

July 3, 1973

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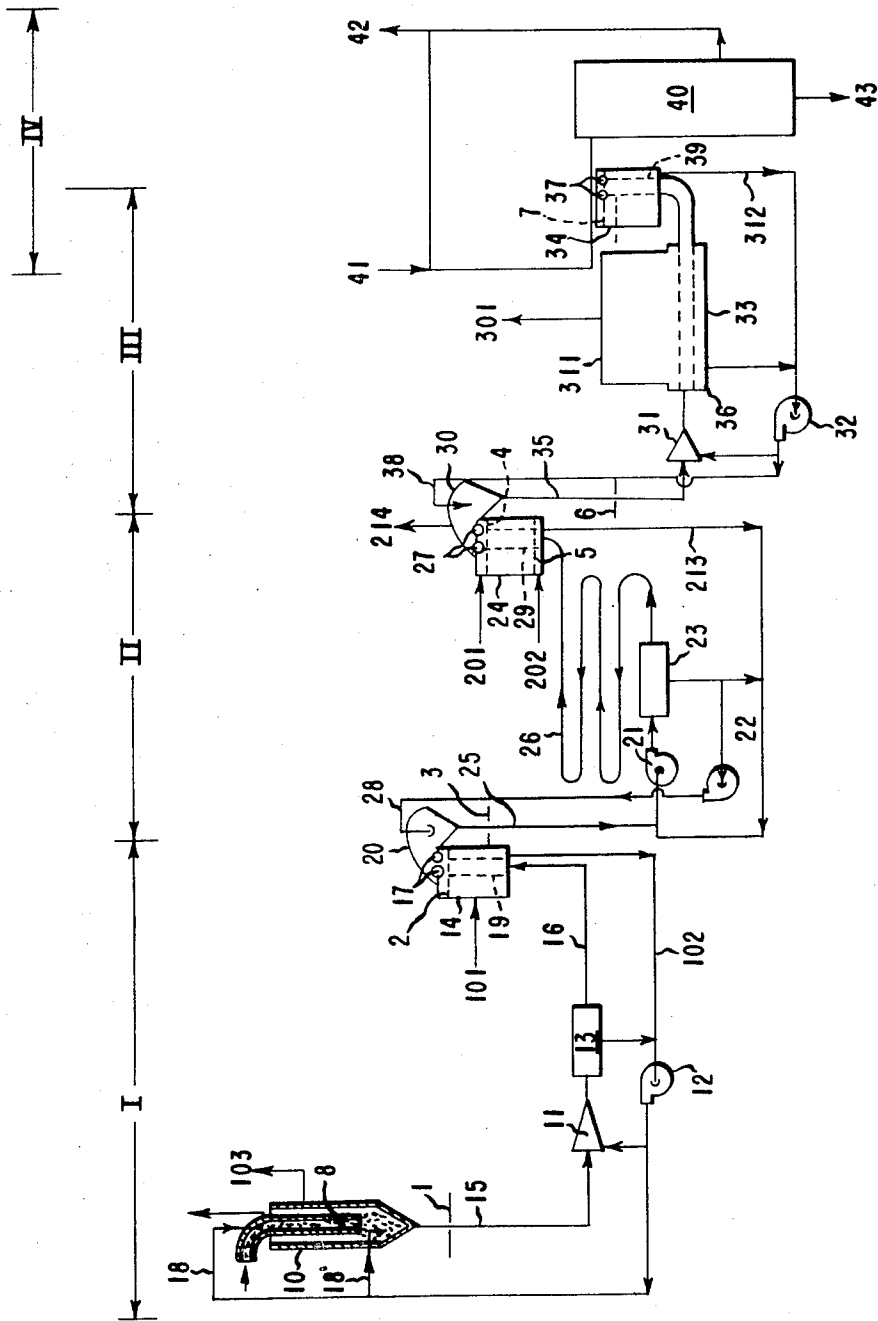
3,743,694

METHOD FOR RENDERING MICROCEL ULAR STAPLE FIBERS SELF-INFLATABLE
IN AIR INVOLVING THE CONTINUOUS TRANSPORTING OF THE FIBERS
THROUGH A REACTION PIPELINE

Filed July 13, 1971

4 Sheets-Sheet 1

FIG. 1



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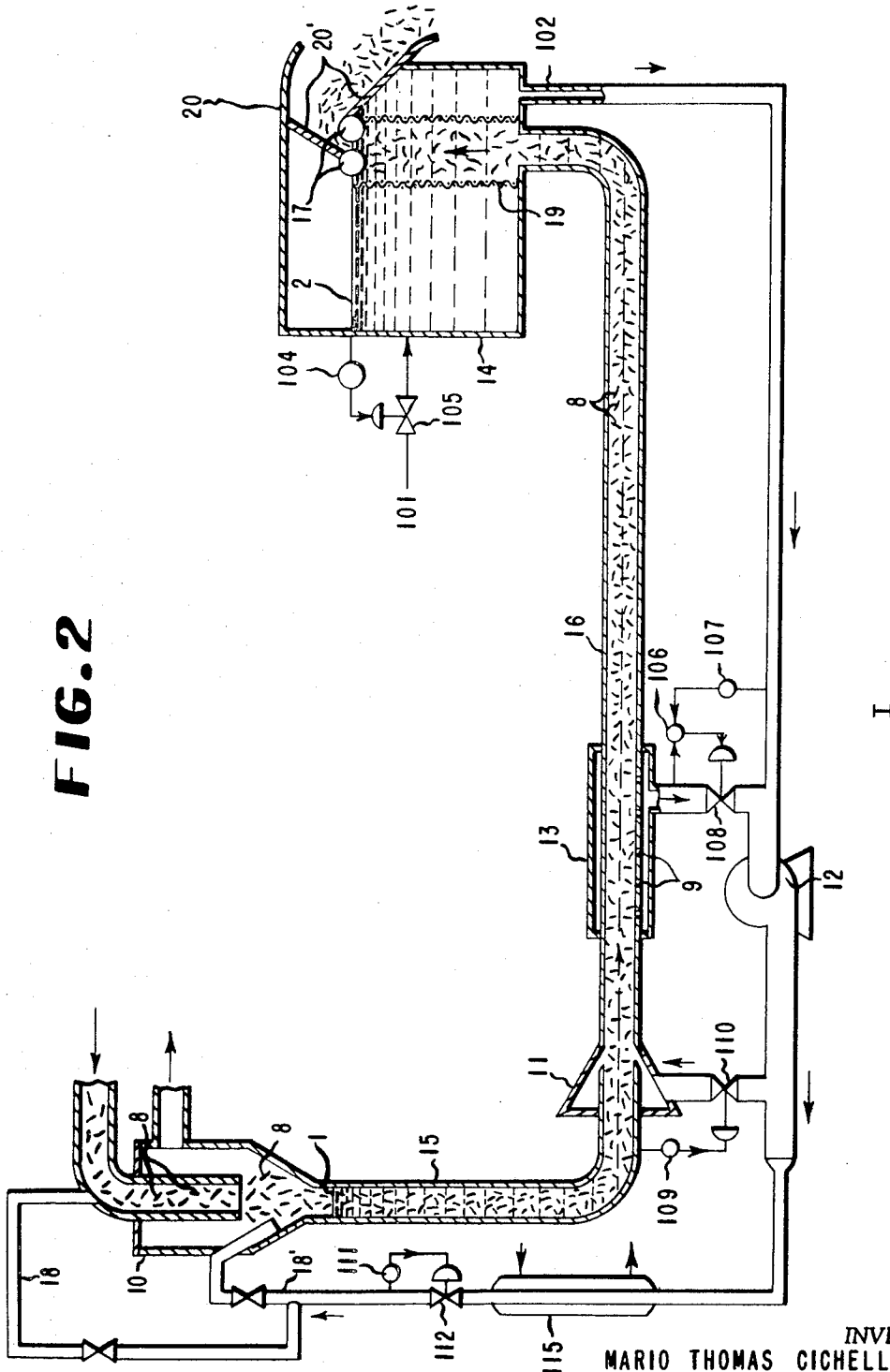


