binding agent or whether they are mixed or covered with other products, have a structure which is substantially isotropic, i.e. without any preferential orientation of the fibres. This property has particular interest $_{25}$ in all the applications envisaged in the following.

In order to favor the formation and development of the fibrous aggregates, germs or primers are introduced into the suspension at the beginning of the treatment. The latter may be constituted by mineral or organic 30 powders, crystals, particles of natural or synthetic resins, sawdust, particles of cork or bark, fibrous agglomerates which may or may not be manufactured for this purpose during a previous operation or produced by the disintegration of paper-making pulp or old papers, disintegrated 35 cardboard or the like.

In order to favor the agglomeration of the fibres, gaseous bubbles may be diffused in the center of the suspension.

The composition of the fibrous suspension treated for 40 the purpose of granulation may be very complex. Concerning the fibres, they may vary considerably in dimension, shape and nature and utilized alone or in a mixture (mineral fibres; asbestos, glass, drawn rock, drawn metals; various natural, regenerated or synthetic fibres which are $_{45}$ or are not chemically modified, and so on). Apart from these fibres, additional products may be introduced into the fluid which are intended either for facilitating or controlling the particulation, or for conferring to the particles particular properties which will substantially be useful. 50

Concerning the additional products intended to facilitate or control the particulation, one may use the above mentioned primers, flocculating agents, dispersing agents, moisturizing agents or others, products in solution or in suspension which are precipitable or not during the treat-55 ment, such as latex for example, agents modifying the physical or chemical properties of the liquid medium viscosity, pH, etc. or modifying the superficial or internal properties of the fibres and other elements in suspension.

Concerning the products intended to give particular 60 properties to the granules for later use, products in solution, emulsion, suspension, encapsulated products and the like may be incorporated in the suspension, so that they become integrated by inclusion, adsorption, chemical contact, grafting, with the fibrous network of 65 the aggregates. These products may be mineral or organic charges, adhesives, gum, polymers or prepolymers, natural or synthetic, thermoplastics or hardenable resins, various monomers, polymerizable or cross-linkable condensable products. 70

The fluid which is generally utilized for placing the fibres in suspension is generally water. But it is obvious that other pure or mixed liquids may be used. In particular, the fluid may be entirely constituted by polymerizable, condensable or cross-linkable products. Reactions may also be provoked either in the medium where the particles are located or in the particles after draining one part of the liquid medium which surrounds them, or even after drying the particles. Moreover, these latter may be treated in the vapor phase by polymerizable, condensable or cross-linkable products. They may also be

latter may be treated in the vapor phase by polymerizable, condensable or cross-linkable products. They may also be covered by a simple projection of various products such as those mentioned above. In any case, the reactions may be triggered by any conventional means such as catalysis, irradiation and so on, possibly permitting graftings on the elements constituting the particles.

Moreover, in order to obtain particles which are expanded in part, porophores or inflating agents may be included, for the purpose of developing small bubbles, forming foam. This expansion of the alveolar product thus obtained may be caused either during the manufacture of the particles or granules, or only at the moment when they are used during later operations, and this expansion may affect all or only part of said particles or granules.

The result is that numerous modifications of the method can be carried out. They may be carried out separately from one another or in various combinations and, consequently, a very wide range of varied products can be obtained.

When the composition of the fibrous suspension is modified during the particulation or granulation by new additions, the program of the development of the operations is established as a function of the aim to be achieved and the product to be obtained. Thus, the particulation or granulation may be begun with a composition comprising cheap raw fibres: mechanical pulp, pulp made from old papers, etc. and terminated with the addition of different fibres which surround the granules by giving them a better appearance, (bleached or colored fibres) or properties which are useful for later transformation of the particles or granules. Whatever is possible with the fibres is also possible with the products accompanying said fibres.

Two or more types of particles or granules can even be produced in which are included products capable of reacting together finally when particles or granules of different types are close to one another by means of an external action: mechanical, physical or chemical, irradiation, catalysis, etc., for the sole purpose of producing the most varied effects: change of color, variations in size or mechanical properties, release of volatile products, etc.

