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PROCESS FOR PUFFING HYDROPHOBIC NYLON TYPE FIBERS BY SWELLING THE FIBERS AND GENERATING OXYGEN IN SITU

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The present invention relates to the production of aerated synthetic fibres, particularly such fibres of the polyamide, polyester, and polyacrylonitrile types.

It is generally known, that fibers consisting of regenerated cellulose have a substantially higher thermal conductivity in comparison with native cellulose fibers, and that the heat insulating capacity of garments, which are made of regenerated cellulose fibers, is accordingly only low. In order to overcome this drawback, it has already been proposed to provide heat-insulating voids within the regenerated cellulose fibers, and such methods have been reported in the literature in several instances. According to one of those methods, a spinning composition having finely divided gas bubbles suspended therein is produced by either adding gas evolving substances to the spinning solution or by directly aerating said solution with air or another gas, and then coagulating the formed dispersion during the spinning operation. Generally the gas is only liberated from the gas evolving substance after the spinning composition emerges from the spinneret. According to another method, some suitable solid or liquid substance is added to the spinning solution, which substance is subsequently dissolved out of the filaments, whereby fine hollow spaces are formed within the fibers. A similar result can be obtained, if the cellulose fiber is swelled in an aqueous medium, which contains unstable compounds, the latter being subsequently decomposed under evolution of gas. However, those above-mentioned procedures have heretofore only been applied to regenerated cellulose fibers.

It is therefore an object of the present invention to provide means facilitating the manufacture of aerated fully synthetic fibers, such as polyamide, polyester or polyacrylonitrile fibers, which will have improved heat insulating properties.

A further object of the invention is to provide means enabling manufacture of aerated fully synthetic fibers, which will have improved optical characteristics.

Still another object of the invention is to provide means contributing to the manufacture of aerated fully synthetic or chemical fibres, which aerated fibers will have unbroken surfaces.

The fully synthetic fibers which may be treated by the process of the invention include polyamide fibres of the polyhexamethylene-diamine-adipic acid type, such as "66 nylon," and the poly-5-aminocaproic acid type, such as "6 nylon," polyester fibres, such as polyethyleneterephthalate ("Terylene"), and polyacrylonitrile fibres, such as "Orlon" and the like.

A great variety of both inorganic and organic substances can be used as swelling agent. Examples of suitable compounds are chloroacetic acid, acetic acid, formic acid, mineral acids, saturated salt solutions, chloral hydrate solution, phenols, glycols etc. It is understood, that the particular chosen swelling agent will depend on the type of synthetic fibers used. Thus for polyamide fibers chloroacetic acid, acetic acid, formic acid and mixtures thereof have been found particularly suit-

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able. For polyester fibers hydroxylic compounds, such as phenol can be conveniently used. For fibers of the polyacrylonitrile type, e.g. an ethanolic solution of calcium thiocyanate was found to be suitable.

5 Among the peroxy-compounds, which will evolve the gas or oxygen respectively within the fibrillae of the treated chemical fibers, hydrogen peroxide has been found most suitable; however, sodium peroxide and similar peroxy compounds are also satisfactory for the method of the invention.

10 The employed catalyst serves to accelerate the rate of decomposition of the peroxides within the fibres. Suitable catalysts include heavy metals, such as silver, gold, platinum, palladium, rhodium, iridium, osmium, mercury, manganese, cobalt, iron and copper, either in metallic form or in the form of their salt-compounds. Aqueous solutions of potassium permanganate, of manganous salts, such as manganous chloride and manganous sulphate, and of cobaltous salts, such as cobaltous sulphate are particularly satisfactory for the purpose of the invention.

20 When carrying out the above-mentioned process, the fibers which may have been previously dried are treated either in filament form or in form of fabric or tissues successively with a suitable swelling agent, with a catalyst for promoting the decomposition of peroxides and a peroxy-compound, preferably hydrogen peroxide. The individual steps of the process can be carried out at room temperature or alternatively at elevated temperature. In some cases the fibers are conveniently immersed in diluted acetic acid after the peroxide treatment for a short period of time. By the process of the invention the gas or oxygen bubbles evolve, and voids are generated within the fibrillae of the fibers. The inflation of the fibers can be controlled as to different degrees by forming smaller or larger bubbles, by employing predetermined concentrations of the reagents or by change of the pH value of the treatment baths. Despite the inflation of the full synthetic fiber structure by means of smaller or larger bubbles, the structure itself and the walls and surfaces thereof will not be destroyed or broken.

