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# PROCESS FOR PRODUCING MATTED FIBERS OF CELLULOSE ESTERS

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This invention relates to producing matted fibers from cellulose esters, and it has for its object to provide a novel and improved process for this purpose.

A related object of the invention is to provide a simple and economical process for producing matted or non-glossy cellulose ester fibers of dull wool-like appearance having desirable stable color tints and shades.

Various other objects and advantages will be apparent as the nature of the invention is more fully disclosed.

In the manufacture of chemical fibers it is known to add titanium dioxide and the like to the parent materials in order to obtain matted threads and fibers. Owing to the white content, however, particularly dark color tones or hues such as black, marine blue, dark brown, etc., are fundamentally changed in an undesirable manner by such means. The also well-known treatment of threads and fibers in a treating bath which contains a melamine-formaldehyde condensation product for the purpose of matting, requires a special process and produces unfavorable changes especially in the appearance and the color tone of the threads and fibers. In these known processes, it is therefore impossible to obtain a matting without changing the appearance and the color tone in some undesirable manner.

I have now discovered a process which does not suffer from the above-mentioned defect and which makes possible the production of matted uncolored or colored threads from cellulose esters, e.g. cellulose acetate, cellulose acetobutyrate, etc., without any special operational precautions. The process consists essentially in that to the parent material finally divided precondensates of melamine and formaldehyde are added under 2%, preferably about 1%, for the production of the fibers, whereupon the threads or fibers are subjected to the action of heat, e.g. hot air or water vapor until the desired matting effect appears.

As formaldehyde-melamine condensation products I prefer particularly di-, tri- or tetramethylol melamine as well as their mixtures.

In the simplest embodiment of the invention there are added to an acetyl cellulose solution small quantities, e.g. 0.7% of a precondensate of melamine and formaldehyde, referred to the acetyl cellulose employed, and if necessary dyestuffs, followed by spinning according to the dry or wet spinning process, and treating before and after cutting the staple threads with hot water, steam and the like, whereby the previously bright fibers acquire a matted or dulled finish.

Through the process of the present invention the fibers made of cellulose esters receive a wool-like matte or dull appearance without, however, a change of the color tone and without the physical properties being impaired, particularly the consistency or stability against the reaction of light.

*Example 1*

In a kneading machine a mixture of 2.8 kg. of dimethylol-melamine and 2.8 kg. of acetylcellulose is kneaded in an acetic acid content of 54.5% and 2.1 kg. of phthalic acid dimethyl ester for 30 minutes. The kneaded mass is taken up with a mixture of 1 kg. methanol and 19.3 kg. acetone and the viscous solution is



filtered in known manner through absorbent cotton molton wadding. 4.5 kg. of this stock or mother paste are added to a spinning solution consisting of 24 kg. cellulose secondary acetate and a mixture of 3.8 kg. acetone and 72.2 kg. of methanol, so that there are present in the spinning solution about 2 parts of dimethylol-melamine to about 100 parts of acetyl cellulose. In spinning according to the dry spinning process and the staple fiber cut in the known manner, there is first obtained a fiber which does not differ from that obtained without the addition. By boiling this fiber in water or treating with saturated steam a permanent turbidity results whereby a matting or dulling effect is obtained similar to that obtained with the addition of titanium oxide. The tearing strength of a matted fiber obtained in this way amounts (with the use of the same cellulose acetate) 1.4–1.5 g. as against 1.3–1.4 g. in the case of titanium oxide addition.

The fiber matted according to the invention behaves with the reaction of light exactly as favorably as a fiber without addition, while with a matting with titanium dioxide in spite of slowly acting additions an impairment through the action of light cannot be avoided.

*Example 2*

A pigment coloring material or dye, for instance 1.3% (referred to acetyl cellulose) indanthrene brilliant Bordeaux RRL or indanthrene scarlet GL is added in the usual way to the spinning solution obtained according to Example 1. Upon spinning a brilliant web is obtained the color of which remains unchanged by boiling or steaming, while it acquires by such boiling or steaming a matte or dull wool-like appearance.

*Example 3*

From the dimethylol-melamine containing spinning solution obtained according to Example 1, there is spun a 30 denier acetate silk in the known manner. For the development of the matting there is inserted in the process a steaming interval, for instance during respooling, whereby the matting takes place through direct treatment with steam.

*Example 4*

In a kneading machine a mixture of 2.8 kg. trimethylol-melamine, 2.8 kg. triacetate is kneaded as in Example 1, with an acetic acid content of 61–62% and 2.1 kg. trichlorethylphosphate. The kneaded mass is taken up with a mixture of 3 kg. methanol and 17.3 kg. methylene chloride and thereupon filtered in the usual known manner. To a spinning solution consisting of 18 kg. cellulose triacetate and a mixture of 12.3 kg. methanol and 69.7 kg. methylene-chloride there are added 1.7 kg. of the filtrated kneaded mass, and otherwise the treatment is according to Example 1. The matted triacetate fiber obtained after steaming or boiling has a tear strength better by about 10 to 20% than a fiber which has the same percentage of content of titanium dioxide. An impairment through light does not take place even in the case of the triacetate fibers.

*Example 5*

The methylol-melamine containing spinning solution according to Example 3 is wet spun in ethylhexanol. The web or fiber obtained behaves in boiling and steaming the same way as the one obtained with dry spinning processes. Also in this case the desired matting takes place at the retreatment whereby in the strength properties no difference is found as against the fabric obtained without addition.

*Example 6*

To a spinning solution, consisting of 24 kg. cellulose acetobutyrate with 21% bonded butyric acid and 76 kg. acetone there are added 4.5 kg. of parent paste which is obtained similarly to Example 1, through kneading of 2.8 kg. of a mixture of di- and tetramethylol-melamine, 2.8 kg. acetobutyrate as above and 2.1 kg. phthalic acid dimethylester, so that to about 100 parts acetobutyrate there are about 2 parts methylol-melamine present.

The spinning solution is processed in the known manner after the dry spinning process to threads, as a result of which they first come out glossy; through boiling or treatment with steam the matting according to the present invention occurs.

Although certain specific examples have been disclosed herein for purposes of illustration, it will be evident to those skilled in the art that the invention is capable of various modifications and adaptations within the scope of the appended claims.

The invention claimed is:

1. Process for producing matted fibers from cellulose esters which comprises preparing a spinning solution



comprising an organic solvent containing a cellulose ester, and a precondensate of melamine and formaldehyde in an amount less than 2%, spinning said solution to form fibers, and then heating said fibers until they become matted.

2. Process for producing matted fibers from cellulose acetate which comprises preparing a spinning solution comprising cellulose acetate, an organic solvent selected from the group consisting of acetone and methylene chloride, and a precondensate of melamine and formaldehyde in an amount less than 2%, spinning said solution to form fibers, and subjecting said fibers to the action of water at a boiling temperature.

3. Process according to claim 2, in which about 1% of precondensate of melamine and formaldehyde is employed.

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