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PARTIAL SAPONIFICATION OF MATERIAL CONTAINING ORGANIC ESTERS OF CELLULOSE

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This invention relates to the treatemnt of products made of or containing organic esters of cellulose.

An object of our invention is to partially saponify products such as textile materials, films and the like containing organic esters of cellulose in such a manner that all the desirable properties of such products are retained, while further desirable properties are imparted thereto.

A further object of our invention is to partially saponify textile materials comprising or consisting of organic esters of cellulose and particularly cellulose acetate, under such conditions that it is possible to dye the material evenly.

Another object of our invention is to treat textile materials such as fabric, yarn or the like containing organic esters of cellulose with a bath containing hydroxyl ions of such concentration and at such temperature, that partial saponification of the organic esters of cellulose takes place in such a manner that the ironing temperature of the textile material is raised to almost the maximum with a minimum degree of saponification, which saponification is uniform. Other objects of our invention will appear from the following detailed description.

The partial saponification of fabrics containing cellulose acetate has formerly been proposed with the view of rendering the same capable of being dyed by cotton dyes. However, in the former practice, the saponification was carried to such a large exent, of the order of 25% loss by weight that many of the valuable properties of cellulose acetate fabrics were destroyed, and the great loss of weight of the material involved economic disadvantages. Moreover, the alkali concentration of the saponification bath and the temperature of the bath were so high and were so conducted that even saponification did not take place, so that the fabric did not dye evenly unless the saponification took place to such an extent that the acetyl value was decreased to an enormous extent and even then the dyeing was not as even as that produced by fabrics treated by our invention.

Fabrics made of or containing organic derivatives of cellulose, particularly cellulose acetate, are now in wide use because of their many highly desirable properties. However, in ironing fabrics composed at least in part of organic esters of cellulose, for instance cellulose acetate, care must be taken to avoid too high temperatures, since if the iron or calender is above 200° to 230° C., sticking of the fabric to the iron or calendar and melting of the cellulose ester will sometimes result. It is therefore desirable to raise the permissible temperature of ironing as much as possible.

It has been found that when a textile material containing cellulose acetate which sticks to an iron above 220° C. is saponified, the safe ironing temperature is progressively raised with progressive increases of degree of saponification until an ironing temperature of above about 260° C. is reached, above which temperature the material, as well as most other fabric materials, scorches regardless of the degree of saponification. Thus if the goods has been saponified 1%, that is it has suffered a loss of 1%, it may be ironed at 230° C., if saponified 3% it may be ironed at 250° C. and if saponified 5 to 6% it may be ironed at above about 260° C. The textile material may be saponified above 6 or 7%, say to 10% or more if desired, but obviously this is of no advantage from the standpoint of raising the safe ironing temperature, although it may be from the dyeing point of view. However, unless the saponification is carried out carefully and under the conditions we have discovered, the material is not saponified uniformly with the result that level dyeing may not be obtained. Fabrics containing irregularly saponified organic derivatives of cellulose moreover, do not have a uniform safe ironing point, so that if an iron is applied at a high temperature that is safe for the properly saponified parts, the threads or spots not uniformly saponified will stick or melt and thus impair the usefulness of the entire fabric or garment.

In accordance with our invention the textile material such as yarns, threads, fabrics or garments made of or containing organic esters of cellulose, and particularly cellulose

acetate, is treated with a solution containing hydroxyl ions of sufficient concentration to cause saponification of the organic esters of cellulose in an even manner, but not of such high concentration to cause irregular saponification. The temperature of the treating solution is preferably high but preferably below the boiling point of water. In order to further promote regular saponification, the addition of a large amount of a neutral salt such as sodium chloride may be made but is not absolutely necessary.