40 The chemical fibers treated in this manner possess a multiplicity of uniformly distributed hollow spaces. The volume of the fibers is thereby substantially increased, resulting in improved heat insulating properties, a lower specific gravity and a higher covering power of said fibers. The numerous internal curved interfaces of the fibrillae further result in increased diffusion of the reflected light, whereby a subdued lustre is conferred upon the fibers. In comparison with massive fibers both the dry and wet tenacity of the described hollow fibers is but slightly reduced and is still higher than the corresponding tenacity of viscose rayon.

Example 1

55 A sample of "6 nylon" fibers is swelled for 2 minutes in concentrated acetic acid. The excess acetic acid is then removed and the fibers are treated for 1 minute in a 1% aqueous solution of potassium permanganate, after this operation the fibers show an intense violet appearance. The sample is then washed with cold water until it assumes a pale rose colour, centrifuged and immersed in 50 cc. of hydrogen peroxide of 40% strength. A vigorous reaction immediately sets in, which results in the inflation of the fibers. After about 5 minutes the reaction is completed. The fibers are thoroughly rinsed with water and dried. The thus treated fibers show a considerable increased water absorption, the latter amounting to approximately 46% as compared with 13% in the untreated material.

Example 2

70 A sample of "6 nylon" fibers (undelustered) is swelled for 1 minute in 70% aqueous chloroacetic acid,

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rinsed with water and subsequently dipped into a 10% aqueous solution of manganous sulphate. The sample is washed for a short time with water and pulled through a 2% solution of caustic soda. The fibers are then treated for 5 minutes with hydrogen peroxide of 40% strength, the "6 nylon," which is soaked with the solution of the catalyst, being strongly puffed up by the decomposing hydrogen peroxide during this operation. After removal from the peroxide bath the sample is washed several times with water and dried. The fibers have now a delustered appearance, and when they are examined under the microscope, they show a multiplicity of elongated bubbles or voids, particularly along the fiber axis.

Example 3

A sample of "6 nylon" fibers is swelled for 1 minute in 70% aqueous chloroacetic acid, washed with water and subsequently treated for 2 minutes with a 2% aqueous solution of potassium permanganate. After washing out the excess catalyst, the sample is immersed for 5 minutes in a 10% aqueous sodium hydroxide solution, rinsed and dipped into a bath of hydrogen peroxide of 40% strength. After 8 minutes it is removed therefrom, washed and dried.

Example 4

A sample of "6 nylon" fibers is swelled for 20 minutes in a mixture of 85 parts of acetic acid and 15 parts of formic acid. The fibers are then dipped into a 10% solution of manganous chloride in hydrochloric acid and left therein for 3 minutes. The sample is then treated with a solution of sodium peroxide in hydrochloric acid. After 3 minutes the fibers are washed with cold water, centrifuged and dried. The fibers show big spherical bubbles or voids.

Example 5

A sample of "66 nylon" fibers is swelled for 1 minute in a mixture of 35 parts of formic acid and 65 parts of acetic acid. After removal of the excess swelling agent, the fibers are treated for 2 minutes with a 1% aqueous solution of potassium permanganate.

The sample is then shortly washed with water, treated for 3 minutes with a 10% aqueous sodium hydroxide solution, washed again with water and finally dipped for 5 minutes into a bath containing hydrogen peroxide of 40% strength. It is then rinsed and dried.

Example 6

A 2 gm. sample of "66 nylon" is swelled for 1 minute in a mixture of 35 parts of formic acid and 65 parts of acetic acid. After removal of the excess swelling agent, the fibers are treated for 2 minutes with a 10% aqueous solution of manganous sulphate. After the fibers have been washed for a short time with water, they are immersed for 3 minutes into a 10% solution of caustic soda, washed again with water and finally dipped for 5 minutes into hydrogen peroxide of 30% strength. The excess hydrogen peroxide is thoroughly washed out with water, and the sample is dried at 60° C. The fibers show a grained appearance and are greatly interspersed with fusiform voids.

Example 7

A hank (11 g.) of "66 nylon" is swelled for 2 minutes in 200 cc. of glacial acetic acid. The excess swelling agent is then squeezed out and the hank is dipped into 300 cc. of a 0.1% aqueous potassium permanganate solution. After 20 seconds it is removed therefrom, rinsed for a short time with water and immersed for 3 minutes into a bath containing 250 cc. of a 5% sodium hydroxide solution. The hank is subsequently thoroughly washed, squeezed out and treated with 300 cc. of hydrogen peroxide of 40% strength. After 10 minutes the hank is thoroughly washed with water and dried. After the treatment the individual fibers are greatly interspersed

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with bubbles or voids of medium size and possess a delustered appearance.

