

1

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METHOD FOR DENSIFYING NITROCELLULOSE
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This invention relates to the treatment of nitrocellulose and, more particularly, to the colloidizing of nitrocellulose to produce fully colloidized particles or objects thereof. In a specific aspect, the invention relates to the treatment of nitrocellulose having a nitrogen content from about 10.7% to about 11.2% by weight to change the physical form of such nitrocellulose from its customary fibrous form into solid, nonporous, fully colloidized particles or objects thereof.

Nitrocellulose having a nitrogen content between about 10.7% and about 11.2% finds application in specialized nitrocellulose lacquers, adhesives and the like, in formulating nitrocellulose plastics, and in formulating black powder substitutes as well as other explosive and propellant compositions. Such nitrocellulose is produced and sold today in fibrous form. The bulk density of the product is low, causing relatively high shipping costs. This disadvantage is partially overcome by compressing the nitrocellulose with a ram to increase barrel loadings. As a result of compression in packing, nitrocellulose users find the fibrous material quite difficult to unload from the barrels. An additional disadvantage of conventional commercial fibrous nitrocellulose is the tendency to agglomerate and form large, slow-dissolving lumps when added to solvents in certain types of dissolving equipment.

Therefore, the primary object of this invention is to produce an improved form of nitrocellulose which can be transported more economically, unloaded from barrels more easily, and dissolved more rapidly in solvents.

A further object of the invention is to provide a new and improved method for converting fibrous nitrocellulose having a nitrogen content from about 10.7% to about 11.2% into solid, nonporous, fully colloidized particles or objects thereof.

Other objects will become apparent from the following description of the invention, the novel features and combinations being set forth in the appended claims.

Generally described, these objectives and others are accomplished in accordance with this invention by dissolving nitrocellulose having from about 10.7% to about 11.2% nitrogen content by weight in glycol having the formula



wherein R is an aliphatic hydrocarbon radical having from 2 to 6 carbon atoms, preforming the resulting solution, introducing the preformed solution into a water bath containing not more than about 90% by weight of said glycol at the point of original contact of said solution with said water bath to gel the nitrocellulose, leaching the gelled nitrocellulose in the presence of water until a predetermined amount of the glycol originally present in the gelled nitrocellulose has been replaced by water, and subjecting the resulting leached nitrocellulose to drying conditions to remove water and produce colloidized nitrocellulose.

In the above-described process, the surface of the body of preformed solution of nitrocellulose in glycol upon contact with the water bath is gelled at the instant of contact and a tough envelope of gelled nitrocellulose

2

composition is formed about the exterior of the body. For convenience, this phenomenon is termed "shock-gelling." This envelope of shock-gelled nitrocellulose composition is of sufficient toughness that the body retains its preformed shape, and is also permeable to the passage of water into the body and the passage of glycol out of the body. Accordingly, as the shock-gelled body is retained in the water bath or is otherwise further contacted with the aqueous medium, water leaches the glycol from the shock-gelled body and water replaces the glycol in the body of composition until an equilibrium condition is approached at which the concentration of glycol is substantially the same in the particle or body as in the surrounding leaching bath. Obviously, of course, gelation progresses inwardly within the shock-gelled particle or body as water permeates thereto until the entire particle or body is in a gelled state. Thus, a solution of nitrocellulose in glycol will be gelled when introduced into the water bath, and the amount of glycol removed from the gelled composition by leaching in the water bath will depend upon the concentration of glycol in the water bath and the time of contact. If the leaching process is conducted stepwise and the gelled material is successively introduced into fresh leaching baths, the point will be reached at which substantially all of the glycol will be removed from the gelled material. Generally, however, it is more convenient and economical to leach the glycol from the gelled material by continuous countercurrent extraction.