In carrying out our invention, the textile material containing cellulose acetate is submerged in a bath containing a dilute solution of an alkali or alkaline salt. The textile material may be yarn or thread in the form of hanks, cops or bobbins, or it may be any woven, knitted or netted fabric. The solution may be applied in any manner other than by immersion, such as by spraying to the textile material. The concentration of the hydroxyl ions should be sufficiently high to cause saponification of the cellulose esters. Thus we have found that an aqueous solution containing less than 0.07 grams of sodium hydroxide per litre does not saponify cellulose acetate yarns or fabrics and for practical purposes of saponification, little action takes place with 0.2 grams per litre at 80° C. after one hour. The bath should, therefore, contain hydroxyl ions of a concentration corresponding to that of an aqueous bath containing at least 0.30 or 0.35 grams of sodium hydroxide per litre at 80° C. On the other hand the concentration of the alkaline solution should be kept below that which will cause uneven saponification and thus irregular dyeing.

While we do not limit ourselves to the specific concentrations, we have found that solutions having a hydroxyl ion content corresponding to 0.4, 0.5, 0.6 or 0.7 grams of sodium hydroxide per litre give eminently satisfactory results. The temperature of the bath is maintained at 70 to 90° C. and preferably at about 80° C. We have found that although the cellulose acetate textile material will not initially be readily saponified in an alkaline bath containing less than 0.2 grams of sodium hydroxide per litre at 80° C., after the initial saponification by a stronger solution, saponification proceeds in a weaker alkaline solution so that the alkaline bath need not necessarily be maintained at the concentration above 0.2 grams of sodium hydroxide per litre. However, we have found that it is preferable to add alkali from time to time to the bath to replace that which has been consumed in the saponification reaction so as to maintain the alkaline content substantially constant, since more regular saponification is obtained in this manner.

The saponification of the organic esters of cellulose is permitted to proceed to any desired percentage of saponification which is here given as the percentage of loss of weight the material suffers by the saponification. The cellulose acetate textile material may be saponified 1% and have a safe ironing point of about 230° C. If saponified 3%, its safe ironing point is about 250° C. If saponified 5 to 6% its safe ironing point is about 260° C. This of course means that for short periods, the temperature of the iron might be 275° C. or more. Since the material scorches above 260° C. regardless of the degree of saponification proceeding further than 5 to 7% saponification presents no considerable advantage from the point of view of increase of ironing point. However, it may be desired to saponify further, say 10% to 25% or more saponification for other reasons and this may be done in accordance with our invention since by our process the textile material produces level dyeing regardless of the degree of saponification. A textile material containing cellulose acetate saponified in accordance with our invention produces level dyeing with SRA dyes and many other classes of dyestuffs, a result not attainable by the old methods of saponification.

The saponification of fabric may be carried out in a tank, on the jig or in any other suitable device. Often it is desirable to thoroughly wet out the fabric prior to subjecting it to the saponification bath. The alkaline material used in the saponification bath may be any compound that produces hydroxyl ions of the required concentration. It may be sodium hydroxide, potassium hydroxide, ammonium hydroxide, or it may be alkaline salts that produce hydroxyl ions by hydrolysis when dissolved in water, such as sodium or potassium carbonate, sodium or potassium phenolate, sodium or potassium sulphide, sodium or potassium silicate or trisodium phosphate, etc. These alkaline agents are used in such concentration as to produce hydroxyl ion content corresponding to that given by the stated proportions of sodium hydroxide. The addition of a large amount of a neutral salt such as sodium chloride or Glauber salt, to the extent of up to 20% to 25% to the saponification solution aids in the production of level saponification, especially where a more concentrated alkaline solution, such as one containing 1.0 grams sodium hydroxide per litre is used.

Examples of organic esters of cellulose that may be used as constitutents of the textile material to be treated are cellulose acetate, cellulose formate, cellulose propionate and cellulose butyrate, etc. While most of the detailed description is given with respect to cellulose acetate, this is done merely by way of example and it is equally applicable to other organic esters of cellulose.