FIG. 2

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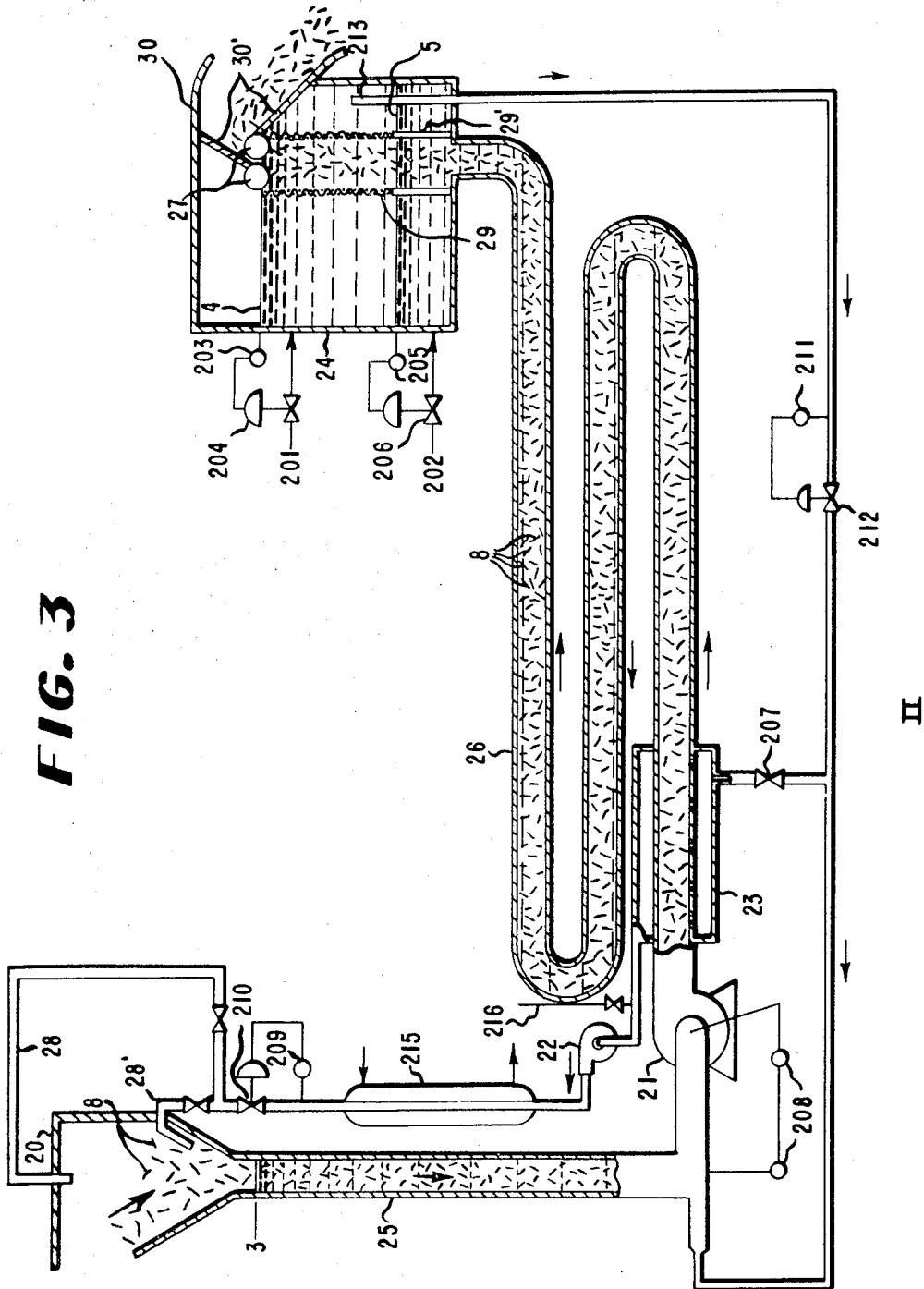


FIG. 3

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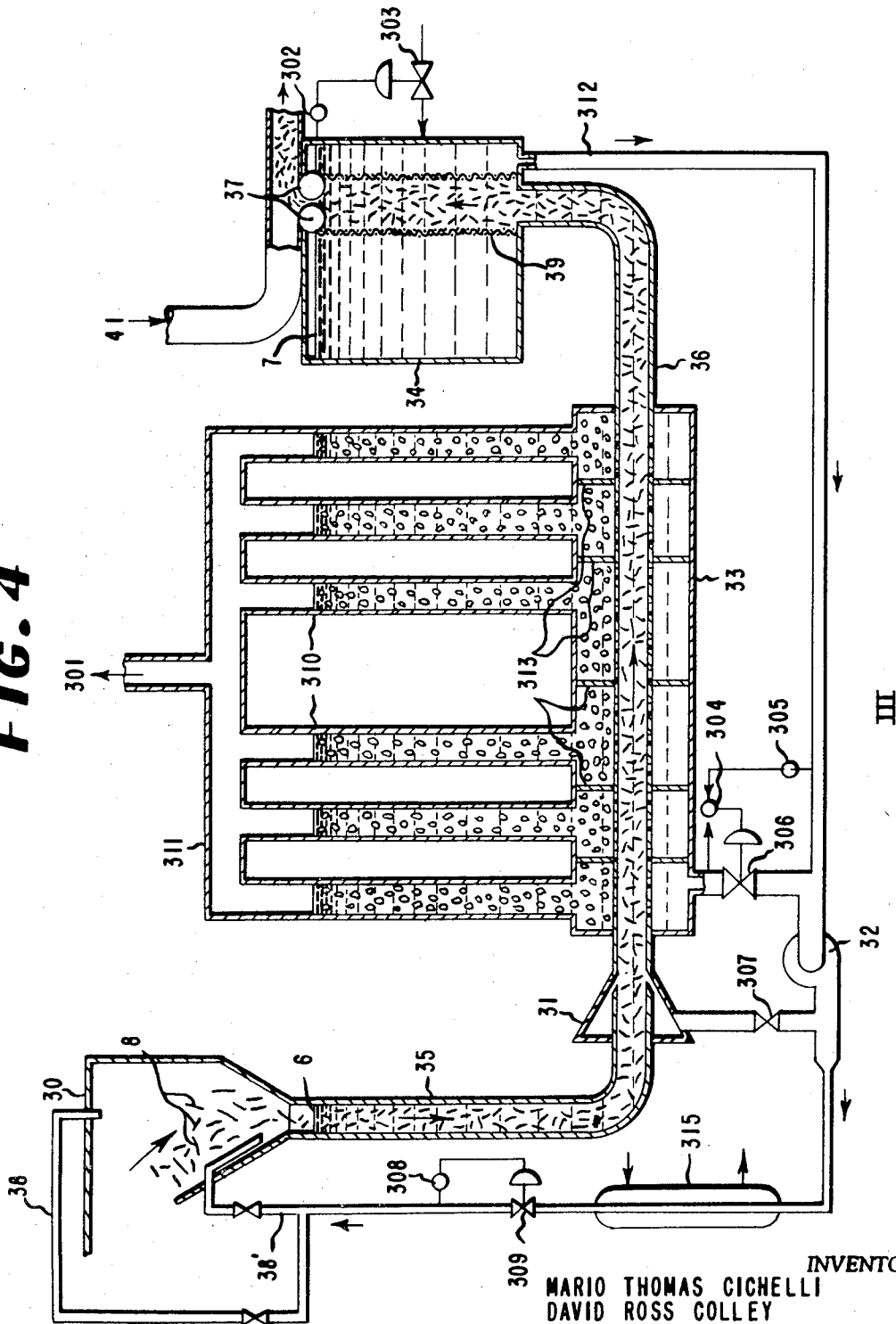
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4 Sheets-Sheet 4