Example 8

A sample of "6 nylon" fibers (undelustered) is swelled for 2 minutes in glacial acetic acid. The excess swelling agent is then squeezed out and the fibers are immersed for 10 minutes in a 10% aqueous solution of cobaltous sulphate. The fibers are then placed for 3 minutes in a 2% solution of caustic soda, washed with water and treated with hydrogen peroxide of 35% strength. The vigorous reaction, which sets in immediately, subsides after 3 minutes, whereupon the sample is removed from the bath, rinsed several times with water, freed from adhering water in the centrifuge and dried. The individual fibers have a delustered appearance which is due to the many very fine bubbles or voids.

Example 9

A 6 nylon fabric is swelled for 5 minutes in a mixture of 15 parts of concentrated formic acid and 85 parts of glacial acetic acid. It is then freed from excess swelling agent and immersed for 2 minutes in a 1% aqueous solution of potassium permanganate. After shortly washing the piece of fabric with water, it is dipped for 5 minutes into a 10% aqueous solution of sodium hydroxide, washed, centrifuged and finally treated with hydrogen peroxide of 40% strength. After 3 minutes the fabric is removed from the hydrogen peroxide bath, is placed for 2 minutes in acetic acid of 10% strength, whereby it assumes a pure white appearance. It is then rinsed with water and dried.

Example 10

A sample of "Terylene" fibers is swelled in phenol for a few minutes at a temperature of about 50° C. The excess swelling agent is then washed out with acetone and the fibers are dipped for 5 minutes into a 1% aqueous solution of potassium permanganate. The sample is centrifuged, placed for 2 minutes in a 2% solution of caustic soda and subsequently in hydrogen peroxide of 35% strength. When the reaction has subsided, the fibers are washed several times with water and dried. The voids within the fibers, which are generated by the above treatment, can be easily observed under the microscope.

Example 11

A sample of "Orlon" fibers is swelled for 3 minutes in a saturated ethanolic solution of calcium thiocyanate and then shortly washed with water.

The fibers are subsequently placed into a 4% hot aqueous solution of potassium permanganate, rinsed again with water and pulled through a 5% solution of caustic soda. The sample is again washed with water and then treated with hydrogen peroxide of 40% strength, which has been warmed to 70° C. The treatment is continued for 10 minutes, whereupon the fibers are rinsed and dried.

Various changes and modifications may be made without departing from the spirit and scope of the present invention and it is intended that such obvious changes and modifications be embraced by the annexed claims.

Having thus described the invention, what is claimed as new and desired to be secured by Letters Patent, is:

1. A method of manufacture of aerated synthetic hydrophobic polyamide fibres of polyhexamethyleneadipamide and poly 5 aminocaproic acid, comprising the steps of treating said fibres for between 1 minute to about 20 minutes with an aqueous solution containing a swelling agent, said swelling agent consisting of at least one member of the group consisting of acetic acid, chloroacetic acid and formic acid, washing said fibres to remove any excess swelling agent therefrom, treating said fibres with an oxygen generating catalyst consisting

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of approximately a 1% aqueous solution of potassium permanganate for about 1 minute until said fibres show an intense violet color, washing said fibres with water until the fibres are a pale rose color, immersing the fibres in a solution of about 40% hydrogen peroxide for about 5 minutes until said hydrogen peroxide is decomposed under the influence of said catalyst evolving gas bubbles inflating said fibres without breaking the walls and surfaces of said fibres and thus creating voids in said fibres.

2. A method of manufacture of aerated synthetic hydrophobic polyamide fibres of polyhexamethylene-adipamide and poly 5 aminocaproic acid, comprising the steps of treating said fibres for between 1 minute to about 20 minutes with an aqueous solution containing a swelling agent, said swelling agent consisting of at least one member of the group consisting of acetic acid, chloroacetic acid and formic acid, washing said fibres to remove any excess swelling agent therefrom, treating said fibres for between 1 minute to about 10 minutes in a solution containing about 0.1 to 10% of an oxygen generating catalyst selected from the group consisting

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of permanganates, manganous salts and cobaltous salts, and immersing said fibres for up to about 3 to 10 minutes in a solution of about 25 to 40% concentration of a peroxide compound selected from the group consisting of sodium peroxide and hydrogen peroxide, until said peroxide compound is decomposed under the influence of said catalyst evolving gas bubbles inflating said fibres without breaking the walls and surfaces of said fibres and thus creating voids in said fibres.

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