FIG. 1

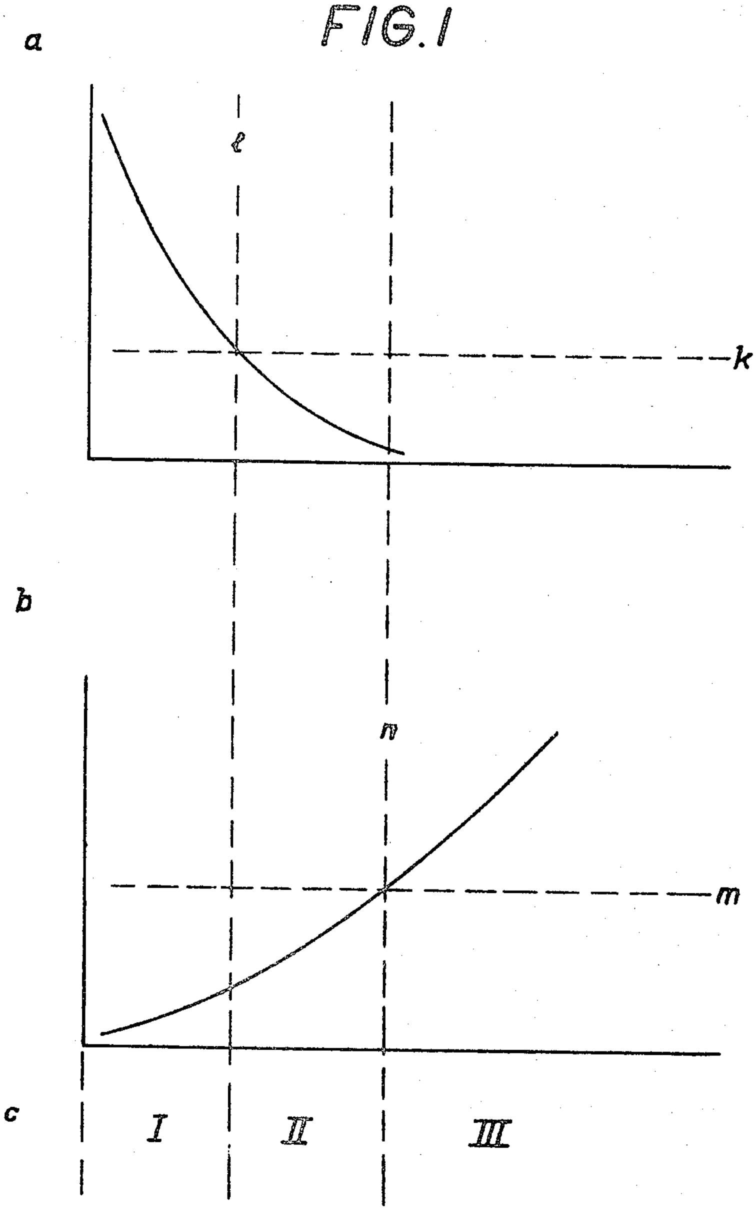

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FIG. 2

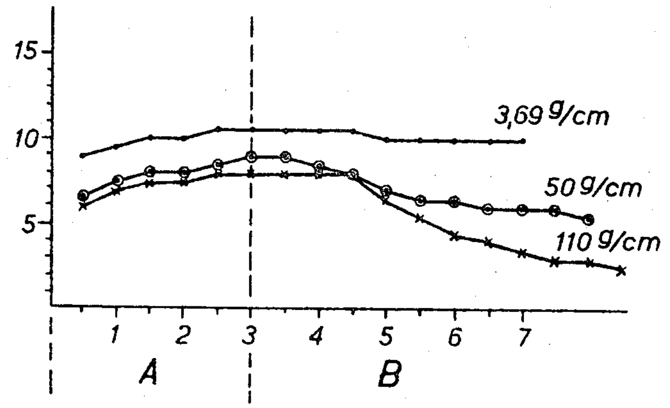

FIG. 3