FIG. 4



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METHOD FOR RENDERING MICROCELLULAR STAPLE FIBERS SELF-INFLATABLE IN AIR INVOLVING THE CONTINUOUS TRANSPORTING OF THE FIBERS THROUGH A REACTION PIPELINE

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Continuation-in-part of abandoned application Ser. No. 738,522, June 20, 1968. This application July 13, 1971, Ser. No. 162,139

Int. Cl. B29c 25/00; B29d 27/00

U.S. Cl. 264—343

4 Claims

ABSTRACT OF THE DISCLOSURE

A process is provided for rendering microcellular staple fibers self-inflatable in air. The fibers are submerged, become entangled to form clumps, or slugs, and pass through treatment pipes in "slug flow" where they are contacted with treatment liquids. All fibers have substantially the same residence time in the treatment liquids. The treatment liquids comprise: (1) "plasticizing" liquids, which plasticize the cell walls to permit fluids to diffuse into the cells, (2) "boosting" liquids, comprising impermeant inflatants which diffuse into the cells while the walls are plasticized, but normally do not permeate the cells when the walls are not plasticized, and (3) "stripping" liquids, which remove plasticizing liquid, deplasticize the cell walls and trap impermeant inflatant within the cells. Fibers so treated when exposed to air become fully inflated.

REFERENCE TO RELATED APPLICATIONS

This application is a continuation-in-part of application Serial No. 738,522, filed June 20, 1968, now abandoned.

BACKGROUND OF THE INVENTION

(1) Field of the invention

This invention relates to buoyant, closed-cell polymeric, foamed, staple fibers, sometimes referred to as microcellular fibers, and more particularly to a method and apparatus for transporting such fibers in "slug flow" while submerged in a special treatment liquid. More specifically, the invention relates to a pipeline process for postinflating such fibers, and to apparatus for carrying out this process.

(2) The prior art

Foamed polymeric materials are widely used for cushioning applications. Generally they fall into three classifications: (1) those of elastomeric polymers, e.g., rubber and certain polyurethanes, which derive their cushioning properties primarily from elasticity of the copolymer; (2) those of relatively rigid polymer structure which tend to provide good impact energy absorption without resilient regain of initial dimensions; (3) and those of thin-walled closed-cell foamed polymer which confine gases within the closed cells and are pneumatically resilient. This invention is concerned with foamed fibers, or microcellular fibers, of the latter type, particularly staple fibers which are collected and adhesively bonded at fiber-to-fiber contacts to yield cushioning batts.

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Known gas-inflated closed-cell foams have had, in general, certain deficiencies. Ordinarily they are air-inflated. Rarely, however, is air the gas originally contained within the cells immediately after closed-cell foam-formation, the original gases having usually permeated out of the cell-walls rapidly. Thus, exposure to air results in an exchange of gases until air becomes the sole gas contained. Depending on the relative permeation rates for air and for the gas originally present, the period during which gas-exchange occurs ordinarily is characterized by dimensional changes in the product, very often by partial or complete collapse of the cells of the foam. The gas pressure within the cells at equilibrium with ambient air cannot exceed atmospheric pressure, and, if collapse occurs during gas-exchange, there remains no spontaneous mechanism for reinflating the cells to their maximum volumes established when the polymer in the cell walls solidified during foam expansion.

For cushioning applications, however, a more serious deficiency characterizes customary pneumatic foams, even if they are prepared in fully inflated form. Air is composed of small, rapidly permeating molecules. While transient compressive loads may result in little or no loss of air, repeated or sustained compressions eventually cause most or all of the air to escape by permeation. Once air is lost in this fashion, it is not spontaneously regained, and pneumatic resilience is permanently diminished or destroyed.

British Pat. No. 1,062,086, published Mar. 15, 1967, discloses a process whereby the above disadvantages of pneumatic, closed-cell foams are overcome. According to this process, an impermeant inflatant is introduced within the closed cells to inflate them. The impermeant inflatant is a normally gaseous material of relatively large molecular size and is chemically inert toward and substantially completely insoluble in the polymer comprising the foam. Examples of impermeant inflatants include sulfur hexafluoride and saturated aliphatic and cycloaliphatic compounds having at least one fluorine-to-carbon covalent bond and wherein the number of fluorine atoms exceeds the number of carbon atoms, such as perfluorocyclobutane and chloropentafluoroethane.

The impermeant inflatants permeate the cell walls so slowly as to be substantially permanently retained within the foam cells—regardless of the number, frequency, or duration of the compressive loadings. The presence of impermeant inflatant within foam cells creates an osmotic gradient for the inward permeation of air. At equilibrium with the ambient atmosphere, the internal partial pressure for air becomes substantially atmospheric. This, combined with the partial pressure for impermeant inflatant, provides a superatmospheric total internal pressure and guarantees full reinflation of the closed cells. Moreover, even if some air is lost during compressive loading, impermeant inflatant remains and causes spontaneous regain of both volume and pneumaticity of the foam structure when the load is removed.

One method for introducing impermeant inflatant to the closed cells of the foam is to incorporate the impermeant inflatant in the foamable composition prior to its extrusion to form foam fibers. However, the foamed fibers are preferably postinflated in place of or in addition to the introduction of impermeant inflatant directly by extrusion.

"Postinflation" is the name applied to the overall process in which previously formed foam, either collapsed

or gas-inflated, is: (1) exposed to impermeant inflatant while the cell walls are plasticized and temporarily of greatly enhanced permeability; (2) treated to quickly strip off the plasticizing agent by vaporization; and (3) equilibrated in air (preferably heated) until the foam at least regains its maximum volume. Further exposure to air might increase the internal gas pressures somewhat, but it should produce substantially no further volume increase.

Step (1) above can be accelerated somewhat by subjecting the foam immersed in impermeant inflatant treatment fluid to elevated pressures, but it is difficult to handle large volumes of continuously formed foam staple in such a process. A continuous, substantially atmospheric pressure process is preferable. Although the treatment fluids may be either gaseous or liquid, liquid-phase treatment is preferred.

In a previously employed atmospheric postinflation process, as disclosed in Bonner, U.S. Pat. 3,381,077, the foamed, closed-cell, polymeric, staple fibers are continuously conveyed on or between reticulate conveyor belts as beds of fiber, being drawn sequentially through three closed vessels where they are either immersed in or showered by the liquid phases contained therein. The first vessel contains a "plasticizing" liquid preferably heated to its normal atmospheric boiling point. The second contains a substantially saturated solution of impermeant inflatant in plasticizing liquid which is called a booster liquid. This booster liquid is also usually maintained at substantially the normal atmospheric boiling point of the mixture. The third contains a "stripping liquid" such as water (or other similarly inert fluid) at a sufficiently elevated temperature to quickly vaporize substantially all of the residual plasticizing fluid and the superficial impermeant inflatant. In this manner, plasticization of the cell walls is so suddenly terminated that impermeant inflatant becomes trapped within the closed cells of the foam fibers. The vapors are simultaneously withdrawn to appropriate solvent-recovery means. Thereafter, postinflation is completed by exposing the fibers to heated air at a temperature below the melting or flowing temperature for the polymer, e.g., usually at 70 to 175° C. Liquid/vapor seals are, of course, provided at both entrance and exit of each vessel.

Alternatively, and as also disclosed in Bonner U.S. Pat. 3,381,077, the plasticizing and the inflating of the fibers (carried out in the foregoing paragraph in the first and second vessels, respectively) may be combined and may be carried out simultaneously.

While the above procedures are workable on a continuous basis, a continuous procedure has heretofore had several disadvantages. It is so mechanically complex, with myriad drive and supporting rolls, that mechanical failure is frequent. This tendency is severely aggravated by the necessity for exposing a majority of the moving parts to corrosive vapors and liquids. Finally, liquid/vapor seals between vessels large enough to accommodate beds of fibers are inefficient; consequently, the load on and expense of solvent-recovery are rather great.

