UNITED STATES PATENT OFFICE

2,011,914

FIBROUS MATERIAL AND PROCESS OF PRODUCING IT

George L. Schwartz, Wilmington, Del., assignor to E. I. du Pont de Nemours & Company, Wilmington, Del., a corporation of Delaware

No Drawing. Application June 29, 1928, Serial No. 289,322

11 Claims. (Cl. 92-21)

My invention relates to a new fibrous material and to a method of preparing said material.

It has been recognized that fibrous materials or felt-like materials can be synthesized by treating fibres with a substance adapted to adhere to the fibres to form a sheeting or mat of said fibrous materials. Such fibrous materials may be formed by beating fibres in the usual beater, together with filling, sizing, or dyeing compounds, as well as particles of solid cellulose esters, and thereafter treating the mixture with some suitable reagent designed to dissolve and deposit the cellulose ester throughout the mass. Another method is to impregnate a mat or felt of fibrous material with cellulose esters in solution, thereafter removing the solvent and leaving the cellulose ester deposited throughout the fibrous These processes invariably produce a fibrous material in which the adhesive material is colloidal and deposited in films throughout the interstices of the fibres. Such materials have low tensile strength and low degree of pliability.

It is an object of my invention to produce an article and establish a process capable of producing an article which has a high degree of pliability. It is a further object of my invention to produce an article which in one of its modifications closely simulates leather. Further objects will appear hereinafter.

30 I have found that if two or more kinds of fibres are mixed together, one of which is capable of being gelatinized or made adhesive, the mixture of fibres may be treated with a gelatinizing or softening reagent, in order to make the gelatinizable or adhesive fibres adhere to the other fibres. When the excess gelatinizing reagent is removed, the adhesive fibres will be in contact with the other, or non-adhesive fibres, and a strong pliable mat or felt of fibrous materials is formed.

By way of example, in carrying out my process I may mix 60 parts of nitrated cellulose fibres with 40 parts of un-nitrated cellulose fibres. The mixture may be beaten for about an hour in the beater without, however, putting down the rolls customarily employed in the usual beaters, since it is my object merely to mix the fibres without cutting or breaking them. A mat or felt of the mixed fibres is then formed and when dried subjected to the vapors of any substance or mixture of substances known to be solvents for nitrated cellulose. The treatment with the solvent vapors is continued until the nitrated cellulose fibres are gelatinized or swollen, so as to be in an adhesive state without, however, dissolving the fibres or causing them to lose their identity as fibres. This capability of the nitrated cellulose fibres, as well as certain other fibres hereinafter referred to, of being rendered adhesive by suitable reagents without solution of the fibres or loss of fibre identity is, for convenience, referred to in the claims as partial gelatinization. A portion of the excess solvent is thereafter removed and the mat or felt rolled at pressures customarily employed in calendering operations, and thereafter dried. The pressing operation employed after the mat was treated with solvent vapors being carried out under such pressures as are customarily employed in calendering operations, does not in any respect modify the dispersed fibres in the mat.

The mat or felt thus produced comprises a mixture of fibres in which the un-nitrated cellulose, or strength giving fibres, are bound together at points of contact with the nitrated cellulose, or adhesive fibres. The interstices of the mat are not filled with gelatinizable material, nor are the individual fibres uniformly coated with adhesive. This structure simulates the structure of natural leather, and gives great resistance to stretching, accompanied by relatively low resistance to collapse, so that if the mat is sharply bent the fibres of one side thereof collapse. This property of low resistance to collapse is generally accompanied by a high degree of pliability.

As a further example of my method, I may mix animal fibres, such as wool waste or tannery hair, with cellulose fibres. After thorough mixing in any suitable manner, such as exemplified above, 20 the cellulose fibres may be treated with a suitable solution of zinc chloride or calcium thiocyanate at somewhat elevated temperature, such as, for example, for zinc chloride 65° C. A dilute solution of sodium hydroxide at low temperatures as may be employed if the animal fibres used are not affected by such caustic solution. The zinc chloride or other solution employed operates to gelatinize the cellulose fibres and make them adhesive, so that they adhere at points of contact 40 with the animal fibres. A mixture of animal fibres and cellulose fibres treated as indicated results in a mat or felt comprised of strengthgiving fibres mixed with adhesive fibres characterized by the open structure referred to in the 45 first example here-above.

Instead of employing animal fibres for the strength giving fibers, I may use cellulose fibres which are relatively ungelatinizable, such as jute, kraft pulp or uncooked cotton linters or rag fibers, and for the adhesive fibres I may use relatively easily gelatinized fibres, such as cooked linters, nitrated linters, or nitrated wood fibres. For gelatinizing reagents, I may use dilute solutions of alkalies varying in concentration from 2 to 11 mols of alkali in 100 mols of water. The gelatinizing may be carried out at moderately low temperatures, as for example, temperatures between -10° C. and $+10^{\circ}$ C., although I do not 60°

wish to be restricted to these specific temperatures.