It has been found that very little shrinkage of the gelled material occurs during the leaching process, even when water has replaced substantially all of the glycol in the gelled material. Consequently, the gelled material at conclusion of the leaching process is still a swollen, highly attenuated gel, somewhat rubbery and elastic in behavior. For convenience, the gelled material upon completion of leaching, but prior to drying, is termed "water-logged." However, upon subjecting to drying conditions, the water-logged material shrinks uniformly as the water is removed in the drying operation to final dimensions determined by the original concentration of nitrocellulose in the glycol solution. In the final dry condition the nitrocellulose material is in the form of hard, tough, nonporous, fully colloidized particles or objects which retain substantially the physical shape of the shock-gelled bodies of material subjected to leaching.

Having generally described the invention, the following examples set forth some specific embodiments of the invention. It is to be understood, however, that these examples, while illustrative, are not to be construed as a limitation of the invention.

Example 1

Five parts by weight of dry fibrous nitrocellulose having a loose bulk density of about 12 lb./cu. ft. and a barrel packed density of about 23 lb./cu. ft., a nitrogen content of 11.0% by weight and a standard ASTM falling ball viscosity characteristic of 13 seconds were dissolved in 95 parts by weight of propylene glycol (1,2-propanediol). This solution was poured into a metal container, the bottom of which contained perforations about 0.09 inch in diameter. The droplets of solution emitted from these holes were allowed to fall about 18 inches through air before striking the surface of a water bath. During this fall the droplets became essentially spherical. The surface of the droplets gelled instantly upon contact with the water bath and were sufficiently tough to withstand passage through the water surface without substantial deformation. The shock-gelled balls thus produced were leached in running water for a period of 2 hours, at which time they were about 0.15 inch in diameter. The water-logged balls were removed from the water and dried over-

night at a temperature of 60° C. The resulting dried nitrocellulose was in the form of solid, nonporous, fully colloided, spheroidal balls approximately 0.05 inch in diameter having a bulk density of about 60–65 lb./cu. ft. The resulting densified nitrocellulose was a free-flowing product which readily poured out of shipping containers without necessity for forking required with commercial fibrous nitrocellulose and which dissolved rapidly in solvents without forming large slow-dissolving lumps.

Example 2

Five parts by weight of the same fibrous nitrocellulose employed in Example 1 were dissolved in 95 parts by weight of 1,5-pentanediol, and the resulting solution was shock-gelled, leached, and the water-logged particles were dried substantially as set forth in Example 1. The resulting densified nitrocellulose product was substantially the same as the product prepared in Example 1.

Examples 3–5

Dry fibrous nitrocellulose, having a nitrogen content of 10.9% by weight and a standard ASTM falling ball viscosity characteristic of about 13.5 seconds, was employed to prepare the following solutions in various glycols.

| Ex. No. | Solvent | Percent by Weight | Weight of Nitrocellulose Dissolved, Percent |
|---------|----------------------|-------------------|---|
| 3----- | 1,2-Propanediol----- | 95.3 | 4.7 |
| 4----- | 1,3-Propanediol----- | 95.0 | 5.0 |
| 5----- | 1,5-Pentanediol----- | 99.0 | 1.0 |

The above solutions were shock-gelled, leached, and the water-logged particles were dried substantially as set forth in Example 1. The resulting dried nitrocellulose in all cases was a free-flowing, fast-dissolving and high bulk density product in the form of solid, nonporous, fully-colloided, tiny spheroids.

Examples 6–13

Dry fibrous nitrocellulose, having a nitrogen content of 11.0% by weight and a standard ASTM falling ball viscosity characteristic of ¼ second, was employed to prepare the following solutions in various glycols.

| Ex. No. | Solvent | Percent by Weight | Weight of Nitrocellulose Dissolved, Percent |
|---------|-------------------------------|-------------------|---|
| 6----- | Ethylene glycol----- | 95.3 | 4.7 |
| 7----- | 1,2-Propanediol----- | 85.0 | 15.0 |
| 8----- | 1,3-Propanediol----- | 95.0 | 5.0 |
| 9----- | 1,3-Butanediol----- | 98.6 | 1.4 |
| 10----- | 1,4-Butanediol----- | 98.5 | 1.5 |
| 11----- | 1,5-Pentanediol----- | 90.0 | 10.0 |
| 12----- | 2-Methyl-2,4-pentanediol----- | 95.0 | 5.0 |
| 13----- | 2,5-Hexanediol----- | 99.7 | 0.3 |

The above solutions were shock-gelled, leached and the water-logged particles were dried substantially as set forth in Example 1. The resulting dried nitrocellulose in all cases was a free-flowing, fast-dissolving, and high bulk density product in the form of solid, nonporous, fully colloided, tiny spheroids.