SUMMARY OF THE INVENTION

This invention provides an improvement in the process for treating microcellular staple fibers at substantially atmospheric pressure to render them postinflatable upon exposure to air by contacting the fibers with a (1) plasticizing liquid, (2) a liquid containing an impermeant inflatant (sometimes referred to as a boosting liquid), and (3) a stripping liquid. Liquids (1) and (2) can be combined, if desired. The improvement comprises continuously transporting the fibers through a reaction pipeline with such treating liquid in slug flow at a rate of one to two feet per second.

The fibers used in this invention tend to collect into coherent, interentangled clumps (or "slugs") as they are submerged in the liquid and begin to move through the

pipe-length; hence, the expression "slug flow." Since no individual fiber can proceed through the pipe slower or faster than the clump (or "slug") it is in, obtainment of slug flow assures that all fibers will receive about equal treatment times in the treating liquid as they move through the pipe. Obtainment of slug flow is the key to usefulness of this invention since it enables treatment of the buoyant fibers to take place in the absence of the complex mechanical equipment described previously. It is surprising and unexpected that slug flow is attained because of the turbulent flow conditions that exist as the fibers and treating liquid pass through the pipe (0.5 to 5 feet per second and preferably 1 to 2 feet per second) and because of the buoyancy of the fibers (a density between 0.005 and 0.1 gm./cc.).

The invention also provides the utilization of apparatus for carrying out the above-described process which comprises a generally horizontal pipe-length, means at the entrance end of the pipe-length for submerging the fibers in the treatment liquid and introducing the fibers and liquid to the pipe-length, means in the pipe-length for providing sufficient head to the liquid to move the fibers and liquid through the pipe-length with the fibers in slug flow, and means at the exit end of the pipe-length for separating the fibers from the liquid.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic representation in elevation of an improved postinflation process and apparatus provided by this invention.

FIG. 2 is an enlarged, schematic, cross-sectional view in elevation of apparatus suitable for the plasticizing section I of FIG. 1.

FIG. 3 is a similar view showing the boosting section II of FIG. 1.

FIG. 4 is a similar view showing the stripping section III of FIG. 1.

DESCRIPTION OF THE INVENTION

The invention is an improvement in the process of post-inflating microcellular staple fibers, i.e., fibers that are buoyant, polymeric, closed-cell, foamed staple fibers. Both these microcellular fibers and processes for inflating them are known in the art. The fibers are made of a foamed polymer having discrete polyhedral shaped closed cells defined by cell walls less than two microns in thickness, with substantially all of the polymer being present in the cell walls. As described in U.S. Pat. 3,227,664 and 3,381,077, these microcellular fibers contain at least 10^3 cells/cc., the average transverse dimension of the cells is ordinarily less than about 1000 microns in the expanded state, and the cell walls exhibit uniplanar orientation and uniform texture. The fibers, when in a collapsed state, have densities usually in the range from 0.05 to 0.1 gm./cc.; but when fully inflated their densities are in the range from 0.016 to 0.030 gm./cc., and their diameters are from about 0.03 to 0.10 inch. The length of the staple fibers is from about 1.5 to 8 inches. Preferably, the fibers are made of polyethylene terephthalate prepared as described in the aforesaid U.S. patents.

The microcellular staple fibers in the collapsed state can be postinflated (called postinflated to distinguish from inflation occurring during the spinning of the fibers) by exposing them to impermeant inflatant while the cell walls are plasticized and temporarily of greatly enhanced permeability. The impermeant inflatant permeates the cell walls easily due to the plasticized state of the walls, but when the walls are not plasticized, the impermeant inflatant permeates the cell walls so slowly as to be substantially permanently retained within the cells. Thus, after exposure to the inflatant the fibers are treated to quickly strip off the plasticizing agent by vaporization. The presence of the inflatant within the walls creates an osmotic gradient for permeation of air in the cells. Thus, on exposure to air, an equilibrium is established wherein the

partial pressure of air in the cells becomes essentially atmospheric. This combined with the partial pressure of the inflatant, produces a superatmospheric pressure within the cells, thereby inflating them. In their inflated state, the fibers are very light weight and highly buoyant, and this pneumaticity makes the fibers highly suited for use in cushioning structures.

As described above, previous art processes for post-inflating the fibers employed conveyor belts and required mechanical handling in transporting them from one conveyor belt to another. In the present invention, it has been discovered that even though the microcellular fibers are of low density, and therefore buoyant, they can be submerged in the treatment liquids and transported through pipes by means of a state termed "slug flow." The term "slug flow" as used herein describes the tendency of the microcellular fibers to collect and become entangled in clumps, or "slugs" as the fibers are submerged in the treatment liquid and moved through the pipe. Because of the velocity and turbulence of the treatment liquids moving through the pipe, it is surprising that the light, microcellular staple fibers are able to form clumps or slugs. Since the fibers travel in slugs, the residence time of each individual fiber in the pipes is about the same, i.e., the individual fibers do not travel at different velocities and do not pass one another, or the like. Very few individual fibers pass through the pipeline independently. When the concentration of the fibers in the treatment liquid is low, small discrete clumps of fiber form, separated by relatively long sections of clear treatment liquid in the pipeline. When the fiber concentration is somewhat higher, but still relatively low, somewhat larger size clumps, approaching the size of tennis balls, can form. These too are transported by the treatment liquid in slug flow through the pipeline as discrete clumps separated by sections of clear treatment liquid. At still higher concentrations of fiber in the treatment liquid, the formed clumps can approach three to four feet in length. These too pass through the pipeline as discrete clumps in slug flow but are separated by shorter sections of clear treatment liquid. Further increases in fiber concentration can cause an essentially continuous clump to form and be transported by the liquid through the pipeline. As the concentration of fiber increases, the size of the clumps increases and the distance of travel in the pipeline before clumps start to grow decreases. However, for all these described conditions, the residence times of all fibers are substantially identical since no fiber can proceed through the pipe slower or faster than the slug it is in.

Although the reason for the formation of clumps or slugs is not completely understood, it is believed to be due at least in part to the unique surface characteristics of the microcellular staple fibers.

Suitable microcellular fibers for use in this invention are prepared from synthetic crystallizable, organic polymers, and as described in U.S. Pat. 3,227,664, include polyhydrocarbons such as linear polyethylene, stereo-regular polypropylene or polystyrene; polyethers such as polyformaldehyde; vinyl polymers such as polyvinylidene fluoride; polyamides both aliphatic and aromatic, such as polyhexamethylene adipamide and polymetaphenylene adipamide and polymetaphenylene isophthalamide; polyurethanes, both aliphatic and aromatic, such as the polymer from ethylene bischloroformate and ethylene diamine; polyesters such as polyhydroxyypivalic acid and polyethylene terephthalate; copolymers such as polyethylene terephthalate-isophthalate, and the like. The polymers are of at least film forming molecular weight.

As described in U.S. Pat. 3,381,077, suitable plasticizing liquids for use in the process of this invention should have relatively small molecules in order to promote permeation through the cell walls and must plasticize (e.g., swell) the cell walls without dissolving other than very minor proportions of the polymer and at below the fluid's atmospheric boiling point. At high temperatures,

and under pressure, most suitable plasticizing liquids dissolve at least a significant portion of the polymer. Since it must be readily and rapidly volatilized on exposure to air, the plasticizing liquid should have an atmospheric boiling point of less than about 85° C. The rate of plasticization increases with increasing temperature; so operation near its boiling point is preferred. Prolonged exposure to plasticizing liquid can change the crystalline structure and molecular orientation of the polymer in the cell-walls, usually increasing their permeability; so the shortest adequate plasticizing times are employed, usually in the range from 5 to 60 seconds. Thus, the term "plasticizing liquid" as used herein means a volatile, low boiling liquid substance that is essentially a non-solvent for the polymer employed at or below the boiling point of the fluid, but has sufficient solvent power to swell, i.e., plasticize, the fiber polymer. Suitable plasticizing liquids for foamed fibers of polyethylene terephthalate include methylene chloride, chloroform, ethylene chloride, and dichlorofluoromethane. Methylene chloride with a boiling point of about 41° C. is preferred not only because it is optimum in the above properties but also because it is relatively inexpensive, nontoxic except in extreme exposures, and substantially nonflammable.

