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TREATMENT OF CASEIN FIBERS

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10 Claims. (Cl. 28—74)

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This invention relates to a process of preparing synthetic fibers derived from casein or from other protein substances for dyeing in certain dyebaths so that these synthetic fibers may be blended or otherwise used together with wool and/or mohair fibers in the manufacture of textile fabrics which are to be dyed in the piece to a substantially uniform shade without objectionably noticeable striations appearing. My improvements are of particular utility in the union dyeing of fabrics, both pile and flat, containing cotton or other cellulose fibers as well as wool and/or mohair. By my improvements a novel fiber and fabric are produced. This application is a division of my prior application, Serial No. 415,631, filed October 18, 1941.

Throughout this specification the term "casein fibers" will for brevity be used, but the term will be understood to include other synthetic protein fibers such as those derived from the soy bean or from zein (corn), as well as those derived from casein.

Synthetic casein fibers are of protein material and accordingly have affinity for many of the same dyes as have wool and mohair, but the initial affinity of the synthetic casein fibers for dyestuffs is much stronger than that of the wool and mohair fibers. Consequently, if untreated casein fibers be blended with wool and/or mohair fibers in yarns which are then dyed by short time immersion methods, as in pad dyeing, the casein fibers will dye so much darker than the wool and mohair as to constitute a difference in color. Where the time of immersion is long, as in kettle dyeing, the stronger initial affinity of the synthetic casein fibers will be partially overtaken but there will nevertheless be a substantial difference in shade between the synthetic casein and the natural wool and/or mohair fibers.

In the manufacture in quantity of textile pile fabrics having a cotton backing or base portion and wool, or mohair, or blends thereof, with or without other fibers, including casein fibers, in the pile, it is very desirable for reasons of speed and economy of production to dye the fabric in the piece in a union dye bath in a continuous operation using pad dyeing methods. A union dye bath which I contemplate is an aqueous bath containing (*a*) neutral dyeing acid dyestuffs for the wool, casein and mohair fibers, and (*b*) substantive dyestuffs intended primarily for the cotton fibers. The wool, casein and mohair fibers also have affinity for the substantive dyestuffs so that the shade or color to which they dye is the combined effect of both components of the union dye bath.

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When untreated synthetic casein fibers are included in the pile yarn blend together with wool and/or mohair, and the fabric union dyed in a pad dyeing operation, the synthetic casein fibers will ordinarily dye much darker or deeper than the wool and mohair fibers. As indicated hereinbefore, if such a fabric be union kettle dyed, the difference will not be as great due to the often repeated immersions, but there will nevertheless be a substantial contrast in shade.

A difference in color or shade will also occur if untreated synthetic casein fibers be blended with wool and/or mohair fibers in fabrics, such as worsted flat fabrics, having no cotton and the fabrics dyed by pad dyeing methods with acid dyestuffs in a substantially neutral bath.

According to my improvements I may produce a treated casein fiber which will dye to a shade approximating that of wool or mohair when the two types of fibers are dyed together in a substantially neutral aqueous union bath containing neutral dyeing acid dyestuffs and substantive dyestuffs using either short time immersion methods, as in pad dyeing, or long time repeated immersion methods, as in kettle dyeing. My treated casein fiber will also dye similarly to wool or mohair when the two types are dyed together by either pad or kettle methods in a substantially neutral aqueous bath containing only neutral dyeing acid dyestuffs or when the two types are pad dyed together with vat dyestuffs. My treated casein fiber does not, however, dye like wool or mohair when the two are dyed together by either pad or kettle methods in acid dye baths.

The treatment given the casein fiber to accomplish the foregoing effects does not weaken the fiber nor does it make the fiber stiff or hard or harsh or gummy. In other words, the treated fiber retains substantially its initial strength, flexibility, elasticity and feel.

My method comprises a two bath treatment whereby a water insoluble metallic tannate is incorporated with the casein fiber. The tannate is preferably light colored so as to affect as little as possible the color or shade to which the fiber is dyed. This is important in connection with producing shades currently used for most automobile upholstery pile fabrics.

According to a preferred embodiment of my improvements, the casein fibers are first subjected to an aqueous solution of tannic acid. The excess solution may then be removed and the fibers next immersed in an aqueous solution, preferably of stannous chloride, whereby tin tannate is incorporated with the fibers. The fibers may then be rinsed and dried. The second bath

may if desired be an aqueous solution of any water soluble aluminum salt such as aluminum sulphate or aluminum acetate, whereby aluminum tannate will be incorporated with the fibers. Or the second bath may be an aqueous solution of tartar emetic, whereby antimony tannate will be incorporated with the fibers. A small amount of acetic acid may preferably be used in each bath, the presence of acetic acid being more important in the second bath than in the first. The time of immersion, the temperature of the baths and the concentration of the solutions will be more fully described hereinafter.

It is an object of this invention to prepare synthetic fibers derived from casein or other protein substances for dyeing so that the said synthetic fibers may be blended or otherwise used together with wool and/or mohair and cotton fibers and dyed in a union bath without objectionably noticeable striations appearing.