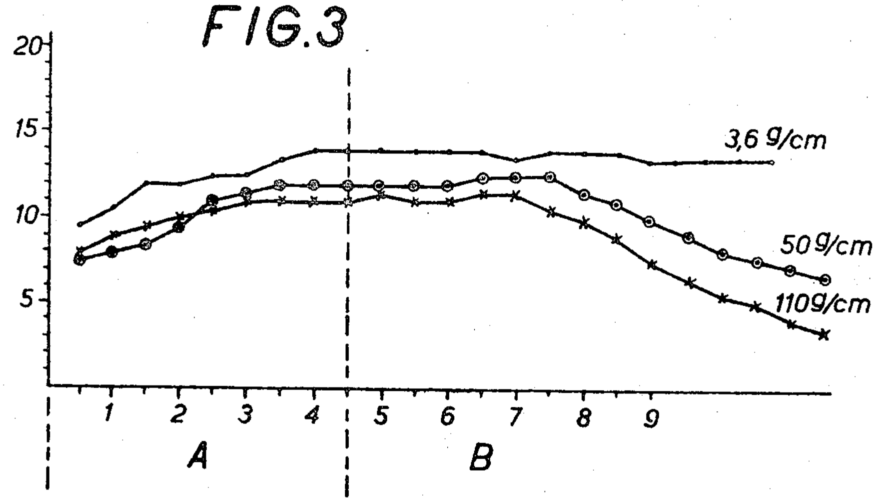

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FIG. 4

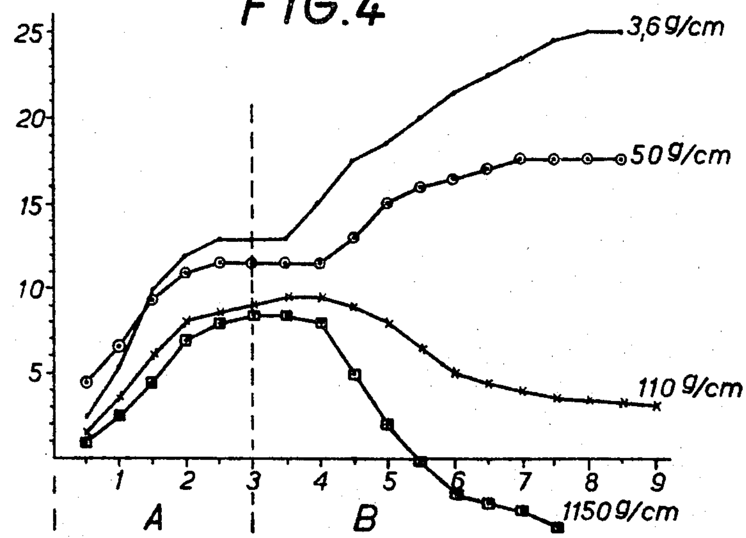
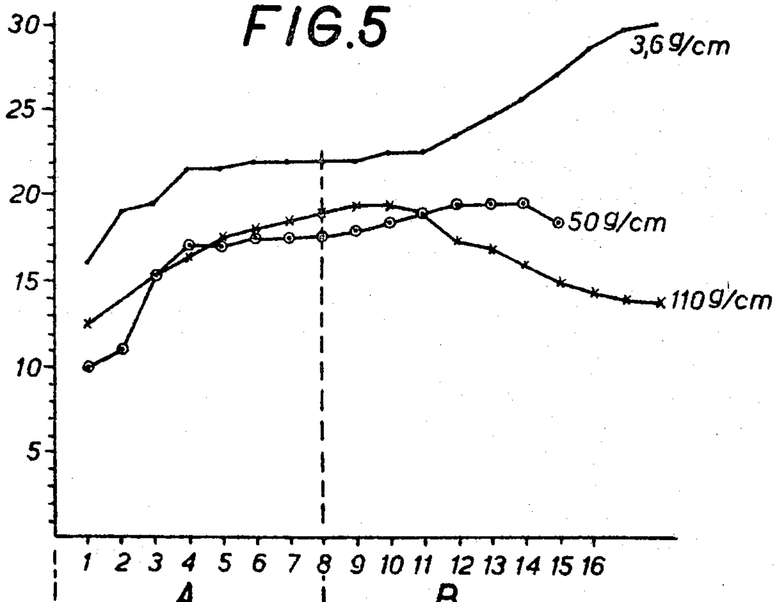