The term "impermeant inflatant" as used herein means a gas or liquid of low boiling point which permeates the cell walls of the unplasticized microcellular staple fibers so slowly, as compared to air, that it is substantially permanently retained within the closed cells, even under compression, but which readily permeates the cell walls of the plasticized fibers, and which has a large molecular size as is consistent with a vapor pressure of at least 50 mm. of mercury at normal room temperature. As described in U.S. Pat. 3,381,077, suitable impermeant inflatants are selected from the group consisting of sulfur hexafluoride and saturated aliphatic or cycloaliphatic compounds having at least one fluorine-to-carbon covalent bond and wherein the number of fluorine atoms preferably exceeds the number of carbon atoms. Preferably these inflatants are perhaloalkanes or perhalocycloalkanes in which at least 50% of the halogen atoms are fluorine. Although these inflatants may contain ether-oxygen linkages, they are preferably free from nitrogen atoms, carbon-to-carbon double bonds, and reactive functional groups. Specific examples of impermeant inflatants include sulfur hexafluoride, perfluorocyclobutane, sym-dichlorotetrafluoroethane, chloropentafluoroethane, perfluoro-1,3-dimethylcyclobutane, perfluorodimethylcyclobutane mixtures, 1,1,2-trichloro-1,2,2-trifluoroethane, $\text{CF}_3\text{CF}_2\text{CF}_2\text{OCF}_2\text{CF}_3$, chlorotrifluoromethane, and dichlorodifluoromethane. Particularly preferred because of its inertness, appreciable molecular size, very low permeability rate, and lack of toxicity is perfluorocyclobutane with an atmospheric boiling point of about -6° C., especially when the microcellular fibers are prepared from polyethylene terephthalate.

The term "stripping liquid" as used herein means a liquid at normal conditions that is a nonsolvent for the microcellular fibers and nonreactive with the impermeant inflatant and the plasticizing fluid. The function of the stripping liquid is to aid in physically stripping excess inflatant and plasticizer from the fibers. A wide range of liquids may be employed, however, water is preferred.

The process of this invention, i.e., the transporting of the buoyant microcellular staple fibers through the pipeline in slug flow, can be carried out with the three stage sequence disclosed in U.S. Pat. 3,381,077, i.e., the treatment of the fibers sequentially (1) in a plasticizing liquid to temporarily increase the permeability of the cell walls; (2) in a boosting liquid comprising impermeant inflatant to introduce impermeant inflatant into the closed cells; and (3) in a stripping liquid substantially inert to and insoluble in both the polymer and previous treatment liquids to vaporize and remove excess external impermeant inflatant and substantially all the residual plasticizing

liquid and to trap the remaining impermeant inflatant within the cells. Alternatively, the process may be carried out by the two stage sequence disclosed in co-assigned copending United States patent application Ser. No. 162,281, filed concurrently herewith, where no separate plasticizing step is used, but rather both the plasticizing and boosting functions are provided by a treatment liquid consisting of a dilute dispersion or solution of plasticizer and impermeant inflatant in water.

In the three-stage procedure, the process of this invention is carried out continuously by submerging the fibers first in the plasticizing liquid and moving the fibers and plasticizing liquid through a first pipe-length in slug flow, separating the fibers from the plasticizing liquid, submerging the fibers in the boosting liquid and moving the fibers and boosting liquid through a second pipe-length in slug flow, separating the fibers from the boosting liquid, submerging the fibers in the stripping liquid and moving the fibers in slug flow and stripping liquid through a third pipe-length, and separating the fibers from the stripping liquid. In the two-stage procedure, the process of this invention is carried out by submerging the fibers in the aqueous dispersion or solution of the plasticizing and boosting liquids, and transporting them in slug flow through a pipeline of sufficient length to permit the cell walls to become plasticized and the impermeant inflatant to enter the cells. The fibers are then separated from this first treatment liquid, submerged in a stripping liquid, moved in slug flow with the stripping liquid through a second pipe-length and then separated from the stripping liquid. The resultant fibers are self-inflatable in air.

The invention also provides an apparatus for performing the slug flow process which comprises three successively coupled pipe-lengths, means at the entrance end of each pipe-length for submerging the fibers in a treatment liquid and introducing the fibers and liquid to the pipe-length, means in each pipe-length for moving the liquid and fibers through the pipe-length with fibers in slug flow, means at the exit end of each of the first two pipe-lengths for continuously separating the fibers from the bulk of the treatment liquid and transferring the fibers to the submerging means of the next pipe-length, means at the exit end of the third pipe-length for continuously separating the fibers from the bulk of the treatment liquid and transferring them to a holdup tank. Of course, in the two-step procedure only two successively coupled pipe-lengths are needed.

The slug flow process will now be described with respect to the three-step postinflation procedure of the prior art and with respect to the drawings (in which the various parts are designated by the same numerals throughout).

PLASTICIZING SECTION

With reference to FIGS. 1 and 2, the plasticizing section is designated by the Roman numeral I. Microcellular foamed staple fibers 8 are continuously supplied to a collection vessel 10. Vessel 10 is usually vented via line 103 to a condenser (not shown) for liquefying vapors of plasticizing fluid and for maintaining substantially atmospheric pressure. A generally horizontal pipe-length 16 has a vertical submerging section 15 and turns upward at its other end to join coupling tank 14. Respective liquid levels for plasticizing liquid in these two portions are indicated by numerals 1 and 2. As the very low-density fibers 8 approach liquid level 1, they are submerged by a stream of plasticizing liquid from lines 18 and 18'. Liquid-liquid eductor 11 moves the fibers and liquid through pipe-length 16 to coupling tank 14. In generating sufficient pressure to forward the fibers and liquid, eductor 11 introduces an excess of plasticizing liquid. Consequently, a regulator 13 is provided which is simply a shell surrounding a portion of pipe-length 16. Within regulator 13, pipe-length 16 has openings 9 sized to allow liquid but not fibers 8 to flow through. In this way, excess plasticizing liquid is drawn from regulator 13 by circulating pump 12.

Fibers 8 are moved through pipe-length 16 in slug flow. On reaching the end of pipe-length 16, fibers 8 float to the liquid surface 2, where they contact cooperating, rotating, squeeze-rolls 17. A screen-enclosed channel 19, larger in cross-section than is pipe-length 16, allows ready flow therethrough of plasticizing liquid while preventing the straying of fibers 8.

Squeeze-rolls 17 form a seal between section I and II by forcing excess plasticizing liquid back into coupling tank 14. Simultaneously they forward fibers 8 via connecting baffle 20, on to section II. Frequently, jets (not shown) of boosting liquid are employed to assist in transfer of fibers 8 through baffle 20. Suitable squeeze-rolls may have either compressible or rigid surfaces, the latter preferably provided with shallow ridges to aid in catching fibers 8. Squeeze-rolls 17 cooperate with surrounding surfaces 20' to form a seal against loss of treatment fluid and ordinarily have diameters approximately equal to the diameter of pipe-length 16 where it enters coupling tank 14, and equal lengths sufficiently greater than said diameter that transport of all fibers floating to the nip is assured.

Liquid recirculation is provided by circulating pump 12 which withdraws liquid from pipe-length 16 via regulator 13 and from coupling tank 14 via pipe 102. Simultaneously, liquid is forced into pipe-length 16 via eductor 11 and through pipe 18 for fiber submersion.