More specifically, it is an object of this invention to prepare artificial fibers derived from casein for dyeing in a union bath containing neutral dyeing acid dyestuffs and substantive dyestuffs, or for dyeing with neutral acid dyestuffs, or with vat dyestuffs using short time immersion methods as in pad dyeing.

Another object is to produce a synthetic casein fiber having a water insoluble metallic tannate incorporated therewith, which when subjected to neutral dyeing acid dyestuffs, or to substantive dyestuffs, or to both as in union dyeing, or to vat dyestuffs in a short time immersion operation will dye to a shade approximating the shade of wool fibers subjected to the same dye bath for the same length of time under the same conditions.

Another object is to provide a method of incorporating a water insoluble tannate with the casein fiber.

Another object is to produce a uniformly dyed textile fabric including casein, cotton and other cellulose fibers, and wool fibers, said casein fibers having a water insoluble metallic tannate incorporated therewith.

Another object is to incorporate tin tannate, or aluminum tannate, or antimony tannate, with the casein fiber, whereby the fiber will have an affinity approximating that of wool fibers for neutral dyeing acid dyestuffs, or substantive dyestuffs, or both as in union dyeing.

Another object is to treat casein fibers so that they may be blended together with wool and/or mohair fibers in the pile of pile fabrics having a cotton backing, and dyed together therewith in a union bath using short time immersion methods as in continuous pad dyeing, all of the blended fibers in the pile being dyed to nearly the same shade.

These and other objects of invention will be manifest from a consideration of the specification and of the claims.

According to a preferred embodiment of my invention, the synthetic casein fibers are immersed in one quarter of one per cent. aqueous solution of tannic acid at 160° F. It is desirable but not essential that the solution contain a small quantity of acetic acid. After about five or more minutes of being worked in the solution as by gentle agitation, the stock may be rinsed or nipped or the solution may be merely permitted to drain off.

The casein fiber stock is then immersed for five or more minutes in the second bath, which is preferably an aqueous solution, at approximately 120° F., containing stannous chloride in an amount approximating three per cent. of the weight of the stock. A temperature substantially higher than 120° F. would damage the available casein fibers, and a substantially lower temperature would give markedly inferior resist effects for the contemplated dyebaths. Acetic acid in an amount approximating one per cent. of the weight of the stock may be contained in the second bath. As a result of the reaction between the tannic acid and the stannous chloride, tin tannate is incorporated with the fibers.

The metallic salt of the second bath may, if desired, be aluminum sulphate or aluminum acetate, in which case aluminum tannate is incorporated with the fibers. Or the second bath may be an aqueous solution of tartar emetic, in which case antimony tannate is incorporated with the fibers. Tin tannate, aluminum tannate and antimony tannate are all light in color and accordingly will not substantially darken the natural color of the casein fiber so as to interfere with or limit the color or shade which it is desired to apply in the subsequent dyeing operation.

In each bath the volume of aqueous solvent should be sufficient to work the fiber stock therein. The weight of the aqueous solution may conveniently be about fifteen times the weight of the fiber stock.

Attention is directed to the fact that in the preferred treatment described above the temperature of the tannic acid bath is approximately 160° F. This temperature is just above the point where the casein fibers swell markedly and at this temperature the fibers will absorb the tannic acid solution more readily than at substantially lower temperatures. Temperatures below 160° F. may be employed if desired, but the time of immersion should be increased accordingly.

Higher temperatures than 160° F. may also be employed, but temperatures substantially in excess of 180° F. should not be used, as the strength of the casein fibers will be impaired. I have obtained satisfactory resist effects using as a first bath a one-half of one percent aqueous solution of tannic acid at a temperature of 180° F., the time of immersion being two minutes. As indicated, this is a maximum temperature and a two minute immersion period is minimum, as shorter periods of immersion do not give sufficiently strong resist effects.

The time of immersion in the second bath should, as indicated above, be five minutes or more and the amount of stannous chloride in solution should neither greatly exceed nor be greatly less than three per cent. of the weight of the fibers being treated, as in either case the shade of the dye would be adversely affected, and if more is used the fibers may be weakened. My improvements are particularly applicable for pad type union dyeing where the time of immersion is very short, that is, a matter of seconds. This type of dyeing is widely used in the production of pile fabrics, as is set forth in United States Patent to Drobile et al. No. 2,071,922. A satisfactory dyebath immersion time in processing according to the prior patent is ten seconds. I have found that after a ten second immersion my treated casein fibers will approximate the shade of wool and/or mohair blended therewith. It is to be understood that wool and mohair do not dye identically, but that they dye sufficiently close in shade to permit blending wtihout objectionably noticeable striations in the finished piece. My treated casein fibers may be brought within a shade range which permits blending with wool or mohair to give a like uniform appearance to the

cloth after a ten second immersion as set forth. This result is accomplished by the treatment illustrated by this preferred embodiment.