FIG. 5



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United States Patent Office

3,406,006

Patented Oct. 15, 1968

3,406,006

PROCESS FOR THE TREATMENT OF FABRICS CONTAINING CELLULOSE FIBRES WITH LIQUID AMMONIA

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Filed Apr. 23, 1965, Ser. No. 450,299
Claims priority, application Norway, Apr. 25, 1964, 152,995
15 Claims. (Cl. 8—125)

This invention relates to a process for imparting to woven and knitted fabrics which contain natural or regenerated cellulosic fibers a combination of increased extensibility or stretch, resistance to shrinkage and a non-ironing quality by treatment of the fabrics with ammonia. The treatment with ammonia of cellulosic fibers and yarns containing cellulosic fibers to increase their strength is known, and the treatment of woven fabrics of such fibers with ammonia has also been mentioned earlier, e.g. in German DAS No. 1,063,572 and in U.S. Patent No. 1,998,551, but without attaining favorable results obtained according to the present invention.

According to the invention the action of liquid ammonia containing less than 10% of water on cellulosic fibers is utilized in such a way that a process is obtained which in comparison with previously known methods is far more effective and economic in producing fabrics with moderate to high extensibility, shrink resistance and non-iron properties.

It is known that extensibility or stretch can be obtained in several ways. The fibers can be swelled, which leads to a contraction of yarn and fabric. The yarn contraction depends on the degree of twist and the contraction of fabric depends on the type of weave, spacing of warp and filling, and on the yarn number. The only method which is used in practice is slack mercerization, i.e. a treatment with about 25% NaOH at room temperature and subsequent rinsing. This method is not used for ordinary rayon fibers or linen.

Owing to the prevalent techniques (load in machine direction during movement of the fabric) it is extremely difficult to maintain the extensibility in the warp direction in subsequent processes. It is, therefore, customary to only impart extensibility of fabrics in the filling direction. If the fabric is not rendered dimensionally stable by means of a resin treatment, the fabric will shrink in the warp direction during washing.

During the normal finishing process of fabrics containing cellulosic fibers, the fabric material is subjected to repeated stresses in the warp direction. If such a fabric is immersed in water, the fabric is subject to relaxation shrinkage, and if it is washed in a washing machine, the fabric is further subjected to washing shrinkage. In the washing machine the fabric is also subjected to compressive forces, in contrast to what is the case during production. There are several known methods of counteracting this shrinkage, and all are based on the practice that in the last processing stage the fabric is given a mechanical compression in the warp direction.

The expression "non-ironing" signifies that it is not necessary to iron the garment after washing. That is, the fabric dries smooth when it is hung up wet after washing or after being dried in a tumble-drier. In the former case the fabric must both be set in a smooth stage and have a good resistance to wet-creasing. In the latter case the fabric, in addition, must have good resistance to dry-creasing.

It is known that setting in smooth state plus good resistance to wet-creasing can be obtained if the setting is performed when the fibers are swelled, e.g. in water. The

setting can be effected by means of a hydrogen bond breaking agent, e.g. NaOH, at temperatures exceeding 70% C. or salts from the one extreme part of the lyotropic series. It is possible also to use cross-linking agents, such as formaldehyde, epichlorohydrin, dichloropropanol, etc., on the swelled fiber together with an alkaline catalyst. The characteristic feature of this treatment is that the fabric becomes much softer in the wet state after the treatment.