Several examples are hereinafter given in a non-limiting manner to illustrate the method of the invention:

EXAMPLE 1

For treating a suspension in water of blanched beech fibres having a concentration of 3%, treatment is effected under the following conditions: the tank (FIGURE 2) is made of polyvinyl chloride and is rotated about its axis, which is inclined at 45° . The ratio of the diameter of the cylinder to its height is about 1. The peripheral speed of the cylindrical wall is 40 meters per minute. After five hours of treatment, almost all the fibres of the suspension are separated into small regular pellets with a diameter of about 1 mm.

EXAMPLE 2

There is treated, under the same conditions as in Example 1, a suspension of resinous fibres. Fibrous pellets are then obtained, the diameter of which is about 3 mm.

EXAMPLE 3

For treating a suspension of spruce fibres (unbleached kraft pulp) at a concentration of 1.5%, the apparatus diagrammatically illustrated in FIGURE 1 is rotated about its axis at a peripheral linear speed of 30 meters per 75 minute. After several hours of agitation, fibrous pellets

8

with a diameter of about 5 mm. are separated from the suspension.

EXAMPLE 4

For treating a suspension of fibres of blanched straw with a concentration of 3%, the apparatus diagrammatically illustrated in FIGURE 3 is used. After six hours of treatment, the suspension contains about 80%of agglomerated fibres in the form of small pellets of different sizes, their diameters being between 0.5 mm. and 2 mm. 10

A great majority of the accessory elements of the straw pulp (vessels, cards, etc.) remains in the suspension and the fibres tend to be regrouped in substantially equal lengths and formed into pellets having corresponding 15 diameters.

EXAMPLE 5

For the production of a very porous conglomerate, the suspension of beech pellets obtained in Example 1 is poured onto a rough sieve. The cake of pellets thus ob-20 tained is dried after being drained. The fine elements and the fibres, which remain in suspension, serve as bonds between the spheroids and thus a very light porous agglomerate is obtained, having a density of about 0.14.

EXAMPLE 6

For the production of a very resistant agglomerate, the suspension of pellets obtained in Example 1 is drained and replaced in suspension without destroying the pellets, in a water/alcohol solution of a resorcinol-formol precondensate. After agitation, drainage and drying, and a slight thermal treatment (15 minutes at 110° C.), a very rigid agglomerate is obtained with a good porosity and good machinability.

EXAMPLE 7

If it is required to produce colored fibrous granules intended to be associated with a larger quantity of bleached granules in order to manufacture a decorative covering, granulation is begun with an aqueous suspension containing 2% by weight of raw resinous paper-making fibres produced by a highly effective baking. After the granules have been formed according to the previously described method, 10% colored fibres are added with respect to the weight of the initial fibres. The treatment 45 is followed until the granules are suitably covered by raw fibres. Thus, the granules with a diameter of 6 to 8 mm. are obtained having the coloring of the fibres which were added last.

EXAMPLE 8

In this example, the requirement is to produce very cheap supple granules for manufacturing sub-layers for covering the ground. The fibres utilized are constituted by mechanical resinous pulp (paper-making pulp constituted 55 by rasped wood); moreover, fragments of disintegrated old paper are added in an amount of 15% and fine sawdust in an amount of 10%. In order to facilitate the granulation, 10%, with respect to the weight of the fibres, is added of a less stable styrene-butadiene latex whose floc- 60 culation will be produced during the granulation treatment.

After treatment of a few hours, granules are obtained with a heterogeneous granulometry or grain size, from about 1 mm. to 8 mm. 65

After draining and slight drying, these granules constitute a light raw material, of apparent density between 0.10 and 0.30, which is supple and economical for the manufacture of agglomerates.

EXAMPLE 9

For manufacturing granules covered with fibres of colored rayon for the purpose of constituting a decorative coating stratified under high pressure, after impregnation with transparent, thermosetting resins, particula-75 gates under the above conditions, the concentration by

tion or granulation is undertaken under the same conditions as for Example 7, but when the raw granules are formed, 10% of fibres of colored rayon of 1.5 denier and 2 mm. length, are added with respect to the weight of the original fibres.

After a few hours of treatment, the granules are coated with colored fibres; they are then separated from the suspension, then dried before being impregnated for the purpose of applying them under pressure onto the surface to be coated.

EXAMPLE 10

For manufacturing granules of fibres of polyvinyl chloride, for the purpose of constituting insulating supple agglomerates, polyvinyl chloride fibres of 2.5 denier and 3 mm. length are dispersed in water in an amount of 2%. After a few hours of treatment, all the fibres are agglomerated in the form of granules whose diameter is about 5 to 6 mm. At this moment 20% of a latex which is made insoluble, on the granules, is added in order to facilitate their final agglomeration.