It is apparent that suitable controls must be provided for this plasticizing section and that a variety of types of controls can provide the desired balance of flow-rates. In a preferred control system, automatic valves controlled by sensing (alternatively sensing and recording) devices are employed. Thus, liquid-level sensing device 104 controls flow of plasticizing liquid at 101 through valve 105 to maintain liquid-level 2. Dual flow-rate sensors 106 and 107 provide a combined signal for withdrawal of liquid from regulator 13 via automatic valve 108. The rate of injection of plasticizing liquid into eductor 11 via automatic valve 110 is controlled by pressure sensor 109 upstream from eductor 11. Finally, flow-rate sensor 111 controls the flow of submerging liquid through automatic valve 112. Though not indicated in FIGS. 1 and 2, all exposed surfaces of apparatus should be thermally insulated, and heaters 115 must be provided at one or more points to keep the plasticizing fluid, preferably methylene chloride, at its boiling point.

BOOSTING SECTION

With reference to FIGS. 1 and 3, the boosting section is designated by Roman numeral II. In this section, the fibers 8 are exposed to impermeant inflatant while plasticization of the cell walls is maintained. Thus, the boosting liquid is usually a solution of impermeant inflatant in the plasticizing liquid.

Operation of this section is substantially equivalent to that for section I. Pipe-length 26 for this treatment is rather great, as indicated by its serpentine shape in the figures. It is, however, preferably in a generally horizontal plane over most of its length. Due to its length and to the consequently large friction losses, a high-capacity pump 21 is ordinarily preferred to an eductor. However, a large liquid/liquid eductor can be employed alternatively.

Fibers 8 conducted from section I via baffle 20 are submerged at liquid level 3 by flow of booster liquid from pipes 28 and 28', this flow-rate being controlled by flow-rate sensor 209 in combination with automatic valve 210. The fiber-slurry is drawn down the vertical submerging pipe 25 and into pipe-length 26 by slurry pump 21. As in section I, a regulator 23 is provided from which submerging liquid is withdrawn by circulating pump 22 and from which further liquid can be recycled through valve 207. The speed of pump 22, and thereby its capacity, is controlled by pressure-sensor 108. Treated fibers 8 float up through coupling tank 24 to liquid level 4 where squeeze-

rolls 27 withdraw fibers 8 and forward them to section III via baffle 30. Seals 30' prevent passage of liquids. Booster liquid is recycled from tank 24 to pump 21 via pipe 213 at a rate detected by flow-rate sensor 211 and controlled by automatic valve 212.

A preferred liquid for a boosting section from which water is completely excluded in the azeotropic mixture of methylene chloride and perfluorocyclobutane. As long as at least 9.1% by weight of the mixture is perfluorocyclobutane, it boils at -6° C. Two equilibrium liquid phases develop, a methylene chloride-rich phase comprising about 9.1% by weight of perfluorocyclobutane and a perfluorocyclobutane-rich phase comprising about 88% by weight of perfluorocyclobutane. Since the lighter, less expensive methylene chloride-rich phase provides a thermodynamic activity for perfluorocyclobutane identical to that of the other phase, the lighter phase is preferably circulated through pipe-length 26 at about -6° C. If water is present in the boosting section, for example, because of inadequate sealing between the boosting section and the downstream aqueous stripping section, boiling of the azeotropic mixture at -6° C. leads to ice formation. Accordingly, when water is present, it is preferred to operate with a boosting liquid comprising a 1 to 5% solution of perfluorocyclobutane in methylene chloride at a temperature between about 1 and 25° C. The details surrounding coupling tank 24 in FIGS. 1 and 3 reflect the use of the azeotropic mixture as booster fluid. Liquid-level 4, the top of the lighter phase, is detected by liquid-level sensor 203 which controls automatic valve 204 for the input of methylene chloride or booster fluid at 201. Liquid-level 5, the top of the denser phase, is detected by liquid-level sensor 205 which controls automatic valve 206 for the input of perfluorocyclobutane-rich liquid 202. Pipe 213 extends into lighter phase, and screen-enclosed channel 29 has an impermeable section 29' extending to about level 5. In addition to a perfluorocyclobutane-rich stream at 202, it is also desirable ordinarily to inject some at 216 in order to prevent exhaustion of perfluorocyclobutane in pipe-length 26.

As in section I, all exposed surfaces of apparatus should be thermally insulated. Cooling by heat transfer to a cooling medium, e.g., refrigerated brine, must be provided at one or more points. A jacket type heat-exchanger 215 in the circulation loop is particularly desirable because the fibers entering through baffle 20 are hot and carry superficial hot methylene chloride.

A booster liquid comprising methylene chloride and chloropentafluoroethane is similarly preferred when no water is present in the boosting section. It, too, is preferably cooled to about -6° C., but it does not form an azeotropic mixture. If it is used, controls 205 and 206 are replaced with concentration-detection devices for controlling input of chloropentafluoroethane at 202 and at 216.

STRIPPING SECTION

FIGS. 1 and 4 depict the preferred stripping section, indicated by Roman numeral III. The purposes of the stripping section are to remove plasticizing and excess boosting liquids, deplasticizing the cell walls and trap the infantant within the closed cells. Operation and construction of this section are substantially as described for section I except that hot water is the preferred stripping liquid and that a manifold 311 for removing released vapors is provided.

Staple fibers 8 arriving via baffle 30 are submerged below water level 6 by flow of water from pipes 38 and 38'. Vapors (generated by the plasticizing and boosting liquids from the preceding pipeline sections) above water level 6 are removed and sent to solvent recovery via line 214 (FIG. 1). Flow-rate sensor 208 detects water flow-rate and controls it through automatic valve 309. Water flow is induced by circulating pump 32, which discharges most of the water via valve 307 to liquid/

liquid eductor 31. Water enters pump 32 via pipe 312 from coupling tank 34 and via valve 306 from regulator 33. Flow-rate sensors 304 and 305 control automatic valve 306. Coupling tank 34 has water-level 7 which is maintained by sensor 302 and automatic valve 303. As before, screen-enclosed channel 39 leads the fibers 8 up to the nip with rollers 37. Heaters 315 are provided at one or more points in the stripping section to maintain the water temperature.

Regulator 33 differs from the regulators 13 and 23 in that it encloses most of pipe-length 36 and provides not only for liquid-flow regulation but also for the removal of vaporized plasticizing liquid and impermeant infantant. Bubbles of this vapor rise through stand-pipes of manifold 311, above liquid levels 310, and on to solvent recovery means (not shown) as indicated by arrow 301. The efficient removal of vapors through pipe-length 36 within regulator 33 requires a high degree of openness in the pipe-wall. Well-screen piping is preferred for that section of pipe-length 36 within regulator 33. Baffles 313 are provided between the well-screen piping and the wall of regulator 33 to prevent the liquid from passing through the well-screen and by-passing the fibers, which would result in the fibers being held up in pipe-length 36. If desired, a pipe connecting the last section of regulator 33 with return pipe 312 can be provided to permit withdrawal of some clear liquid and to diminish the quantity of liquid being separated from the fibers through screen 39 in coupling tank 40. A hand operated valve in this pipe provides adequate control.

Pipes 18', 28', and 38' are positioned so as to introduce liquid along the inner wall of the upper conical portion of the respective vertical submerging pipes 15, 25 and 35 in a manner which will impart a downward spiraling motion to the liquid. The purpose is to centrifuge out gas bubbles. The gas bubbles and fibers are of similar densities. Thus, velocity sufficient to submerge the fibers will also submerge the gas bubbles. However, with the swirling motion of the liquid, the fibers, due to their length, are pulled down the sides of the vertical pipes, whereas the gas bubbles are centrifuged to the center and escape. A portion of the liquid in each section is introduced through pipe 18, 28 or 38 and permitted to flow down as a centrally directed stream, preferably a spray. This is particularly important in the plasticization section (FIG. 2) where dry staple is to be submerged.