Broadly, the present invention relates to a process for treating fabrics containing cellulosic fibers, such as cotton, linen, hemp, jute or rayon, by mechanical and chemical means. The characteristic feature of the process is that, in order to impart to the fabric a given combination of (a) non-ironing quality, (b) resistance to shrinkage and (c) extensibility, the fabric is impregnated with liquid ammonia; whereby the fibers swell, the ammonia is evaporated, and the fabric during the swelling and/or evaporation:

(A) is kept under a certain load or tension or alternatively a normal pressure which, in order to obtain a material which needs no ironing, is at least sufficient to obtain a fabric which is free from mussiness after the treatment,

(B) is kept under a tension which, in order to obtain a shrink-resistant material, is lower than the tension which results in the maximum allowed shrinkage in the finished fabric conforming with the regulations and norms for washing shrinkage which have to be satisfied,

(C) is kept under a tension which, in order to get maximum extensibility in the warp direction, is maximum in the filling direction and minimum in the warp direction, and in order to get minimum extensibility in the warp direction, is minimum in the filling direction and maximum in the warp direction, and in order to get maximum extensibility in the filling direction, is maximum in the warp direction and minimum in the filling direction, and in order to get minimum extensibility in the filling direction, is minimum in the warp direction and maximum in the filling direction, and in order to get maximum extensibility in both directions, is minimum in both directions, and in order to get minimum extensibility in both directions, is maximum in both directions,

the three said tension conditions being combined during the ammonia treatment. One or two of these tension conditions can, if necessary, be excluded, due regard being paid to the structure of the untreated material and the properties desired for the finished fabric.

By means of the process according to the invention the following primary and secondary properties in the fabric are obtained:

(I) Primary properties:

(1) The commonly occurring relaxation and washing shrinkage in many fabrics of the types mentioned above can be eliminated by means of the present process.

(2) The mussed appearance which a fabric shows after washing is due partly to a relaxation of internal tensions in the fabric during washing and partly to mechanical creasing during washing and centrifuging, or during drying.

The proposed process eliminates entirely or partially the mussiness due to relaxation of the fabric.

(3) A substantially increased extensibility or stretch in the fabrics can be obtained as a result of the shrinkage which occurs under the present treatment.

(II) Secondary properties:

Changes in these properties occur as a result of a sufficiently long action of NH_3 on the fabric without need for the presence of any other special conditions. Among other effects the material becomes much softer in the wet state and, as a result of this, the resistance to washing wear is increased. The fabric shows greater resistance to creasing in the wet state and in consequence of this an improved non-ironing quality in addition to the elimination of mussiness.

The risk of seam-slippage during washing is substantially decreased, owing to the fact that fiber and yarn are set in their wavy form in the fabric.

Similarly to what happens during mercerization, the fabric becomes more resistant to dirt, and a color, if it exists, acquires a deeper shade, provided that the dyestuff is fast toward liquid ammonia.

By a particularly preferred embodiment of the invention the fabric is impregnated and swelled either (I) by being conveyed continuously through a liquid ammonia bath and kept immersed in the bath during the whole time the swelling of the fibers proceeds, or (II) by applying the liquid ammonia, to the extent of at least 50 percent by weight, preferably 50–500% by weight, based on the weight of the untreated material, to the fabric either by immersion in a liquid ammonia bath with or without subsequent compression, or by spraying, after which the swelling is allowed to proceed in a chamber for at least 15 seconds at pressures between 0.5 and 20 atmospheres abs. giving temperatures of the fabric of between -70 and $+50°$ C., lower partial pressure of the superheated ammonia vapor giving lower temperature of the fabric for one and the same total pressure as long as liquid ammonia is present in the fabric. The conditions should be arranged in such a manner that liquid ammonia in all essential respects remains in the material during the swelling, and finally the ammonia is evaporated, viz. by hot air, by overheated ammonia vapor, by pressing the fabric against a hot cylinder with or without the aid of felt, with hot or cold water, or with steam, in the course of which the tension of the fabric in either direction during the swelling and/or the evaporation can be regulated by means of any known technical method. The residue of the ammonia gas in the material after evaporation can be removed by suction with air or by steaming with superheated steam for a few seconds.