Examples 14–16

Dry fibrous nitrocellulose, having a nitrogen content of 11.06% by weight and a standard ASTM falling ball

viscosity characteristic of 5 seconds, was employed to prepare the following solutions in various glycols.

| Ex. No. | Solvent | Percent by Weight | Weight of Nitrocellulose Dissolved, Percent |
|---------|----------------------|-------------------|---|
| 14----- | 1,2-Propanediol----- | 95.3 | 4.7 |
| 15----- | 1,3-Propanediol----- | 95.0 | 5.0 |
| 16----- | 1,5-Pentanediol----- | 99.0 | 1.0 |

The above solutions were shock-gelled, leached and the water-logged particles were dried substantially as set forth in Example 1. The resulting dried nitrocellulose in all cases was a free-flowing, fast-dissolving, and high bulk density product in the form of solid, nonporous, fully colloided, tiny spheroids.

Examples 17–22

Dry fibrous nitrocellulose, having a nitrogen content of 11.1% by weight and a standard ASTM falling ball viscosity characteristic of 13 seconds, was employed to prepare the following solutions in various glycols.

| Ex. No. | Solvent | Percent by Weight | Weight of Nitrocellulose Dissolved, Percent |
|---------|-------------------------------|-------------------|---|
| 17----- | Ethylene glycol----- | 98.3 | 1.7 |
| 18----- | 1,2-Propanediol----- | 91.0 | 9.0 |
| 19----- | 1,3-Propanediol----- | 95.0 | 5.0 |
| 20----- | 1,3-Butanediol----- | 99.8 | 0.2 |
| 21----- | 1,5-Pentanediol----- | 95.3 | 4.7 |
| 22----- | 2-Methyl-2,4-pentanediol----- | 95.0 | 5.0 |

The above solutions were shock-gelled, leached and the water-logged particles were dried substantially as set forth in Example 1. The resulting dried nitrocellulose in all cases was a free-flowing, fast-dissolving and high bulk density product in the form of solid, nonporous, fully colloided, tiny spheroids.

Examples 23–25

Dry fibrous nitrocellulose, having a nitrogen content of 11.0% by weight and a standard ASTM falling ball viscosity characteristic of 85 seconds, was employed to prepare the following solutions in various glycols.

| Ex. No. | Solvent | Percent by Weight | Weight of Nitrocellulose Dissolved, Percent |
|---------|----------------------|-------------------|---|
| 23----- | 1,2-Propanediol----- | 95.3 | 4.7 |
| 24----- | 1,3-Propanediol----- | 95.0 | 5.0 |
| 25----- | 1,5-Pentanediol----- | 95.0 | 5.0 |

The above solutions were shock-gelled, leached and the water-logged particles were dried substantially as set forth in Example 1. The resulting dried nitrocellulose in all cases was a free-flowing, fast-dissolving and high bulk density product in the form of solid, nonporous, fully colloided, tiny spheroids.

Example 26

Five parts by weight of dry fibrous nitrocellulose, having a nitrogen content of 11.2% by weight and a standard ASTM falling ball viscosity characteristic of 5 seconds, was dissolved in 95 parts by weight of 1,2-propanediol, and the resulting solution was shock-gelled, leached, and the water-logged particles were dried substantially as set forth in Example 1. The resulting densified nitrocellulose product was similar in physical form and properties to the product produced in Example 1.

Example 27

Five parts by weight of dry fibrous nitrocellulose, having a nitrogen content of 10.7% by weight and a standard ASTM falling ball viscosity characteristic of 4 seconds, was dissolved in 95 parts by weight of 2-butene-1,4 diol, and the resulting solution was shock-gelled, leached, and the water-logged particles were dried substantially as set forth in Example 1. The resulting densified nitrocellulose product was similar in physical form and properties to the product produced in Example 1.

