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(54) **METHOD AND COMPOSITION FOR THE ALKALI TREATMENT OF CELLULOSIC SUBSTRATES.**

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Description

The present invention provides a composition for the alkali treatment of a cellulosic substrate and a method for such treatment which comprises applying the composition to such a substrate.

It is well known in the art to treat cotton material, particularly in the form of yarn or woven textiles, with aqueous caustic to improve its dyeability and other properties. While British Patent Specification 490,567 does teach the treatment of yarn with caustic potash, the industrial alkali treatment of cotton has been conducted essentially with sodium hydroxide as the causticizing agent. British Patent Specification 715,376 also discloses the treatment of cotton with a caustic potash solution, but this is a very specific treatment which is effected at a temperature below 0°C.

The composition of the present invention is an aqueous liquor containing as an essential component potassium hydroxide.

The treatment liquor may contain, in addition to the potassium hydroxide, other alkali metal hydroxides, such as sodium hydroxide. However, at least 75%, by weight, of the total alkali metal content must be potassium hydroxide. Preferably, potassium hydroxide is the sole alkali metal hydroxide present.

The amount of alkali metal hydroxide in the treatment liquor should be enough to improve the dyeability of the cellulosic substrate and is preferably in the range 120 to 400 g/l, more preferably 200 to 330 g/l, most preferably 240 to 300 g/l. The alkalinity of the treatmet liquor desirably is in the range of 180—350 g/l (18 to 30° Bé), especially 200—300 g/l (20 to 27° Bé).

In addition to the alkali metal hydroxide the treatment liquor preferably contains an alkali metal silicate. More preferably, the alkali metal silicate is sodium or potassium silicate.

The amount of alkali metal silicate present is generally up to 100 g/l and is preferably in the range 5 to 85 g/l, more preferably 10 to 70 g/l, most preferably 15 to 35 g/l, the amount being such that the alkalinity of the treatment liquor will be as stated above.

The ratio of alkali metal hydroxide to alkali metal silicate may range from 2:1 to 20:1, preferably 4:1 to 16:1, more preferably 6:1 to 13:1, by weight.

A further preferred constituent of the alkali treatment liquor is a wetting agent.

Preferred wetting agents are anionic, non-ionic or amphoteric surfactants which are stable to aqueous potassium hydroxide in the amounts employed. Such compounds are known and commerically available. More preferably the wetting agent is of the anionic type, optionally in the form of a mixture with one or more other anionic wetting agents or with a non-ionic or amphoteric wetting agent. Suitable anionic wetting agents include:

i) sulphated C_{4-24} alcohols or glycols, optionally ethoxylated with 2 to 25 ethyleneoxy units;

ii) alkyl C_{7-20} phosphoric acid esters or semi-esters;

iii) alkyl C_{1-20} poly (1—25) glycol ether phosphoric acid esters;

iv) arylsulphonates, e.g., cumenesulphonates;

v) sulphated fatty acids, e.g., sulphated aliphatic saturated or unsaturated fatty acids, preferably C_{16-18} fatty acids,

vi) sulphated fatty acid esters, mono- or diamides;

vii) sulphonated fatty acid mono- or diamides, and

viii) carboxymethylated addition products of 1 to 25 moles of ethylene oxide to a C_{4-24} alcohol.

Preferred anionic wetting agents are those of types i), iii), iv) and vii) above. The most preferred anionic wetting agent is sodium 2-ethylhexyl sulphate.

The amount of wetting agent, when present, should be sufficient to promote uniform impregnation of the substrate with the treatment liquor during the application step and is generally up to 20 g/l, preferably 0.1 to 10, more preferably 0.5 to 7.5, most preferably 1 to 5 grams, per liter of treatment liquor.