If the swelling takes place while the fabric is completely immersed in the ammonia bath, as described under item (I) above, it presents greater technical difficulties to control the tension or dimensions than if the swelling takes place in an ammonia atmosphere, as described under item (II). In general the swelling must proceed for at least 15 seconds, e.g. from 15 seconds to 10 minutes, in order to obtain an acceptable result. This, however, is not critical.

Part of the moisture which normally exists in the fabric can, under certain circumstances, be expelled together with the ammonia. By a direct regeneration of the ammonia this water will quickly accumulate. It presents no difficulty to remove water down to about 2%, which is approximately the same water content as is obtained by a regeneration via a water solution of ammonia. If water accumulates in the liquid ammonia, it is difficult and expensive to get the water content to less than 0.5% by a regeneration procedure. Increasing the water content reduces the effect of the ammonia treatment, and the water content should therefore not exceed 10%.

In general, to obtain a smooth material as the final product (there exists material which by means of constructional variations can be given a regular and desired mussiness e.g. sear-sucker fabrics) it is necessary to take measures which prevent the fabric from obtaining a mussiness during the treatment. Such mussiness appears, e.g. when the fabric is without tension either in water or in liquid ammonia, and the degree of mussiness depends, for the most part, on the type of material and the degree of tension control during spinning and weaving. Mussiness by the present method is prevented in the following way: The fabric must have a certain tension

(or the treatment shrinkage must be stopped at a certain level) in one or both of the directions, either during the swelling stage or the evaporation stage, or both stages, depending on the type of material. The necessary tension is adapted to a level which lies as near as possible to the limit where mussiness is just eliminated. This limit is, as already mentioned, dependent on the type of material and the production control during spinning and weaving. Mussiness can be removed or prevented from forming by passing the fabric between a drying cylinder and a drying felt, or being pressed against the drying cylinder by means of rollers, in which the pressure against the cylinder is sufficient to render the fabric smooth. The degree of mussiness or creasing can be measured by means of a reference standard consisting of photographs of fabrics with different degrees of mussiness.

If the treatment is effected entirely without tension, one will theoretically obtain a fabric which shows no washing shrinkage. The definition of no washing shrinkage is that the dimension of the fabric does not change more than plus or minus an acceptable value. The upper limit for tension (lower limit for treatment shrinkage) which gives an acceptable washing shrinkage, varies from material to material, and can be defined in the following way:

If a non-shrinking material is desired the tension in any of the directions during treatment must not exceed a value which has the effect that a subsequent single washing of the woven materials performed in accordance with a method which is described in a proposed Swedish textile standard, Standard sheet No. 76: "Determination of dimension changes in textile goods," and which is applied and recommended for the types of material in question, will give a washing shrinkage exceeding $\pm 5\%$ in any of the directions, and of knitted materials in accordance with the method which is described in Swedish Standard SIS 650040, affords a washing shrinkage exceeding $\pm 7\%$ in any of the directions.

In FIG. 1 it is shown how the necessary and sufficient tension during the treatment in order to obtain non-ironing quality (FIG. 1*a*) and/or shrink-resistance (1*b*) is determined. If the tension is kept above the tension that is required in order to obtain non-ironing quality and below the tension which must not be exceeded in order to obtain adequate shrink-resistance, a fabric is obtained that has both non-ironing quality and adequate shrink-resistance. Outside the said range there will be obtained only one of the two properties.