Following stripping, fibers 8 are dried in air. For polyethylene terephthalate fibers, air temperatures from 70° C. to 175° C. are found effective. Heated air is passed at high velocity across the outlet from rollers 37, as indicated by arrow 41. Some purge of vapor-laden air via 42 is provided. Retention of the fibers in hold-up tank 40 at 70° C. to 175° C. (preferably about 125° C.) for 1 to 30 minutes, varying inversely with the air temperature employed, causes air reinflation of the fibers. It is apparent that continuous conveyance of the fibers through an air oven can replace hold-up tank 40. It is unnecessary to complete the inflation of dried fibers at this point, and subsequent re-exposure to heated air to establish approximate osmotic equilibrium for air is often desirable.

OPERATING CONDITIONS AND DIMENSIONS

In general, the process and apparatus of this invention are applicable to a wide range of flow rates for the fibers. The important variables for the flow in each pipe-length can be expressed by the equation

$$v = \frac{L}{T} = \frac{W}{A} \frac{1}{S} \quad (1)$$

where

L is the length of the pipe,

T is the desired time of exposure to each liquid,

W is the flow rate for fibers expressed as weight of fibers per unit of time,

A is cross-sectional area of the pipe,

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d is density of the staple fibers,
 S is the fraction of the total volume of pipe actually filled by flowing fibers, and
 v is linear velocity of fibers in the pipe.

The units for variables used in Equation 1 must, of course, be selected from one consistent system of units. For the postinflation of the preferred polyethylene terephthalate ultramicrocellular staple, flow-rates (W) in the range of from about 100 to about 2500 pounds of polymer per hour (about 45 to about 1100 kg. of polymer per hour) are of particular interest. Density (d) of the staple during treatment is determined largely by the temperature of the treatment liquid. Thus, in the heated plasticizing section (I) and stripping section (III), the preferred staple has a density (d) of the order of 1 lb./ft.³ (0.02 gm./cc.); but in the cold boosting section (II), density (d) is of the order of 3.0 to 4.5 lb./ft.³ (0.048 to 0.072 gm./cc.). Treatment times (T) vary from section to section. In the plasticizing section (I), residence times (T) of from 10 to 25 seconds are effective. In the boosting section (II), from 1 to 30 minutes is the range in which sufficient impermeant inflatant is introduced within the closed cells. Adequate removal of excess solvents in stripping section (III) occurs ordinarily in treatment times of from 15 to 35 seconds. Volume percentages ($100 \cdot S$) of the fibers in the pipes are ordinarily from 2 to 20% to provide slug flow while avoiding excessive frictional resistance to flow. With a concentration of about 2% or greater, the fibers become entangled and form coherent slugs (or clumps) almost immediately upon introduction into a pipe-length. Even with lower concentrations, say $\frac{1}{2}$ volume percent or less, the fibers collect to form clumps within a short time.

Thus, the only variables of Equation 1 which remain unspecified are L , A , and v . Although fiber-velocity (v) can vary over quite wide limits, values of from 1.0 to 2.0 ft./sec. are ordinarily preferred. From a knowledge of these variables, Equation 1 can be solved for suitable values of pipe-length (L) and cross-sectional area (A) of the pipe. Actually, an infinite set of A - L values results, but the range is narrowed by the desirability that L be much greater than the transverse cross-sectional size of the pipe. Also, cross-sectional areas (A) equal to those of circles with diameters from about 4 to 24 inches (10 to 60 cm.) are found most suitable, increasing with increasing flow rate (W).

EXAMPLE I

Preferred ultramicrocellular fibers as hereinabove defined are continuously prepared by extrusion at a rate of about 100 lb./hr. (45 kg./hr.) of polymer through 24 extrusion orifices. The uniform foamable composition is composed of about 65% polyethylene terephthalate and 35% methylene chloride (both percentages by weight). Temperature and pressure of this solution just prior to passage through the extrusion orifices are about 215° C. and about 650 p.s.i.g. (46 kg./cm.² gauge), respectively. Each extrusion orifice is a cylindrical hole about 0.012 inch (0.30 mm.) in diameter and about 0.006 inch (0.15 mm.) long. If the extruded fibers are collected directly in ambient air, the fibers collapse to a density of the order of 0.1 gm./cc. or higher within seconds, due both to condensation of methylene chloride within the cells and to its rapid escape by diffusion. Thereafter, the fibers are not spontaneously reinflatable and remain only slightly pneumatic.

In this example, the fibers are extruded into methylene chloride vapor in equilibrium with the boiling liquid (41° C.). Collapse is thereby prevented, the expanded fibers are cut to staple in this atmosphere, and the staple is discharged at about 100 lb./hr. (45 kg./hr.) to collection vessel 10 where they are immersed in boiling methylene chloride (41° C.) in submerging section 15 of pipe 16. Submerging pipe-length 15, leading to eductor 11, is 4.0 inches (10.2 cm.) in inside diameter, and the re-

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mainder of pipe-length 16 is 6.0 inches (15.2 cm.) in inside diameter. Fiber velocity in pipe-length 16 is about 2.0 ft./sec. (0.61 m./sec.), and total residence time in boiling methylene chloride is about 15 seconds. The corresponding liquid velocity is about 2.7 ft./sec. (0.82 m./sec.). Sufficient liquid methylene chloride flows via pipe 18 to provide a liquid velocity of about 3 ft./sec. (0.91 m./sec.) in the 4.0 inch (10.2 cm.) diameter pipe. Eductor 11 and circulating pump 12 are selected to provide these velocities as well as a range of variability as hereinabove specified. Screen-enclosed channel 19 is square with about 8.0 inch (20 cm.) sides. Squeeze-rolls 17 have compressible elastomeric surfaces, are 6.0 inches (15.2 cm.) in diameter, and form a nip about 0.5 inch (1.3 cm.) above liquid level 2.

Booster section (II) is operated as hereinbefore detailed. Again, the vertical submerging section is 4.0 inches (10.2 cm.) in inside diameter, and the remainder (nearly all) of pipe-length 26 is 6.0 inches (15.2 cm.) in inside diameter. The liquid circulated is composed of about 9 parts by weight of perfluorocyclobutane and 91 parts of methylene chloride and is maintained at about -6° C. The staple resides in this liquid about 15 minutes. In pipe-length 26, staple velocity is about 1.0 ft./sec. (0.30 m./sec.), and liquid velocity is about 1.3 ft./sec. (0.40 m./sec.). Liquid is introduced via pipe 28 sufficient to provide a liquid velocity in submerging section 25 of about 5.0 ft./sec. (1.52 m./sec.). Sizes of screen-enclosed channel 29 and squeeze rolls 27 are identical to those in section I.

In section III, the stripping fluid is water maintained at about 70° C. Submerging pipe-length 35 is 6.0 inches (15.2 cm.) in diameter, but pipe-length 36 following eductor 31 is oval in cross-section with 4 and 12 inch (10.16 and 30.48 cm.) axes, the larger axis in the horizontal plane. Most of pipe-length 36 is well-known well-screen piping to allow escape of liberated gases to solvent recovery. Treatment time in water is about 30 seconds at a fiber-velocity in the oval pipe of 1.0 ft./sec. (0.31 m./sec.) and a water-velocity of about 2.3 ft./sec. (0.70 m./sec.). Water is injected via pipe 38 to provide a water-velocity in the submerging section of about 3.0 ft./sec. (0.91 m./sec.). Squeeze-rolls 37 are as previously described, but screen-enclosed channel 39 is increased to an 8 x 16 inch (20.3 x 40.6 cm.) rectangle in order to enclose the exit of the oval pipe.

The staple is removed from the stripping section (III) in a stream of hot air and is held in air at about 125° C. for about 20 minutes. Thereafter, it becomes fully inflated, round in cross-section, and highly pneumatic.