A further preferred component of the treatment liquor is an alkali-resistant agent capable of sequestering or complexing with heavy metal ions. It is believed that such an agent inhibits the formation of less water-soluble metal silicates which might interfere with the removal of the alkali metal silicate during the rinsing of the substrate which follows the alkali treatment step. Compounds useful for tying up heavy metals ions in aqueous media are known. The preferred such compounds for use in the process of the present invention are alkali metal salts of hydroxycarboxylic acids, particularly pentonic, hexonic and heptonic acids, and more particularly gluconic acid, especially sodium gluconate. Other compounds known to be useful as cation sequestering agents include alkali metal glucoheptonates and alkali metal salts of nitrilocarboxylic acids and of ethylenediamine-tetraacetic acid.

The sequestering or complexing agent is conveniently added to the treatment liquor in an amount which may range up to 40 g/l, depending, for instance on the hardness of the water. Preferably the amount of this component is in the range 0.2 to 20, more preferably 0.5 to 10, most preferably 2.5 to 5 g/l.

The metal-sequestering or complexing agent is conveniently added to the treatment liquor in admixture with a dispersing agent, preferably an anionic dispersant, such as a sulphonated fatty acid amide. A particularly preferred mixture is an aqueous composition comprising by weight, 15% sodium gluconate and 5% sulphonated fatty acid amide.

It is also advantageous to include in the alkali treatment liquor a compound having textile lubricating as well as dispersing properties, and particularly such compounds which have the further property of acting as retarding leveling agents for reactive dyes. Anionic compounds are preferred, especially

sulphonated or sulphated castor oil. The amount of such a compound is preferably 0.1 to 15, more preferably 1 to 15 grams per liter of alkali treatment bath.

The aqueous alkali treatment liquor is applied to the substrate at a temperature which is above 7°C and is generally in the range 10 to 100°C. Preferably the temperature is in the range 18—80°C, most preferably 20—35°C.

The liquor can be applied to the substrate by various methods, such as spraying, foam application or immersion. Preferably, the liquor is applied by padding.

By controlling the rate of initial application and/or by removing excess liquor, as by squeezing, a pick-up of 70 to 180%, more preferably 80 to 150%, most preferably 90 to 140% is achieved. All parts and percentages are by weight and all temperatures are in degrees Centigrade, unless otherwise indicated.

When the alkali treatment liquor is applied in the form of a foam, techniques similar to those known in the art for effecting various textile treatments can be employed. Of course, the amount of foam applied will be sufficient to bring into contact with the substrate an amount of alkali treatment components equivalent to those applied e.g. by padding.

Following the impregnation with the treatment liquor, the substrate is allowed to dwell for a period of time sufficient to permit the components of the liquor to act on the cellulosic material. Normally, the dwell time is at least 0.5 hour, preferably 0.5 to 10 hours, more preferably 1 to 4 hours. This can be conveniently effected by winding the impregnated substrate on a beam, which is optionally rotated during the dwelling. Alternatively, the goods may be stored in any suitable receptacle during the dwelling period. Dwelling is normally effected at ambient temperature, e.g. 18 to 35°C, preferably 21 to 20°C.

With bulkier materials, such as corduroy, it may not be practical to effect a dwelling in the manner described above. Rather, it is preferred to carry out the process in a continuous manner with the material being drawn from the application, e.g. padding, operation to a rinsing operation at such a rate as to allow for a relatively brief interval between these operations for the treatment liquor components to act on the material. Depending on the equipment used and the space available, this brief dwelling interval will be at least 10 seconds, preferably 15 to 120 seconds, and most preferably 30 to 60 seconds. In order to accelerate the action of the treatment liquor components on the substrate, moist heat, e.g. steam, or dry heat, e.g. at 82—105°C may be employed.

Following the dwelling, the substrate can be washed, bleached and dried in conventional fashion. Washing is generally effected with water, preferably softened or demineralized, at room temperature to boiling, preferably at 65 to 94°C. The bleaching can be effected with a conventional peroxide bleaching composition.

The substrate treated according to the present invention can be a blend of cellulosic fibers with other fibers, e.g., polyester. Preferably, the substrate is 100% cellulosic, more preferably cotton. Furthermore, the substrate can be in a variety of forms, e.g., woven, knitted or yarn. It is a particular advantage of the process of the invention that it can be used for the alkali treatment of knitted goods, more particularly tubular knitted goods. Corduroy is another material for which the alkali treatment of this invention is especially advantageous.