FIG. 1*a* shows mussiness of the finished material according to a visual appraisement in points (ordinate) as a function of the tension during the treatment (abscissa). Further, a limit is given for acceptable mussiness (k) and the minimum tension consequent thereof (l).

FIG. 1*b* shows washing shrinkage in percent (ordinate) as a function of the tension (abscissa), and the limit for shrink-resistance according to definition (m) and the maximum tension consequent thereof (n).

FIG. 1*c* shows the effect of the tension on the properties of the finished material, wherein I designates shrink-resistant fabric, II shrink-resistant and non-ironing fabric, and III non-ironing fabric.

In general it may be said that the extensibility (alternative terms used in practice are stretch or elasticity) is directly dependent on the collective shrinkage during swelling and evaporation which is obtained. The degree of extensibility obtained depends on the construction and type of fiber. Maximum treatment shrinkage, and thereby a corresponding extensibility in one direction (1), is obtained if the material during the treatment is given a—within practical limits—maximum tension in the other direction (2). The maximum and minimum tension in any direction will depend on the tension condition(s) to be satisfied with respect to the desired mussiness and/or to the desired resistance to shrinkage. If, for example, maximum extensibility or stretch in the warp direction of the treated material is desired, the tension during the treatment should be minimum in the warp direction and maximum in the filling direction. Maximum treatment shrinkage, and thereby simultaneous extensibility in both directions, is obtained if the treatment is performed without tension in any direction. Intermediate tension during treatment will give a corresponding intermediate extensibility in conformity with the above general rule. All combinations from minimum to maximum extensibility in different directions can be obtained by means of a suitably adapted control of dimension or tension during swelling and/or evaporation. Extensibility is defined as the elongation in percent which a fabric acquires when it is subjected to a load of 350 g./cm. width of material.

As will be seen from Example 2 below, different types of fabric exhibit different behaviour with respect to shrinkage during swelling and evaporation. In the case of each single type of material it is necessary therefore to select the technical procedure which, with a view to control of tension or dimensions, is the most favorable.

The invention is more fully set forth in the following examples which are the presently preferred specific embodiments, it being understood that these are purely illustrative and not for the purpose of limiting the invention.

Example 1

Specimens of a fabric were immersed in liquid ammonia, and were thereafter directly transferred to a new container, where they were allowed to remain for 3 minutes in a NH_3 atmosphere. During this process the material is not subjected to any external form of pressure, i.e., the swelling proceeds free from tension.

The ammonia is removed by pressing the fabric against a hot cylinder surface by means of a cloth which is stretched firmly over the cylinder, thereby fixing the dimensions of the fabric by expulsion of the ammonia. In the case of linen and rayon this may be compared to an ordinary drying under tension. (According to the results given in Example 2, rayon and linen show that the evaporation proceeds under tension).

As test material the following types of fabric are used:

(1) Cotton, sheeting, plain weave.
(2) Cotton/rayon 70/30, dungaree.
(3) Rayon, staple, decoration material.
(4) Linen, bleached canvas, plain weave.

After treatment in liquid ammonia the fabrics possessed the following properties:

(1) *Washing shrinkage.*—The specimens were washed 6 times according to the method given in the specification (washing temp. for cotton and dungaree 85° C., washing temp. for linen and rayon 60° C.). In the case of NH_3-treated and untreated specimens the results are:

	Untreated, washed 6 times		Treated, washed 6 times	
	Warp	Filling	Warp	Filling
Washing shrinkage in percent:				
Cotton, sheeting	10.0	*−1.3	*−0.6	*−4.5
Dungaree	18.3	5.0	1.2	0.7
Rayon, staple	23.3	10.0	4.7	0.0
Linen	19.0	10.0	0.3	3.0

*Signifies expansion.

(2) *Extensibility.*—Measurement of extensibility was performed according to the method given in the specification. The measurements were done on unwashed specimens.