Example 28

Ten parts by weight of dry fibrous nitrocellulose, having a nitrogen content of 11.1% by weight and a standard ASTM falling ball viscosity characteristic of 5 seconds, and 0.1 part by weight of 2-nitrodiphenylamine were dissolved in 89.9 parts by weight of 1,2-propylene glycol. The resulting solution was extruded through a 0.19 inch diameter opening using a pressure of 3 p.s.i. into a water bath to form a continuous coil of shock-gelled nitrocellulose about 0.23 inch in diameter, which was cut below the water level in the bath into strands about 6 inches in length. The strands were leached and the water-logged strands were dried substantially as set forth in Example 1. The resulting nitrocellulose product was a hard, high density, fully colloided material, each strand being about 0.075 inch in diameter and 3 inches in length.

This invention is specific both with respect to the nitrogen content of the nitrocellulose employed and with respect to the glycols employed to prepare the nitrocellulose solutions.

Nitrocelluloses in accordance with this invention have nitrogen contents between about 10.7% and about 11.2% by weight. Nitrocelluloses having nitrogen contents of about 10.6% or lower and about 11.3% or higher by weight are insoluble in the glycols of this invention and are, therefore, inoperative in accordance with this invention. It is to be understood, however, that the above limitation of about 10.7% and about 11.2% nitrogen content permits leeway of a few hundredths of a percent on both the low side and the high side of the defined operative range. There is no limitation with respect to the viscosity characteristic of the nitrocellulose employed, and all commercially available viscosity types of nitrocellulose, ranging in viscosities from 10 cps. to 377,000 cps. (1000-second type) in 12.2% solution in a solvent composed of 20% ethyl acetate, 25% ethyl alcohol, and 55% benzene, are densified in accordance with this invention.

All glycols having the formula



wherein R is an aliphatic hydrocarbon radical having from 2 to 6 carbon atoms are operative for the purposes of this invention. This includes all straight-chain, branched-chain and unsaturated aliphatic hydrocarbon radicals having 2 to 6 carbon atoms. Glycols wherein R is an aliphatic hydrocarbon radical having 7 or more carbon atoms are such poor solvents for the nitrocelluloses of this invention that they dissolve only vanishingly small amounts of nitrocellulose and are, therefore, not practical to employ. Typical glycols within the scope of this invention include, for example, ethylene glycol; 1,2-propanediol; 1,3-propanediol; 1,2-butanediol; 1,3-butanediol; 1,4-butanediol; 1,5-pentanediol; 1,6-hexanediol; 2-methyl-2,4-pentanediol; 2,5-hexanediol; 2-butene-1,4-diol; 2-pentene-1,5-diol; 3-hexene-1,5-diol; and the like. A mixture of two or more glycols may be employed as nitrocellulose solvent if desired.

On the other hand, glycols which contain an ether-oxygen linkage within the molecule, as for example, di-

ethylene glycol or dipropylene glycol, and the like, are not operative for the purposes of this invention, for solutions of nitrocellulose having about 10.7% to about 11.2% nitrogen in such glycols do not shock-gel in accordance with this invention. Such solutions upon contact with water either do not gel at all or if they do gel, there is formed a fibrous or porous precipitate.

In practicing this invention, it has been found generally that glycols having an odd number of carbon atoms in the hydrocarbon radical are more active solvents for the nitrocelluloses of this invention than glycols having an even number of carbon atoms in the hydrocarbon radical. It has also been observed that introduction of an unsaturation into the molecule greatly enhances nitrocellulose solubility, for example, 2-butene-1,4-diol is a much better solvent than 1,4-butanediol. Similarly, a branched-chain glycol usually has greater solvent power for the nitrocelluloses of this invention than straight-chain glycols of the same number of carbon atoms. Generally, the more active a glycol solvent is for the nitrocelluloses of this invention, the greater is the amount of any given nitrocellulose which can be dissolved therein to produce a solution of any preselected viscosity.