Cellulosic fiber goods treated with a composition or process according to the invention have a smooth appearance with increased luster (gloss), tensile strength, and elongation strength and stability. They are also characterized by their improved dyeability, e.g., with reactive dyes. It has also been observed that the treated goods undergo substantially less shrinkage than is usually experienced in alkali treatments of cellulosic materials wherein the sole or major alkali metal hydroxide component is sodium hydroxide.

Because of the aforementioned reduced shrinkage, it is a further advantage of this invention that the process can be effected without the need for having the goods under tension (except the normal lengthwise tension exerted when the material is drawn between rollers during treatment). Thus, the need for a tenter frame to keep the material under tension is avoided and the process can be employed for the treatment of tubular knitted goods. The reduced shrinkage also makes this process attractive for the treatment of pile-surfaced substrates, such as corduroy.

Examples

Preparation of treatment baths

Alkaline treatment baths are made up as follows:

Half of the final bath volume of soft water at a temp. of 26—33°C is poured into the bath and the required amount of potassium hydroxide is added and stirred. The silicate is then stirred in, followed by the sodium gluconate-sulphonated fatty amide mixture, specifically described above and the wetting agent, sodium 2-ethylhexyl sulphate. Finally, the bath volume is made up to the required amount by the addition of cold soft water.

The composition of each bath is given in Table 1 below.

TABLE 1

	Bath 1	Bath 2	Bath 3
KOH (dry wt.)	200 g/l	200 g/l	250 g/l
Potassium silicate (30%)	75 g/l		75 g/l
Sodium silicate (30%)		75 g/l	
Wetting agent (17%)	10 g/l	10 g/l	20 g/l
Sodium gluconate—sulphonated fatty amide	30 g/l	30 g/l	30 g/l

Example 1

a) A flattened length of tubular knit grey (unbleached) cotton is continuously drawn from a basket through Bath 1 above, which is at approximately room temperature. The immersion time in the bath is 1 to 2 seconds. The wetted substrate is then squeezed between a pair of rollers to a wet pick-up of 90%. During this sequence the only tension on the cotton substrate is that caused by the lengthwise pulling of said substrate from its initial slack position in the basket through the nip of the squeezing rollers at a speed of 9.1 m (10 yards) per minute. The wetted substrate is then fed into a second basket where it is allowed to dwell in a tension-free condition for a period of two hours at room temperature.

b) The above-treated substrate is then washed continuously for 5 to 10 minutes with water at 71—82°C, treated with sufficient aqueous acetic acid to neutralize any residual alkali and washed again briefly with water to remove any excess acetic acid. The substrate is squeezed between rollers to a moisture content of 80% and passed for 5 to 10 seconds through a peroxide saturation bath which typically contains 20—30 g/l caustic soda (100%), 4.5 g/l surfactant, 3—4 g/l chelating agent, 50—70 g/l sodium silicate and 30—50 g/l hydrogen peroxide and which is at a temperature of 48—55°C. The material is again squeezed between rollers to ensure uniform distribution of the peroxide solution and then drawn into a J box where it is subjected to steam at 94 to 100°C for 90 minutes. The substrate is then washed with water at 71 to 82°C to remove any residual peroxide solution components, squeezed through rollers and dried by passage over heated perforated cylinders in conventional manner. During this sequence of steps the substrate is essentially under only that amount of tension created by the rollers drawing it from its tension-free condition in the dwelling basket and in the J box.

Example 2

A flattened length of tubular knit grey cotton is treated as described in paragraphs a) and b) of Example 1, except that it is immersed in Bath 2 instead of Bath 1.

Example 3

A flattened length of tubular knit grey cotton is treated as described in paragraphs a) and b) of Example 1, except that it is immersed in Bath 3 instead of Bath 1.

Comparative Example C1

A substrate identical to that treated in Examples 1 and 2 is treated only according to paragraph b) of Example 1.

Compared to this Sample C1, the shrinkage of the substrates of Examples 1 and 2 is set forth in Table 2.