In a particularly preferred embodiment of the apparatus of this invention, the inner walls of the pipe-lengths are coated with a low-friction coating, e.g., polytetrafluoroethylene. This is particularly advantageous for very long pipe-lengths such as are characteristic of the boosting section. Pressure drop which occurs on pumping closed-cell, foamed, staple fibers through such a coated pipe-length is reduced to only one-quarter to one-half that required in an identical uncoated pipe-length. In the pumping of customary heavy slurries, e.g., sand, little or no pressure drop from interaction of the slurried solids with pipe-walls has been observed. Low-density foamed fibers, however, possess high surface friction, are urged by buoyancy against pipe-walls, and transmit frictional resistance-to-flow at the walls by fiber-entanglement to the whole flowing slug of fibers. In order to maintain a given flow rate of fibers, the liquid must be pumped correspondingly faster to overcome the friction at the pipe-walls. Thus, low-friction coating of the inner walls of a pipe-length greatly decreases the power required to maintain a given fiber-flow velocity.

EXAMPLE II

In this example, microcellular staple fibers of polyethylene terephthalate of the same type as used in Example I are postinflated in substantially the same equipment as in

Example I and under the same conditions, with the following exceptions:

	Section of equipment		
	Plasticizing	Boosting	Stripping
Fluid.....	Methylene chloride.	3.1% perfluorocyclobutane in methylene chloride.	Water.
Temperature.....	28° C.	4° C.	70° C.
Velocities, ft./sec.:			
Submergence.....	1.0.....	1.0.....	2.5.
Liquid in pipe.....	2.1.....	1.0.....	4.0.
Fiber in pipe.....	1.8.....	0.7.....	2.0.
Residence time.....	20 sec.....	4.5 min.....	20 sec.

The fibers, which are treated in the above-described manner at a rate of 10 pounds per hour, are then heated in air at 125° C. for 20 minutes. The fibers are then cooled.

EXAMPLE III

This example illustrates the use of the process and apparatus of this invention in treating buoyant staple fibers of closed-cell foamed organic polymer with a boosting fluid consisting of a very dilute concentration of plasticizer and impermeant inflatant in water to render the fibers postinflatable.

Ultramicrocellular staple fibers, spun and cut in accordance with Example I, are collected in bags and then hand fed at a rate of about 20 pounds per hour to collection vessel 10 of FIG. 1, where they are immersed in water at about 18° C., submerged in submerging section 15, forwarded to eductor 11 and then passed through pipe-length 16 where the fibers travel at a velocity of about 2.6 to 2.7 ft./sec. and the water travels at about 3.2 ft./sec. At the end of pipe-length 16, the fibers and water flow vertically into a fiber-liquid separating device. This latter device has a horizontal open-mesh screen onto which the fibers and water are dropped. The water passes through the screen and is recycled. The fibers are forwarded on the screen through squeeze rolls and then are doffed from the screen by rapidly rotating brushes which advance the fibers to the vertical submerging portion of Section II. Note that in this example, Section I is filled with water and is used for distributing and feeding fibers to booster section II (of FIG. 1) and for preventing loss of organic vapors from section II. No plasticizing occurs.

In booster section II of FIG. 1, the treatment (i.e., "boosting") liquid comprises at least about 97 weight percent water and small amounts of plasticizer and impermeant inflatant. The liquid has two phases, a solution phase and a dispersed phase. The solution phase contains about 2 weight percent methylene chloride plasticizer and very small amounts of perfluorocyclobutane impermeant inflatant. The dispersed phase contains 96 weight percent methylene chloride and 4 weight percent perfluorocyclobutane. At the inlet to booster section II, the treatment liquid is 99 weight percent solution and 1 weight percent dispersed phase. At the outlet to booster section II, the concentration of the dispersed phase is reduced to 0.65 weight percent, due to the entry of the plasticizer and impermeant inflatant into the cells of the staple fibers. The temperature of the treatment liquid in the boosting section is maintained at 8° C. In 900-foot long, 6-inch Schedule 10 pipe-length 26, the fiber velocity is about 1.0 ft./sec., and the boosting liquid velocity is about 1.5 ft./sec. The fibers, which pass through the boosting section entangled in discrete clumps (i.e., in slug flow), have a residence time in boosting section II of about 15 minutes. The liquid velocity in submerging section 25 is about 1.5 ft./sec. Fibers are separated from the boosting liquid at the end of the boosting section by a device which consists of a rotating open mesh cylindrical screen on which the fibers are dropped and through which the liquid passes for recirculation. After passage through a squeeze-roll nip, the fibers are doffed from the screen by means

of rapidly rotating brushes and advanced to the vertical submergence pipe-length 35 of stripping section III.

In section III, the treatment liquid (i.e., stripping liquid) is water maintained at about 63° C. The fibers are submerged in 6-inch, Schedule 10-pipe-length 35 and forwarded by eductor 31 to pipe-length 36 which in this Example is a 8-inch, Schedule 10 pipe, the upper 120 degrees of which is well-screen piping to allow escape of perfluorocyclobutane and methylene chloride vapors to solvent recovery. Treatment time in the stripping section is about 15 seconds. The fibers flow through the pipe entangled in discrete clumps at a velocity of about 2 ft./sec. The water velocity is about 4.7 ft./sec. Liquid velocity in the submergence section at the entrance to the stripping section is about 2.5 ft./sec. A fiber-liquid separator of the type coupled to Section I removes the fibers from the stripping section. At this point at least about 8 grams of impermeant inflatant (i.e., perfluorocyclobutane with essentially no methylene chloride) per 100 grams of fiber polymer are contained within the closed cells of the fibers.

The fibers removed from the stripping section are then forwarded to a bin through which hot air at 80-125° C. is passed for between 20 and 60 minutes. The fibers are then cooled.

As a result of the treatment given the fibers in this example, the fibers become fully inflated, turgid, round in cross-section and highly pneumatic.

While dimension, controls, conditions, etc., given hereinabove are specific to two preferred systems, the description is believed adequately detailed to enable one skilled in the art to readily adapt the invention to other systems and other applications. As many alternatives and alternations are apparent without departing from the scope of this invention, it is not intended to be limited by the foregoing details.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. In the substantially atmospheric-pressure process for treating buoyant, closed-cell, foamed, microcellular staple fibers prepared from synthetic crystallizable organic polymers to render them postinflatable upon exposure to air, which process comprises contacting the fibers with treating liquids, said treating liquids comprising either (a) a plasticizing liquid that swells the cell walls of the fiber but is essentially a nonsolvent for the polymer of the fiber, liquid containing an impermeant inflatant that is sulfur hexafluoride or a saturated aliphatic or cycloaliphatic compound having at least one fluorine-to-carbon covalent bond and wherein the number of fluorine atoms exceeds the number of carbon atoms, and stripping liquid that is a nonsolvent for the fibers and nonreactive with the impermeant inflatant and the plasticizing liquid, employed in sequence, or (b) said plasticizing liquid and said liquid containing impermeant inflatant employed simultaneously, followed by said stripping liquid, the improvement which comprises:

submerging the fibers sequentially in each afore-described treating liquid and transporting said fibers with said liquid or mixture thereof in slug flow through a pipeline at a rate of about 0.5 to about 5 feet per second and at a fiber volume such that the fibers occupy from one to twenty percent of any given cross-section of the pipeline.

2. The process improvement of claim 1 wherein the fibers are submerged and moved through one pipeline with the plasticizing liquid, separated from the liquid, submerged and moved through a second pipeline with the liquid containing an impermeant inflatant, separated from said liquid, and submerged and moved through another pipeline with the stripping liquid.

3. The process improvement of claim 1 wherein the fibers are submerged and moved through one pipeline with a mixture of the plasticizing liquid and the impermeant inflatant, separated from the mixture, and submerged and

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moved through another pipeline with the stripping liquid.
 4. The process improvement of claim 2 wherein the fibers are prepared from polyethylene terephthalate, the plasticizing liquid is methylene chloride, the impermeant inflatant is selected from the class consisting of perfluorocyclobutane and chloropentafluoroethane, and the stripping liquid is water.

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