	Untreated		Treated	
	Warp	Filling	Warp	Filling
Extensibility in percent:				
Cotton, sheeting	11.3	12.5	9.0	18.0
Dungaree	3.3	4.0	11.5	5.0
Rayon, staple	3.0	3.5	20.5	1.5
Linen	1.0	0.5	24.0	1.0

(3) *Non-ironing quality.*—The specimens were washed according to the above methods and were evaluated in respect to degree of creasing by comparison with a reference standard (Monsanto creasing standard).

	Untreated	Treated
Dungaree	1	3-4
Rayon, staple	2	4-3
Linen	2	3

Lowest value=1. Best value=5.

Example 2

The specimens were mounted between two clamps. The one clamp was fastened to a rack, while the other clamp was allowed to hang freely to the specimen. To the free clamp were attached different loads, which were constant during the whole test.

The loaded specimen was immersed in liquid ammonia, and the change in length of the specimen was recorded each ½ minute. The treatment continued as long as the specimen showed tendency to change in length. The ammonia was evaporated at room temperature, and the alteration in length was registered in the same way as mentioned earlier.

After the treatment the specimens were washed according to the previous method and the washing shrinkage calculated.

FIGS. 2–5 show the change in dimension percent (ordinate) as a function of the time (minutes, abscissa) spent during treatment (A) and during evaporation (B) at a certain loading in g. per cm. width of material, for cotton, sheeting (FIG. 2), dungaree (FIG. 3), rayon staple (FIG. 4) and linen (FIG. 5). Table 1 shows the shrinkage of treated specimens after a single washing.

TABLE 1

Loading during NH_3 treatment, g./cm.	3.6	50	110
Shrinkage percent, single washing:			
Cotton, sheeting	*−0.6	1.5	3.0
Dungaree	*0	4.7	6.4
Rayon, staple	*−1.4	4.9	13.7
Linen	*−4.5	0.7	3.0

*Gives expansion.

A visual appraisement shows that cotton, sheeting, rayon and linen had a non-acceptable mussiness with the lowest loading, while the surface smoothness was acceptable with a loading of 50 g./cm. in the case of all the specimens.

Example 3

In the first stage the fabrics were treated with ammonia on a frame. This consists of two rollers which can rotate freely. These are fastened to a rack at a distant of about 20 cm. The fabrics were laid round the rollers and sewn together. The purpose of this arrangement was to prevent treatment shrinkage in the longitudinal direction and at the same time obtain maximum treatment shrinkage in the transverse direction. During the whole process the rollers were rotated, so as to obtain uniform shrinkage in the transverse direction. The treatment period was 3 minutes and evaporation was effected at room temperature (15°–30° C.).

For purposes of comparison specimens were treated with NaOH (23%, room temperature 10 minutes), in which the same arrangement was used. After the treatment the fabrics were neutralized, rinsed and dried on the frame. The extensibility in a transverse direction was measured according to the stated method and the results are given in the table below.

In the next stage the treatment was effected in NH_3 and NaOH respectively, without having tension applied to the fabrics any direction. The tests were carried out on different types of cotton fabrics.

(1) Printed calico, plain weave, open fabric.
(2) Sheeting, plain weave, tight fabric.

	Extensibility in percent			
	On frame		Without frame	
	NH_3	NaOH	NH_3	NaOH
Printed calico:				
Longd. direction			24	28
Transv. direction	36	32	33	28
Sheeting:				
Longd. direction			26	24
Transv. direction	31	34	21	22

Having thus disclosed the invention, what is claimed is:

1. In a method of treating woven or knitted fabric containing natural or regenerated cellulosic fibers, the improvement including the steps of (a) impregnating said fabric with liquid ammonia ranging, with respect to water content, from anhydrous liquid ammonia to liquid ammonia containing up to 10% by weight of water, by applying said liquid ammonia to the fabric in an impregnating zone, (b) removing the saturated impregnated fabric to an atmosphere having an ammonia vapor pressure sufficient to retain said liquid ammonia in the fabric until ensuing swelling of the fabric has taken place, (c) removing the ammonia from the swollen fabric, and (d) applying tension to said fabric during at least one of steps (b) and (c), the tension being sufficient to prevent the formation of mussiness in said fabric but being less than the tension which causes a substantial washing shrinkage and being minimum in the direction in which maximum extensibility is desired and maximum in the direction perpendicular thereto, and being minimum in both directions when maximum extensibility in both directions is desired.