Another factor which has been observed in preparing glycol solutions of the nitrocelluloses of this invention is that such glycol solutions exhibit the phenomenon of thermal gelling to a greater or lesser extent, depending upon the glycol being employed. By thermal gelling is meant that a temperature will be found for any given glycol solution of nitrocellulose, above which the solution will gel. Generally, glycols wherein R is a saturated straight-chain aliphatic hydrocarbon radical having an even number of carbon atoms, such as ethylene glycol, the straight-chain butanediols and the straight-chain hexanediols exhibit this thermal gelling phenomenon to a more pronounced degree than glycols in which R is a branched chain, a straight chain of odd number of carbon atoms, or an unsaturated aliphatic hydrocarbon radical. Because of this thermal gelling phenomenon, it has been found necessary to cool ethylene glycol and other straight-chain glycols to about 0° C. in order to effect solution of the nitrocellulose of this invention therein. In fact, cooling any of the glycols of this invention improves their solvent power for the nitrocelluloses of this invention.

The solution of nitrocellulose in glycol may be performed for introduction into the gelling bath in a number of ways, depending largely upon the size and shape of the particles or objects desired. When very small particles of nitrocellulose composition on the order of 100 microns or less in size are desired, it is most convenient to atomize the solution from a suitable spray nozzle or orifice to form a spray of minute droplets, and the droplets thus formed are allowed to fall into the gelling bath. If desired, the spray droplets may first be allowed to fall through a mist of the gelling medium to initiate gelation. When the glycol solution of nitrocellulose is performed by atomizing from a spray nozzle or orifice, the viscosity of the solution obviously must be adjusted to a level which can be suitably atomized by spraying, as is well recognized by those skilled in the art.

It will be apparent, of course, that the viscosity of the glycol solution of nitrocellulose will depend both upon the viscosity characteristic of the nitrocellulose employed as well as the amount of nitrocellulose which is dissolved in the glycol. It follows, therefore, that for any desired solution viscosity, the amount of nitrocellulose necessary to produce that desired viscosity will decrease as the viscosity characteristic of the nitrocellulose increases. It also follows that with any selected nitrocellulose, the viscosity of the solution will increase with increasing amount of nitrocellulose dissolved therein. The art is fully cognizant of these relationships.

In another embodiment of the invention, the solution of nitrocellulose in glycol is formed into droplets of the

desired size by passage through a perforated plate. In practicing this embodiment, the diameter of the perforations and the viscosity of the glycol solution of nitrocellulose are selected so that the solution upon flowing through the perforations breaks up and forms individual drops instead of flowing in unbroken streams from the orifice. The droplets thus formed are preferably allowed to fall freely until they assume a spherical or spheroidal configuration prior to contacting the gelling bath. Upon striking the surface of the bath, the gelation of the surface of such particles is so rapid that no substantial distortion occurs. By this embodiment small, dense, spheroidal balls of fully colloided nitrocellulose are produced which are larger than can be produced by spraying methods. By proper selection of orifice size and solution concentration and viscosity, production of uniformly sized spheroidal particles can be obtained in any desired size from as small as 0.01 inch diameter or less to 1/4 inch diameter or more. When employing either the spray or freely falling droplet techniques, a nitrocellulose concentration in the glycol solution of between about 1% and about 10% will be employed, preferably between about 2% and about 10%.

Instead of preforming the glycol solution of nitrocellulose into droplets or spray particles, the solution may be introduced into the gelling bath as a continuous stream by gravity or low pressure extrusion. In accordance with this technique a glycol solution of nitrocellulose, preferably characterized by a nitrocellulose concentration of between about 2% and about 20%, is preformed by allowing it to flow through one or more orifices of the desired size in an unbroken stream into the gelling bath. The resulting gelled material will be cord-like and can be leached in this form, after which the water-logged cord can be cut into any granulation desired and dried.