TABLE 2

	Width	Length
Ex. 1	1%	7%
Ex. 2	2%	3.5%

Comparative Example C2

A substrate identical to that treated in Example 3 is treated only according to paragraph b) of Example 1.

Compared to this Sample C2, the shrinkage of the substrate of Example 3 was 0.6% in width and 8.5% in length.

Affinity for reactive dyes

The substrates treated according to Examples 1 and 2 and C1 were dyed under identical conditions with the same reactive dye by the pad batch method.

Compared to C1 the substrate of Example 1 exhibited 23.7% greater dye affinity and the substrate of Example 2 exhibited 13.9% greater dye affinity.

The substrate treated according to Examples 3 and C2 were dyed under identical conditions with the same reactive dye by the pad batch method. Compared to C2 the substrate of Example 3 exhibited more than 40% greater dye affinity.

Wash shrinkage

The dyed substrates of Examples 1 and 2 and C1 were laundered and tumbled dry under identical conditions. The shrinkage which took place during this treatment is set forth in Table 3.

TABLE 3

	Width	Length	Overall
Ex. 1	8.8%	10%	17.9%
Ex. 2	8.8%	11.3%	19%
Ex. C1	11.3%	8.8%	19%

The dyed substrate of Examples 3 and C2 were laundered and tumbled dry under identical conditions. The shrinkage which took place during this treatment is set forth in Table 4.

TABLE 4

	Width	Length	Overall
Ex. 3	7.8%	8.8%	15.6%
Ex. C2	11.0%	12.2%	21.8%

Example 4

A continuous length of tubular single knit grey cotton jersey is drawn open width at a speed of 45.5 m/min (50 yds./min.) over a series of rollers, through a treatment bath, through a pair of squeeze rollers and onto a perforated roller. During this passage the material is under tension only in a lengthwise direction.

The temperature of the treatment bath is 25°C and its composition is as follows: 270 g/l potassium hydroxide (as 90% flakes), 75 g/l sodium silicate (as a 520 g/l (42° Bé) aqueous solution); 15 g/l of the sodium gluconate-sulphonated fatty acid amide mixture specifically described above; 5 g/l sulphonated castor oil (35% active) and 3 g/l sodium 2-ethylhexyl sulphate (20% active).

A bubble of air is maintained in the length of tubular material between a roller in the treatment bath and the squeeze rollers.

The speed of travel of the material and the pressure of the squeeze rollers in such as to give a wet pick-up of 116%, based on the weight of the material.

The impregnated material is allowed to dwell on the perforated roller for 2 hours at room temperature.

Following the dwelling, the material is treated as in paragraph b) of Example 1,

The thus-treated substrate is characterized by improved luster and dyeability along with very good hand and stretchability.

Example 5

The procedure of Ex is repeated, except that the pick-up is 130% and the treatment liquor comprises: 250 g/l potassium hydroxide (100%); 130 g/l sodium silicate (30%); 15 g/l sodium gluconate-sulphonated fatty acid amide; 7 g/l sulphonated castor oil (35%); and 12 g/l sodium 2-ethylhexyl sulphate (20%).

The resulting knitted cotton substrate has good luster, dyeability, and dimensional stability.

Claims

1. A method for the alkali treatment of a cellulosic woven or knitted substrate which comprises applying to said substrate an aqueous alkali liquor containing potassium hydroxide at an amount of from 200 to 330 g/l and an alkali metal silicate.

2. A method according to Claim 1 in which the amount of alkali metal silicate in the aqueous alkali liquor is in the range 5 to 85 g/l.

3. A method according to any one of Claims 1 or 2, in which the aqueous alkali liquor contains a wetting agent.

4. A method according to any one of the preceding claims in which the aqueous alkali liquor contains a cation-sequestering or -complexing agent and optionally a compound having textile lubricating properties.

5. A method according to any one of the preceding claims in which the substrate consists of cotton or a blend of cotton with polyester.