2. The improvement according to claim 1, wherein the impregnation is effected by immersion in liquid ammonia.

3. The improvement according to claim 1, wherein tension is applied to the fabric during both steps (b) and (c).

4. The improvement according to claim 1, wherein tension is applied to the fabric during step (b).

5. The improvement according to claim 1, wherein tension is applied to the fabric during step (c).

6. The improvement according to claim 1, wherein the impregnation is effected by spraying on of liquid ammonia.

7. The improvement according to claim 1, wherein the fabric is maintained in said atmosphere for at least 15 seconds and the amount of liquid ammonia retained in the fabric after 15 seconds is at least 50% by weight, based on the weight of the untreated dry fabric, and the total pressure in the impregnating zone is between 0.5 and 20 atmospheres abs.

8. The improvement according to claim 7, wherein the impregnation is effected by immersion in liquid ammonia.

9. The improvement according to claim 7, wherein tension is applied to the fabric during both steps (b) and (c).

10. The improvement according to claim 7, wherein tension is applied to the fabric during step (b).

11. The improvement according to claim 7, wherein tension is applied to the fabric during step (c).

12. In a method of treating woven or knitted fabric containing natural or regenerated cellulosic fibers, the

improvement including the steps of (a) impregnating said fabric with liquid ammonia containing water in an amount insufficient to affect its character as liquid ammonia, (b) removing the saturated impregnated fabric to an atmosphere having an ammonia vapor pressure sufficient to retain said liquid ammonia in the fabric until ensuing swelling of the fabric has taken place, (c) removing the ammonia from the swollen fabric, and (d) applying tension to said fabric during at least one of steps (b) and (c), the tension being sufficient to prevent the formation of mussiness in said fabric but being less than the tension which causes a substantial washing shrinkage and being minimum in the direction in which maximum extensibility is desired and maximum in the directon perpendicular thereto, and being minimum in both directions when maximum extensibility in both directions is desired.

13. The improvement according to claim 12, wherein the fabric is maintained in said atmosphere for at least 15 seconds and the amount of liquid ammonia retained in the fabric after 15 seconds is at least 50% by weight, based on the weight of the untreated dry fabric, and the total pressure in the impregnating zone is between 0.5 and 20 atmospheres abs.

14. In a method of treating woven or knitted fabric containing natural or regenerated cellulosic fibers, the improvement including the steps of (a) impregnating said fabric with liquid ammonia in an impregnating zone, (b) removing the saturated impregnated fabric to an atmosphere having an ammonia vapor pressure sufficient to retain said liquid ammonia in the fabric until ensuing swelling of the fabric has taken place, (c) removing the ammonia from the swollen fabric, and (d) applying tension to said fabric during at least one of steps (b) and (c), then tension being sufficient to prevent the formation of mussiness in said fabric but being less than the tension which causes a substantial washing shrinkage and being minimum in the direction in which maximum extensibility is desired and maximum in the direction perpendicular thereto, and being minimum in both directions when maximum extensibility in both directions is desired.

15. The improvement according to claim 14, wherein the fabric is maintained in said atmosphere for at least 15 seconds and the amount of liquid ammonia retained in the fabric after 15 seconds is at least 50% by weight, based on the weight of the untreated dry fabric, and the total pressure in the impregnating zone is between 0.5 and 20 atmospheres abs.

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