In yet another embodiment of the invention, a glycol solution of nitrocellulose can be produced which contains from about 20% to about 50% or more of nitrocellulose. Such solutions are stiff pastes and must be preformed by pressure extrusion techniques comparable to extrusion techniques employed in the celluloid art or in the solvent process for manufacture of smokeless powder. By this technique the stiff paste-like solution of nitrocellulose is preformed by pressure extrusion through a die of any desired cross-sectional area and configuration, and the extruded shape is introduced into the water bath for gelation and removal of the glycol by leaching. In this technique the pressure-extruded shape may be cut into predetermined lengths prior to introduction into the gelling bath, after gelation, or after leaching, as convenient or desired.

It has also been found that the glycol solutions of nitrocellulose in accordance with this invention can be cast into water-wet molds to shock-gel the castings, after which the surface-gelled castings are leached in a water bath, and the water-logged castings are then dried. Similarly, objects can be coated with the glycol solutions of this invention and such coatings can then be shock-gelled, leached and dried to produce tough, nonporous, fully colloided nitrocellulose coatings thereon.

In all of these techniques the gelled particle, shape or coating shrinks uniformly as the water is removed in the drying operation to final dimensions determined by the original concentration of nitrocellulose in the glycol solution which is shock-gelled and then leached.

From the foregoing description, it will be seen that the concentration of nitrocellulose, in glycol solution is largely a matter of choice and convenience, depending somewhat, of course, on the method of preforming which is employed, and the size and shape of particle or object desired. A controlling factor at the low end of concentration will be the physical strength of the shock-gelled material. In general, concentrations much under 2% will be found to be somewhat tender to handle properly for processing. The upper limit of concentration is deter-

mined largely by viscosity, castability, extrusion pressure, ease of droplet formation and the like. For most practical purposes, the upper limit is about 50% for nitrocelluloses of the lower viscosity characteristics, such as 1/4-second and 1/2-second types, and about 30% for nitrocelluloses of the higher viscosity characteristics, such as plastic type (Herculoid) nitrocellulose.

Shock-gelling may be done using an aqueous bath at room temperature. However, if difficulty is encountered due to thermal gelling of the glycol solutions employed, it will be preferable to cool the gelling bath rather than warm it. Excellent shock gels have been prepared using an ice water bath. The composition of the shock-gelling bath will be either straight water or water containing up to and including about 90% by weight of one or a mixture of the glycols which are operative as nitrocellulose solvents in accordance with this invention.

Conventional additives to nitrocellulose and smokeless powder compositions may be added at various points in the process without detracting from the method as hereinabove set forth. For example, nitrocellulose plasticizers and other additives such as stabilizers and ballistic modifiers for the nitrocellulose may be incorporated into the original glycol solution so that after shock-gelling, leaching and drying a finished, fully colloided nitrocellulose article is obtained in which the plasticizers and/or additives are uniformly incorporated. Similarly, pigments or other insoluble material may be uniformly incorporated into the nitrocellulose composition by adding such materials to the original glycol solution prior to shock-gelling, leaching and drying.

Alternatively, plasticizers and the like may be introduced into the nitrocellulose composition after gelling and extraction of the glycol in the leaching bath by contacting the water-logged nitrocellulose material with an aqueous solution, suspension or emulsion of the plasticizer or additive for a period necessary to obtain the desired degree of absorption. While in the water-logged condition, the nitrocellulose can be saturated with salts, or other solutions and compounds may be deposited in the particles by means of metathetical reactions or by simple evaporation of solution-impregnated material.

After leaching, the water-logged nitrocellulose may be exchanged with other fluids such as nonsolvent alcohols or other water-soluble nonsolvents for nitrocellulose, and by a suitable succession of impregnations the nitrocellulose system can ultimately be made to contain high percentages of hydrocarbons or the like. For example, the water present in the water-logged nitrocellulose may be displaced by exhaustive extraction with isopropyl alcohol, and the alcohol in turn may be displaced by a toluene solution of mineral oil. Upon final drying of the nitrocellulose structure, a tough, rugged, elastic, fully colloided nitrocellulose plastic composition plasticized with mineral oil is obtained which is characterized by surprisingly high strength and impact resistance.