A typical treatment of a commercial quantity of casein fibers according to my method is as follows:

500 lbs. of synthetic casein fibers is placed in a suitable container or kettle. The aqueous solution of tannic acid at about 135° F. is drawn from a reservoir, placed in the kettle and then brought to a temperature of 160° F. and held there for approximately ten minutes, during which the fiber stock may be gently agitated. The tannic acid solution is then drained out and pumped back into the reservoir. A light rinse may then be given the casein fibers, after which any excess water or solution may be removed. The fibers are now ready for the second bath. Water and half of the required acetic acid is poured into the kettle, then the solution of stannous chloride and the remainder of the acetic acid is added and the entire solution brought to 120° F. and held there. The fibers are kept in this solution for approximately five minutes, after which the kettle is drained, the fibers rinsed and then dried. In this condition the fibers are ready for blending with wool or other fibers or for use alone in the formation of yarns.

As indicated hereinbefore, casein fibers treated according to my methods will dye approximately like wool when the two types of fibers are dyed together, by either pad or kettle methods, in a substantially neutral aqueous medium containing neutral dyeing acid dyestuffs or substantive dyestuffs, or both as in union dyeing, or pad dyed with vat dyestuffs. Casein fibers treated according to my method will not, however, dye like wool where the two are immersed together in an acid bath containing acid dyeing acid dyestuffs.

Having described my invention, I claim:

1. In a process of making pile fabrics which contain natural staple animal fibers and synthetic fibers derived from casein in the pile portion, the steps which include treating the synthetic fibers derived from casein so as to incorporate a light colored metallic tannate in the fibers, blending the casein fibers with the natural animal fibers, and fabricating said blended fibers into the pile portion of a pile fabric and then pad dyeing the animal and synthetic fibers of the pile to approximately the same shade.

2. In a process of making pile fabrics which contain natural animal fibers and synthetic fibers derived from casein in the pile portion, the steps which include treating the synthetic fibers derived from casein so as to incorporate a light colored metallic tannate in the fibers, blending the treated casein fibers with the natural animal fibers and fabricating said blended fibers into the pile portion of a pile fabric having a cotton backing, and then pad dyeing the pile fabric to approximately a uniform shade in the pile portion.

3. In a process of making pile fabrics the pile portion of which contains natural staple animal fibers and synthetic fibers derived from casein, the steps which consist of treating the synthetic fibers so as to incorporate a light colored metallic tannate in the fibers, blending the casein fibers with the natural animal fibers and fabricating the blended fibers into the pile portion of a pile fabric, and then dyeing both types of said pile fibers to approximately the same shade by the use of a neutral dye bath containing neutral dyeing acid colors.

4. In a process of making pile fabrics containing natural staple animal fibers and synthetic fibers derived from casein in the pile portion, the steps which consist of treating the synthetic fibers derived from casein so as to incorporate a light colored metallic tannate in the fibers, blending the casein fibers with the natural animal fibers, and fabricating said blended fibers into the pile portion of a pile fabric having a cellulose backing, and then dyeing the animal and synthetic fibers to approximately the same shade with a neutral dye bath containing neutral dyeing acid colors and substantive dyes.

5. A process of making pile fabrics of approximately uniform shade which includes the steps of treating synthetic fibers derived from casein so as to form a light colored metallic tannate in the fiber, blending the casein fibers with natural animal staple fibers and forming a yarn, fabricating said yarn into the pile portion of a pile fabric and piece dyeing the pile face to an approximately uniform shade with a substantially neutral dye bath containing neutral dyeing acid colors.

6. A process of making pile fabrics of approximately uniform shade in the face portion, which process includes the steps of treating synthetic fibers derived from casein so as to form a light colored metallic tannate in the fibers, blending the casein fibers with natural animal staple fibers and forming a yarn, fabricating said yarn into the pile portion of a pile fabric having a cotton backing and union piece dyeing the pile fabric to an approximately uniform shade with a neutral dye bath containing neutral dyeing acid colors and substantive dyes.

7. A process of making pile fabric having a pile portion of approximately uniform shade which includes the steps of treating synthetic fibers derived from casein so as to form a light colored metallic tannate in the fibers, blending the casein fibers with natural animal staple fibers and forming a yarn, fabricating said yarn into the pile portion of a pile fabric, and pad dyeing the pile fibers to an approximately uniform shade with a substantially neutral dye bath containing neutral dyeing acid colors.

8. A process of making pile fabric having a pile portion of approximately uniform shade, which includes the steps of treating synthetic fibers derived from casein so as to form a light colored metallic tannate in the fibers, blending the casein fibers with natural animal fibers and forming a yarn, fabricating said yarn into the pile portion of a pile fabric having a cotton backing and union pad dyeing the pile fabric to an approximately uniform shade with a substantially neutral dye bath containing neutral dyeing acid colors and substantive dyes.

9. A process of making pile fabrics of approximately uniform shade which includes the steps of treating synthetic fibers derived from casein so as to form a tin tannate on the fibers, blending the casein fibers with natural animal fibers and forming a yarn, forming a fabric having a cotton base portion and a pile portion of the blended yarn and dyeing the entire fabric to approximately the same shade with a substantially neutral dye bath containing neutral dyeing acid colors and substantive dyestuffs.

10. A process as set forth in claim 9, further characterized in that the tin tannate is formed by treating the casein fibers with each of two separate solutions, one of which contains tannic acid and the other of which contains a tin salt.

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