

1

3,116,163

RENDERING POLYVINYL ALCOHOL FIBERS CONTAINING STRUCTURE CHLORINE-FAST

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8 Claims. (Cl. 117—140)

This invention relates to a method of making chlorine-fast sheet structures containing polyvinyl-alcohol fibers.

Many methods for the production of non-woven sheet structures from fibers are known and are in technical use. It is essential thereby that the single fibers adhere sufficiently to one another, which can be brought about by different methods. One of them consists in the use of swellable fibers on the basis of e.g. polyvinyl alcohol per se or in combination with other common non-swellable fibers.

The sheet structures made in this way are not, however, resistant to boiling and chlorine-releasing bleaching agents since the polyvinyl alcohol fibers dissolve in hot water.

It has now surprisingly been found that polyvinyl alcohol containing structures can be made resistant to washing, boiling and chlorine-releasing bleaching agents by after-impregnating non-woven sheet material containing polyvinyl alcohol fibers—the former being made according to any of the known methods—with a mixture of an aminoplast and a polymer containing active radicals, such as —CN, —COOH or NH₂.

Suitable aminoplasts are e.g. di-methylol-thio-urea, di-methylol-dihydroxy-ethylene-urea and trimethylol-melamine. Suitable —CN, —COOH or NH₂ containing polymers are methacrylic acid, acrylic acid, acrylonitrile, acryloamide and copolymers thereof.

When carrying out the present invention under the conditions of practice, the above named components are dissolved in a solvent such as water or alcohol. The solvent should be present in abundance, since concentrated solutions would change the character of the material to be treated. It has been found that 1–5% solutions of the above mentioned compositions cause the desired effect without impairing the appearance, hand, softness etc. of the starting fiber batt. The sheet material containing polyvinyl alcohol fibers is then impregnated with said solution as described in the following examples.

Example 1

A fiber batt weighing 40 grams per square meter consisting of

	Percent
Viscous rayon	90
Watersoluble polyvinyl alcohol fibers	10

is impregnated in a padding mangle with water until a wet pick up of 200 percent is reached, then dried in a felt calender and thereby stabilized.

This stabilized fibrous sheet material is then impregnated in a padding mangle with a solution consisting of

	Percent
Water	97.9
Ammoniumoxalate (catalyst)	0.1
Of a copolymer consisting of 30 parts by weight acrylonitrile, 15 parts by weight methacrylic acid and 55 parts by weight butadiene (this copolymer is commercially available under the trade name "Perbunan")	1.0
Aminoplast (dimethylol-urea)	1.0

The impregnated sheet material is then squeezed off

2

until a wet pick up of 100 percent is obtained, dried at 115° C. on a roller dryer and eventually subjected to a temperature of 150° C. for one and a half minutes in order to convert the condensation resin into the insoluble final state.

Example 2

The same fiber batt as described in Example 1 is impregnated with water in the same way as in Example 1, prestabilized in a felt-calender and then impregnated with a solution consisting of

	Percent
Water	96.5
Trimethylol-melamine	1.5
Copolymer which consists of 50 parts by weight of methacrylic acid butylic ester, 15 parts by weight of methacrylic acid methylester and 35 parts by weight of methacrylic acid (commercially available as "Plextol")	2.0

The further steps are the same as in Example 1.

Example 3

A fiber batt made and prestabilized as described in Example 1 is impregnated with a solution consisting of

	Percent
Methyl alcohol	97.9
Zinc chloride (catalyst)	0.1
Dimethylol-urea	1.0
Acrylic acid	1.0

The further steps are the same as in Example 1. The resulting non-woven fabric is not so soft as that of the preceding examples where the acrylic acid has been replaced by "Perbunan" or "Plextol."

Example 4

A fiber batt made and prestabilized as described in Example 1 is impregnated with a solution consisting of

98.0 percent water
1.0 percent dimethylol-dihydroxy-ethylene-urea
1.0 percent of a copolymer which consists of 70 percent methacrylic acid butylester, 20 percent acrylamide and 10 percent methacrylic acid methylester.

The acryloamide is partially methylated. (The copolymer is being sold as "Acronal.")

The further steps are the same as in Example 1.

Example 5

A fiber batt made and prestabilized as described in Example 1 is impregnated with a solution consisting of

95.7 percent water
0.3 percent aluminum chloride
2.0 percent of a copolymer consisting of equal parts of methacrylic butylester and acrylonitrile.
2.0 percent trimethylolmelamine

The further steps are the same as in Example 1.