EXAMPLE 11

For manufacturing particles or granules containing a thermoplastics polymer for the purpose of manufacturing parts for extrusion, and an aqueous suspension containing 2% of fibres of a kraft paper-making pulp and an equal weight of polyvinyl chloride in the form of fine powder, is treated. In order to facilitate the dispersion of the powder, 0.02% of a tension-active agent is added. In order to increase the fixing of the powder in the fibrous network, a flocculant polyethylene-amine for example may be added.

After a few hours of treatment, the particles or granules are drained and dried and can be used for supply-³⁵ ing a machine for pressing toilet soap into cylinders.

EXAMPLE 12

For the production of particles or granules intended for manufacturing molded parts such as packing cases, an aqueous suspension containing 1% of mechanical pulp and 2% of a pulp of highly effective birch fibres is treated. A solution of soda alginate is added in an amount of 8% with respect to the fibres; when the adjuvent is well distributed in the suspension, 5% of aluminum sulphate in solution is added by fractions. There is created a precipitate of aluminum alginate which accelerates the formation of the fibrous granules and gives them after drying an increase in cohesion and rigidity.

EXAMPLE 13

For manufacturing thermoplastics granules reinforced with fibres, for extrusion or molding purposes, a suspension of bleached beech paper-making fibres is produced in an amount of 2% in a monomer, methyl methacrylate for example. After forming the granules, the excess monomer is separated and polymerization is effected by the separated granules, by a conventional means (atomization of a catalyst, irradiation, etc.). The granules may be kept on a rotating sieve in order to limit their agglomeration. Thus reinforced granules containing a high proportion of polymer and suitable for manufacturing molded or extruded objects are thus obtained.

What is claimed is:

1. A method of treating a suspension of cellulosic fibres to form aggregates comprising subjecting a suspension of the fibres in a liquid in a cylindrical tank of a diameter of about 20 cm. to a gentle rotational agitation, with a peripheral speed of the order of 35 meters per minute whereby a speed gradient is established between different points in the suspension causing the formation of eddies, which result in the fibres moving together into groups and becoming mechanically interlocked, the grouped fibers being constituted so as to cling to one another to form aggregates under the above conditions, the concentration by

50

70

weight of the fibres in the suspension, at the beginning of the treatment, being less than 15%.

2. A method according to claim 1 wherein the concentration of the fibres is modified during the treatment.

3. A method according to claim 1 wherein to improve the formation and development of fibre aggregates, seeds are introduced into the suspension at the beginning of the treatment. 5

4. A method according to claim 1 comprising the further step of following the agitation of the bath by a filtration to separate the aggregates from the liquid medium and from the non-fibrous elements and any non-aggregated fibres present.

5. A method according to claim 4 comprising the further step of disintegrating the aggregates separated by 15 filtration and replacing them in homogeneous suspension by agitation in a liquid medium to constitute a refined pulp.

6. A method according to claim 4 comprising the further step of draining the fibrous aggregates separated by $_{20}$ filtration and then drying them in a current of warm gas.

7. A method according to claim 1, wherein the fibrous aggregates are agglomerated by bonding, and the porous conglomerate thus obtained is dried by thermal treatment.

8. A method according to claim 1 comprising the fur-25 ther step of impregnating the fibrous aggregates with a binder to constitute fibrous granules compressed while hot to form a part.

9. A method according to claim 1, wherein said agitation is produced by rotating a tank containing the suspension.

10. A method according to claim 1, wherein filler products controlling the granulation of the fibrous aggregates and chemical products are introduced into the suspension.

11. A method according to claim 10, wherein the composition of the fibrous suspension is modified during the treatment.

12. A method as claimed in claim 1 comprising impregnating the fibrous aggregates with a pre-polymer to constitute fibrous granules which are compressible while hot, to form a body.

13. A method as claimed in claim 1, wherein said agitation is produced by injecting gas into the suspension.

References Cited

UNITED STATES PATENTS

2,685,825	8/1954	Novak 162—3
3,170,834	2/1965	Oesterheld 162—3
3,297,516	1/1967	Naumann 162—3
3,421,975	1/1969	Woolery 162-3

HOWARD R. CAINE, Primary Examiner

162-3; 210-49

U.S. Cl. X.R.