The water-logged nitrocellulose compositions of this invention may be partially dried to a water content of 20% to 30% water for storage and shipment if desired. Alternatively, the water-logged material may be dehydrated for storage and shipment by displacement of the water with a nonsolvent volatile alcohol such as isopropyl alcohol and drying to a volatile content of about 25% to 35% by weight. If desired, the water-logged material may be partly dried to a water content of about 30% by weight and then the residual water may be displaced with isopropanol, or the like.

It is apparent from the foregoing description that this invention provides a novel and improved process for converting fibrous nitrocellulose having a nitrogen content from about 10.7% to about 11.2% into solid, nonporous, fully colloided particles or objects thereof. The improved form of nitrocellulose produced can be transported more economically, unloaded from barrels more easily, and dissolves more rapidly in solvents than commercial

fibrous nitrocellulose since there is no tendency for the densified nitrocellulose of this invention to agglomerate into large, slow-dissolving lumps. The higher bulk density of the nitrocellulose products of this invention results in transportation savings. Commercial fibrous nitrocellulose of 10.7% to 11.2% nitrogen content has a loose bulk density of about 12 lb. (dry basis) per cubic foot and is rammed into barrels to a bulk density of about 23 lb./cu. ft. (dry basis). Fully colloided nitrocellulose in accordance with this invention in the form of tiny, solid, nonporous spheroids averaging 0.05 inch diameter has a bulk density on the order of 60 to 65 lb./cu. ft. (dry basis), thus permitting substantially heavier barrel loadings than with fibrous nitrocellulose. Moreover, the fully colloided nitrocellulose particles of this invention can be poured from the barrels for very easy unloading. Present commercial fibrous nitrocellulose must be dug out of the barrel. The elimination of the costly and labor-consuming pressing or centrifuging and block-breaking operations usually following nitrocellulose dehydration is an important advantage of the fully colloided nitrocellulose particles of this invention. Furthermore, the free-flowing, fast-dissolving and high bulk density nitrocellulose products of this invention can be used in any application where commercial fibrous nitrocellulose of the same nitrogen content is now used, such as lacquers, adhesives, plastics, propellants, and the like.

What I claim and desire to protect by letters Patent is:

1. A process for colloiding nitrocellulose which comprises dissolving nitrocellulose having from about 10.7% to about 11.2% nitrogen content by weight in a solvent consisting of water soluble glycol having the formula



wherein R is an aliphatic hydrocarbon radical having from 2 to 6 carbon atoms, preforming the resulting nitrocellulose solution into shaped bodies, introducing the shaped bodies of preformed nitrocellulose solution into a water bath containing not more than about 90% by weight of said glycol at the point of original contact of said shaped bodies with said water bath to shock-gel said shaped bodies by rapidly forming a tough envelope of gelled nitrocellulose on the surfaces of said bodies so that said bodies retain their preformed shape, leaching the resulting shock-gelled bodies in the presence of additional water until substantially all of the glycol present therein has been replaced by water to form water-logged gels, and subject-

ing the resulting water-logged gels to drying conditions to remove water and produce dry, nonporous, fully colloided nitrocellulose bodies having substantially the same physical shape as the shock gelled bodies subjected to leaching.

2. A process according to claim 1 in which the solution is poured through a perforated plate to form droplets and the droplets thus formed are allowed to fall into the bath.

3. A process according to claim 1 in which the solution is gravity extruded into the bath.

4. A process according to claim 1 in which the solution is preformed by pressing through an orifice, and the preformed solution is introduced into the bath.

5. A process according to claim 1 in which the solution is atomized by spraying, and the atomized spray particles are introduced into the bath.

6. A process according to claim 1 in which said glycol having the formula



is selected from the group of glycols in which R consists of 4-carbon, 5-carbon and 6-carbon unsaturated aliphatic hydrocarbon radicals.

7. A process according to claim 1 in which said glycol having the formula



is selected from the group of glycols in which R consists of 3-carbon and 5-carbon saturated aliphatic hydrocarbon radicals.

8. A process according to claim 1 in which the glycol is 1,2-propanediol.

9. A process according to claim 1 in which the glycol is 1,3-propanediol.

10. A process according to claim 1 in which the glycol is 2-butene-1,4-diol.

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