As stated before, the product made of or containing organic esters of cellulose may be

of any character such as films or textile materials. By textile material is meant such material as yarns, threads, fabrics, garments, etc. The fabric may be knitted, woven or netted or of any other suitable construction. The fabric may consist wholly of yarns of one or more organic esters of cellulose. Moreover, the fabric may be a mixed fabric containing yarns of one or more organic esters of cellulose and yarns of one or more other fibres such as cotton, reconstituted cellulose, (artificial silk) silk, wool or linen. Examples of such mixed fabrics are mixed cellulose acetate and silk and mixed cellulose acetate and cotton.

The treatment of a mixed fabric containing organic esters of cellulose and silk in accordance with our invention is of particular advantage since if such mixed fabric contains silk in the gum, partial saponification of the organic esters of cellulose and degumming of the silk take place simultaneously. A crepe made of a cellulose acetate yarn warp and a natural silk weft has been treated by our process with highly satisfactory results.

In one mode of carrying out our invention, the goods are scoured in an open bath on strings or in any suitable machine such as a jig with a bath containing olive oil soap for an hour or so. The goods may then be removed from the bath and saponified in a separate bath containing fresh water and alkali or they may be saponified directly in the same scouring bath by the addition of a requisite amount of caustic soda. The concentration of the alkali may be maintained by the addition of small amounts of caustic soda until the desired amount of saponification is obtained. Until the operator has gained sufficient experience, it is preferable to check up the amount of caustic soda present in the bath by titration with sulphuric acid. After treatment of a few batches, the method may be standardized for any given machine. In those cases where the volume of the bath is large as compared with the weight of the goods being treated, it may not be necessary to add more alkali, the degree of saponification of the material being determined by titration of the bath to measure the amount of alkali consumed. Where the proportion of the goods is large as compared to the size of the bath, it is necessary to add alkali after the saponification has proceeded, in order to supply sufficient alkali to produce the desired amount of saponification. After the treatment with the alkaline bath, the textile material is preferably thoroughly washed to remove any trace of alkali or salts that may remain therein, since the presence of these materials causes a lowering of the safe ironing point.

In order to further illustrate our invention but without in any way being limited thereto, we give the following example:

Example

100 kilograms of a crepe fabric containing 70% of cellulose acetate yarn and 30% of natural silk are treated in an aqueous bath of 6500 litres containing 5 to 10 grams of olive oil soap per litre at a temperature of 80° C. After scouring the fabric in this bath and without removal of the fabric from the bath, there are added caustic soda in an amount corresponding to 0.40 grams per litre of bath. The duration of this treatment is ¾ of an hour. The bath is then tested for its alkali content and further quantities of caustic soda amounting to 0.2 grams per litre are added in portions, maintaining a concentration of 0.35 grams per litre of alkali until the additions are complete. The fabric is then worked for two hours to give complete degumming and saponification. The resulting fabric is found to be saponified to the extent of 5% of the cellulose acetate material, and has an ironing point of 260° C., which is equal to that of cotton. By this process the silk is completely degummed and is uninjured.

It is to be understood that the foregoing detailed description is given merely by way of illustration and that many variation may be made therein without departing from the spirit of this invention.

Having described our invention what we claim and desire to secure by Letters Patent is:

Method of increasing the ironing point of a mixed fabric containing cellulose acetate yarn and natural silk and simultaneously degumming the natural silk comprising immersing the same in a bath containing hydroxyl ions corresponding in concentration to that of an aqueous solution containing about 0.5 grams of sodium hydroxide per litre at a temperature of about 80° C., replenishing the supply of alkaline material as the same is consumed in the saponification process so as to maintain the alkalinity of the bath substantially constant and containing the treatment until the cellulose acetate is saponified not exceeding 11%.

In testimony whereof, we have hereunto subscribed our names.

CAMILLE DREYFUS.
HERBERT PLATT.

CERTIFICATE OF CORRECTION.

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CAMILLE DREYFUS, ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 3, line 114, the claim, for "containing" read "continuing"; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 9th day of May, A. D. 1933.

(Seal)

M. J. Moore,

Acting Commisioner of Patents.