

1

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**PROCESS AND COMPOSITIONS FOR ENZYMATIC DESIZING AND BLEACHING OF TEXTILES**

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Sodium chlorite is being increasingly used for bleaching textile fabrics. With sodium chlorite as a bleaching agent, it is chlorous acid or chlorine dioxide (chlorine peroxide) which is producing the bleaching effect, chlorine dioxide being formed by acidifying sodium chlorite solution. Chlorine dioxide having only a low degrading action on the starch present in the fabric as a size, it has hitherto been necessary and customary to subject the fabrics to a separate desizing process before bleaching them. Also the improved continuous bleaching process in which chlorite is used has so far required a separate desizing step.

The desizing agents most frequently used are enzymatic products, and enzymes being destroyed by oxidizing substances, the simultaneous use of enzymes in the bleaching liquor seemed impossible.

Now the object of the present invention is a textile finishing process in which desizing and bleaching are effected in one operation. In a process according to the present invention enzymatic desizing agents which are active in a neutral medium at temperatures under 60° C., and substances which split off acid or form an acid only at temperatures over 60° C., are added to the neutral bleaching liquor, the fabric being first impregnated with the cold liquor and then heated up to temperatures where acid is split off or formed, i.e. temperatures above 60° C.

Enzymatic desizing agents suitable for use in a process according to the present invention are, e.g., pancreas amylases, amylases of microorganisms, and mixtures of both. Such amylases of microorganisms are, e.g., the enzymes obtained from stocks of *Bacillus subtilis*, *Bacillus mesentericus*, or *Bacillus thermophilus*, as well as the enzymatic products prepared technically from *Aspergillus oryzae* or *Aspergillus niger*. Said enzymatic agents are not damaged by neutral chlorite solutions and are able to easily decompose starch at temperatures under 60° C., even as low as 30–40° C.

The substances splitting off or forming acid required for initiating the bleaching process, which process according to the present invention is to set in and to be carried through only after a previous desizing of the fabric and at elevated temperatures, may be organic or inorganic compounds. They are, for instance, either salts of weak organic or inorganic bases with strong acids, or alkyl esters capable of splitting off acid at higher temperatures. Examples for such compounds are: ammonium sulfate, ammonium chloride, ammonium nitrate, ethyl lactate, ethyl tartrate, ethyl phthalate. Compounds containing aldehyde groups, such as glucose, are also suitable.

In a process according to the present invention the sized fabric is impregnated at 20–30° C. with a liquor which besides sodium chlorite contains a desizing agent containing as the active ferment pancreas amylase, or amylase of microorganisms, or a mixture of both, and a substance which splits off an acid at higher temperatures. Then the fabric is squeezed until its content of impregnating liquor is reduced to about 100–120% of its

2

weight. In a heating chamber, a heatable hank container or the like, the fabric is then slowly heated to temperatures above 60° C., preferably to temperatures about 75–97° C. By the acid which is gradually set free in this latter range of temperature, the sodium chlorite is caused to exert its bleaching activity. However, before the split-off acid becomes active and while the temperature rises slowly, there is ample time for the desizing agent to degrade the starch present as a size in the fabric to such a degree that the transformation products of the starch can be removed by the subsequent washing and rinsing process.

It may be of advantage to control the heat input in such a way that the textile fabric, after having been impregnated at room temperature with the desizing and bleaching liquor, is first heated to 50–60° C. for a period which, depending on the kind of fabric and size used, may vary between 10 minutes as the minimum and 60 minutes as the approximate maximum, whereupon it is exposed to the temperatures required for bleaching, i.e. approximately 75° C., in particular to temperatures i.e. 95–97° C., suitable for obtaining complete bleaching on an average of one hour's exposure.

Alternatively, the raw fabric may be soaked in the desizing and bleaching liquor warmed to a temperature of about 45–50° C., then left standing for from 10 to 60 minutes while protecting it from heat losses and maintaining the temperature of the impregnated fabric substantially at 45° C. or somewhat higher up to 50° C., whereupon the bleaching process is initiated by heating the impregnated fabric to 75–100° C.

By choosing the right temperature a practically complete bleaching effect is obtained simultaneously with a practically complete desizing. The following table shows the results obtained by the process according to the present invention and illustrates the technical progress achieved.

Strips of a textile fabric were impregnated at a temperature of 30° C. with a liquor containing per liter of water 15 grams of sodium chlorite, 10 grams of an activator, and 5 grams of a wetting agent, then heated for one hour to the temperature indicated in the table, rinsed hot and cold, and finally dried. Subsequently, the degree of desizing and subjective degree of whiteness were measured. As a comparison test, strips of the same textile fabric were treated with the same liquor, to which 5 grams per liter of pancreas amylase had been added.