I have found it convenient to expose a mat formed of mixtures of nitrated cellulose fibres with un-nitrated cellulose fibres to solvent vapors until the nitrocellulose fibres are swollen but not dissolved. It is obvious, however, that other methods of treatment of the fibre mixture may be employed, for example, the gelatinizable fibres may be swollen by immersing the fibre mixture in a solution which contains a small enough content of active solvent to swell, without dissolving and dispersing, the gelatinizable fibres. I have also found it possible to swell the gelatinizable fibres, such as nitrocellulose fibres, with a softener, such as tricresyl phosphate or any of the well-known nitrocellulose softeners. When the fibre mixture or mat is macerated in a solution containing the softeners, the nitrocellulose fibres become swollen and adhesive. The excess solution, before or after the pressing operation previously mentioned, can be washed out of the mat with a liquid such as benzol, which is not a solvent for nitrocellulose, but which is a solvent for the softener.

Though I have described the mixing of the fibres in the usual type of fibre or pulp beater, it is to be understood that my description was for the purposes of exemplification only. My invention may be practiced in different embodiments. For example, as a modification of my process described I have found that the fibres may be mixed by a method I find convenient to call dry deposition. The dry deposition is accomplished by dropping gelatinizable fibres and relatively ungelatinizable fibres from a screen or from a picker onto a moving endless belt, or from a slightly inclined rotating cylinder adapted to thoroughly mix the fibres. The mixed fibres may then be formed into a mat or felt and then treated to swell the adhesive or gelatinizable fibres in any convenient manner. I have found that the fibrous material or artificial leather formed by this method of dry deposition differs from fibrous materials heretofore known in that the fibres, both adhesive and strength-giving, are disposed in heterogeneous or unlaminated planes. By reason of this dispositon of the mixed fibres in heterogeneous or unlaminated planes, a fibrous material is formed which has not only a considerable degree of pliability but remarkable tensile strength as well.

Variations in the ratios of strength-giving or ungelatinizable to adhesive or gelatinizable fibres 55 cause variations in the characters of the fibrous material ultimately formed. A high ratio of gelatinizable or adhesive fibres tends to produce a compact felt with very few or a minimum of air spaces. On the other hand, a low ratio of gelatinizable or adhesive fibres lowers the strength of the felt, and increases the number of air spaces. Similarly, the degree to which the gelatinizable or adhesive fibres are gelatinized or swollen modifies the character of the fibrous material ultimately formed. A high ratio of a gelatinizing reagent or solvent, or a prolonged exposure to the gelatinizing reagent or solvent tends to increase the cementing action of the gelatinizable fibre and consequently increases the stiffness of the felt formed.

As many apparently widely different embodiments of this invention may be made without departing from the spirit thereof, it is to be understood that I do not limit myself to the foregoing

examples or descriptions except as indicated in the following patent claims:

I claim:

1. A process which comprises treating an intimate mixture of gelatinizable and ungelatinizable fibers of paper making length with a solvent for the gelatinizable fibers until they are swollen but are not dissolved or coalesced, and pressing the fibers in sheet form without substantial modification of the fibers therein, and thereby obtaining by said process a flexible fibrous material in which said gelatinizable fibers are adhesively joined at points of contact with the non-gelatinizable fibers.

2. A process which comprises treating an intimate mixture of gelatinizable and ungelatinizable fibers of paper making length with a solvent for the gelatinizable fibers until they are swollen but are not dissolved or coalesced, removing excess solvent, and pressing the fibers in sheet form without substantial modification of the fibers therein, and thereby obtaining by said process a flexible fibrous material in which said gelatinizable fibers are adhesively joined at points of contact with the non-gelatinizable fibers.

3. A process for preparing a fibrous material which comprises intimately mixing gelatinizable and ungelatinizable fibers of paper making length, forming the mixed fibers into a mat, 20 treating the mat with a solvent for the gelatinizable fibers until they are swollen but not dissolved or coalesced, and pressing the mat under a pressure such as is customarily employed in calendering operations.

4. A process for preparing a fibrous material which comprises treating an intimate mixture of nitrocellulose fibers and ungelatinizable fibers of paper making length with the vapors of a volatile solvent which are a solvent for the nitrocellulose fibers only, the treatment being continued until the nitrocellulose fibers are swollen but not dissolved or coalesced, removing the excess solvent by evaporation, and pressing the fibers in sheet form without substantial modification of the fibers therein.

, 5. The process set forth in claim 1 in which the gelatinizable fibers are nitrocellulose.

6. The process set forth in claim 1 in which the gelatinizable fibers are cellulose fibers and the ungelatinizable fibers are animal fibers.

7. The process set forth in claim 1 in which the mixture of fibers consists primarily of relatively gelatinizable and ungelatinizable cellulose fibers.

8. A flexible, pliable, tough artificial leather product consisting of a mat of compressed gelatinizable fibers and ungelatinizable strength giving fibers adhesively joined at points of contact, the adhesive material joining said fibers consisting of the material of said gelatinizable fibers at said points of contact, the interstices of the mat not being filled with said gelatinizable material, and the individual non-gelatinizable fibers not being uniformly coated therewith.

9. The product set forth in claim 8 in which the gelatinizable fibers are nitrocellulose.

10. The product set forth in claim 8 in which the gelatinizable fibers are cellulose fibers and the ungelatinizable fibers are animal fibers.

11. The product set forth in claim 8 in which the mixture of fibers consists primarily of relatively gelatinizable and ungelatinizable cellulose fibers.

GEORGE L. SCHWARTZ.

75