6. A method according to any one of the preceding claims in which the substrate is knitted goods.

7. A method according to Claim 6 wherein the substrate is in the form of tubular knitted goods.

8. A method according to any one of the preceding claims wherein following application of the aqueous alkali liquor, the substrate is dwelled for a period of time sufficient for the treatment liquor components to act on the substrate and is then washed to remove the alkali metal hydroxide.

9. A method according to Claim 8, wherein during the application, dwelling and washing steps, the substrate is free from tension across its width.

Patentansprüche

1. Ein Verfahren zur Alkalibehandlung eines gewobenen oder gestrickten Zellulosesubstrats, dadurch gekennzeichnet, dass dieses Substrat mit einer wässrigen Alkaliflotte, die von 200 bis 330 g/l Kaliumhydroxid und ein Alkalisilikat enthält, behandelt wird.

2. Ein Verfahren gemäss Anspruch 1, worin die wässrige Alkaliflotte von 5 bis 85 g/l Alkalisilikat enthält.

3. Ein Verfahren gemäss einem der Ansprüche 1 oder 2, worin die wässrige Alkaliflotte ein Netzmittel enthält.

4. Ein Verfahren gemäss einem der vorgehenden Ansprüche, worin die wässrige Alkaliflotte ein Kation sequestrierendes oder komplexierendes Mittel und gegebenenfalls ein Gleitmittel für Textil enthält.

5. Ein Verfahren gemäss einem der vorangehenden Ansprüche, worin das Substrat aus Baumwolle oder aus einem Baumwolle-Polyester-Gemisch besteht.

6. Ein Verfahren gemäss einem der vorangehenden Ansprüche, worin das Substrat aus Maschenware besteht.

7. Ein Verfahren gemäss Anspruch 6, worin das Substrat als schlauchförmige Maschenware vorliegt.

8. Ein Verfahren gemäss einem der vorangehenden Ansprüche, worin man das Substrat nach Applikation der wässrigen Alkaliflotte eine gewisse Zeit, die ausreicht, um die Wirkstoffe der Behandlungsflotte auf das Substrat einwirken zu lassen, verweilen lässt und dann wäscht, um das Alkalihydroxid zu entfernen.

9. Ein Verfahren gemäss Anspruch 8, worin das Substrat während der Applikation, dem Verweilen und dem Waschen spannungsfrei in der Breite ist.

Revendications

1. Un procédé de traitement alcalin d'un substrat cellulosique tissé ou tricoté, caractérisé en ce qu'il comprend l'application au-dit substrat d'un bain alcalin aqueux contenant de l'hydroxyde de potassium en une quantité comprise entre 200 et 330 g/l et un silicate de métal alcalin.

2. Un procédé selon la revendication 1, caractérisé en ce que la quantité de silicate de métal alcalin dans le bain alcalin aqueux est comprise entre 5 et 85 g/l.

3. Un procédé selon l'une quelconque des revendications 1 ou 2, caractérisé en ce que le bain alcalin aqueux contient un agent mouillant.

4. Un procédé selon l'une quelconque des revendications précédentes, caractérisé en ce que le bain alcalin aqueux contient un agent séquestrant ou complexant des cations et éventuellement un composé ayant des propriétés lubrifiantes pour les textiles.

5. Un procédé selon l'une quelconque des revendications précédentes, caractérisé en ce que le substrat est constitué de coton ou d'un mélange de coton et de polyester.

6. Un procédé selon l'une quelconque des revendications précédentes, caractérisé en ce que le substrat est un article tricoté.

7. Un procédé selon la revendication 6, caractérisé en ce que le substrat est sous forme d'articles tricotés tubulaires.

8. Un procédé selon l'une quelconque des revendications précédentes, caractérisé en ce que après l'application du bain alcalin aqueux, on laisse reposer le substrat pendant une période de temps suffisante pour que les composants du bain de traitement agissent sur le substrat et on le lave ensuite pour éliminer l'hydroxyde de métal alcalin.

9. Un procédé selon la revendication 8, caractérisé en ce que pendant les étapes d'application, de repos et de lavage, le substrat est exempt de tension dans le sens de sa largeur.