What we claim is:

1. A process for rendering non-woven fabric containing water soluble polyvinyl alcohol fibers resistant to the action of hot water and chlorine-releasing agents, which comprises impregnating such non-woven fabric with a 1 to 5% solution of substantially equal amounts of a member selected from the group consisting of dimethylol-urea, trimethylol-melamine, and dimethylol-dihydroxy-ethylene urea and of a member selected from the group consisting of: copolymers of acrylonitrile, methacrylic acid, and butadiene, copolymers of methacrylic acid butylic ester, methacrylic acid methylester, and methacrylic acid, copolymers of methacrylic acid butyl ester, acrylamide, and methacrylic acid methyl ester, copolymers

of methacrylic butyl ester and acrylonitrile and polymers of acrylic acid for a wet pick-up of about 100%, and thereafter subjecting the impregnated fabric to temperatures of about 150° C. whereby the polyvinyl alcohol fibers in said non-woven fabric are modified and said fabric rendered resistant to the action of hot water and chlorine-releasing agents.

2. Process according to claim 1, wherein the solution used in said impregnating contains a catalyst.

3. A process for rendering non-woven fabrics containing water soluble polyvinyl alcohol fibers resistant to the action of hot water and chlorine-releasing agents, which comprises impregnating said non-woven fabric with a 1-5% solution composed of

97.9% water,
0.1% ammoniumoxalate, as catalyst,
1.0% of a copolymer consisting of 30 parts by weight of acrylonitrile, 15 parts by weight of methacrylic acid, and 55 parts by weight of butadiene,
1.0% dimethylol-urea as aminoplast.

4. A process for rendering non-woven fabrics containing water soluble polyvinyl alcohol fibers resistant to the action of hot water and chlorine-releasing agents, which comprises impregnating said non-woven fabric with a 1-5% solution composed of

96.5% water,
1.5% trimethylol-melamine,
2.0% of a copolymer consisting of 50 parts by way of methacrylic acid butylic ester, 15 parts by weight of methacrylic acid methylester, and 35 parts by weight of methacrylic acid.

5. A process for rendering non-woven fabrics containing water soluble polyvinyl alcohol fibers resistant to the action of hot water and chlorine-releasing agents, which comprises impregnating said non-woven fabric with a 1-5% solution composed of

97.9% methyl alcohol,
0.1% zinc chloride, as catalyst,
1.0% dimethylol-urea, and
1.0% acrylic acid.

6. A process for rendering non-woven fabrics containing water soluble polyvinyl alcohol fibers resistant to the action of hot water and chlorine-releasing agents, which

comprises impregnating said non-woven fabric with a 1-5% solution composed of

98% water,
1% dimethylol-dihydroxy-ethylene-urea,

5 1% of a copolymer consisting of 70% methacrylic acid butylester, 20% acryloamide, and 10% methacrylic acid methylester.

7. A process for rendering non-woven fabrics containing water soluble polyvinyl alcohol fibers resistant to the action of hot water and chlorine-releasing agents, which comprises impregnating said non-woven fabric with a 1-5% solution composed of

97.5% water,
15 0.3% aluminum chloride,
2.0% of a copolymer consisting of equal parts of methacrylic butylester and acrylonitrile,
2.0% trimethylol-melamine.

20 8. A washable, high-temperature, chlorine-resistant, non-woven fabric, comprising a fabric containing water-soluble polyvinyl alcohol fibers which has been impregnated with a 1 to 5% solution of substantially equal amounts of a member selected from the group consisting of dimethylol-urea, trimethylol-melamine, and dimethylol dihydroxy ethylene urea, and a member selected from the group consisting of polymers of acrylic acid, copolymers of acrylonitrile, methacrylic acid, and butadiene, copolymers of methacrylic acid butylic ester, methacrylic acid methylester, and methacrylic acid, copolymers of methacrylic acid butyl ester, acrylamide, and methacrylic acid methylester, and copolymers of methacrylic butyl ester, and acrylonitrile for a wet pick-up of about 100%, and thereafter subjected to a temperature of about 150° C. 30 whereby said polyvinyl alcohol fibers in said non-woven fabric are modified and said fabric rendered resistant to the action of hot water and chlorine-releasing agents.

References Cited in the file of this patent

UNITED STATES PATENTS	
2,433,000	Manning ----- Dec. 23, 1947
2,497,117	Dreyfus ----- Feb. 14, 1950
2,719,806	Nottebohm ----- Oct. 4, 1955
2,864,093	Sumner et al. ----- Dec. 16, 1958
2,962,762	Hartmann et al. ----- Dec. 6, 1960
2,973,284	Semegen ----- Feb. 28, 1961

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

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Carl-Ludwig Nottebohm et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 4, line 14, for "97.5%" read -- 95.7% --.

Signed and sealed this 19th day of May 1964.

(SEAL)

Attest:

ERNEST W. SWIDER

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