Temperature in Centigrade	subj. degree of whiteness	degree of desizing
60—without desizing agent.....	84.9	5.0
60—with desizing agent.....	84.1	9.8
70—without desizing agent.....	86.1	5.0
70—with desizing agent.....	86.8	9.0

The degree of desizing was measured by the method described in "Melliand Textilberichte," on pages 1293–1294 of vol. 36 (1955); by said method, grade 10 represents complete absence of starch.

Advantageously, wetting agents are added to the desizing and bleaching liquors. Because of their neutrality towards enzymes, non-ionic products, such as the ones obtained by reacting alkyl phenols with ethylene oxide, are especially suited.

The following examples are inserted for the purpose of illustrating only the present invention. It is not intended to restrict the scope of the invention to what is disclosed in the examples.

*Example I*

A cotton fabric sized with potato starch and weighing about 170 grams per square meter, which has a low content of husks and a calico weave of 24/24, is treated at 20–40° C. with a padding liquor containing, per liter of water,

10 grams of ammonium sulfate,  
5 grams of a non-ionic surface-active agent,  
15 grams of sodium chlorite, and  
10 grams of a pancreas amylase corresponding to 20 amylase units according to Willstätter.

The thus treated fabric is then squeezed until its humidity content is reduced to 100%, and then heated for 60 minutes to 75° C. or, in order to produce full bleach, to 95–98° C. Subsequently, at a temperature of 90° C., the fabric is washed neutral with water or a very dilute soda solution (1 gram of soda per 1 liter of water) and then dried as usual. Almost complete desizing of the fabric and good bleaching effect are the results.

*Example II*

A cotton fabric sized with potato starch and weighing about 170 grams per square meter, which has a low content of husks and a calico weave of 24/24, is treated at 20–40° C. with a padding liquor containing, per liter of water,

10 grams of ammonium nitrate,  
5 grams of a non-ionic surface-active agent,  
15 grams of sodium chlorite, and  
10 grams of a pancreas amylase corresponding to 20 amylase units according to Willstätter.

The fabric is first squeezed until its humidity content is reduced to 100%, and then heated for 60 minutes to 75° C. or, in order to produce full bleach, to 95–98° C. Then, at about 90° C., the fabric is washed neutral with water or a very dilute soda solution (1 gram of soda per 1 liter of water), and finally dried as usual. As in Example I, almost complete desizing of the fabric and a good bleaching effect are the results.

Equally good results are obtained if, instead of ammonium sulfate or ammonium nitrate, the same quantity is used of ethyl lactate or ammonium chloride.

*Example III*

A cotton fabric sized with potato starch and weighing about 170 grams per square meter, which has a low content of husks and a calico weave of 24/24, is treated at 20–40° C. with a padding liquor containing, per liter of water,

10 grams of ammonium sulfate,  
5 grams of non-ionic surface-active agent,  
15 grams of sodium chlorite,  
5 grams of a bacteria amylase corresponding to 2 amylase units according to Willstätter, and  
5 grams of pancreas amylase corresponding to 10 amylase units according to Willstätter.

By squeezing the fabric the humidity content is reduced to 100% and the fabric is then heated for 60 minutes to 75° C. or, for the purpose of producing full bleach, to 95–98° C. Then, at 90° C., the fabric is washed neutral with water or a very dilute soda solution (1 gram of soda per liter of water) and finally dried as usual. Almost complete desizing of the fabric and a good bleaching effect are the results.

*Example IV*

A cotton fabric sized with potato starch, weighing about 170 grams per square meter and having a calico weave of 24/24, is treated at 30° C. with a padding liquor containing, per liter of water,

10 grams of ammonium sulfate,  
5 grams of a non-ionic surface-active wetting agent,

15 grams of sodium chlorite, and  
10 grams of pancreas amylase corresponding to 20 amylase units according to Willstätter.

The thus treated fabric is squeezed until its humidity content is reduced to 100% and then heated first to 50° C. for 20 minutes and subsequently to 95–100° C. for 60 minutes. Thereafter, the fabric is boiled in a very dilute soda solution (1 gram of soda per 1 liter of water) and rinsed hot and cold. Almost complete desizing of the fabric and a good bleaching effect are the results.

Instead of ammonium sulfate, ammonium chloride or ammonium nitrate may be used with the same good results.

*Example V*

A cotton fabric of relief texture, which has been sized with potato starch and weighs about 200 grams per square meter is treated at 40° C. with a padding liquor containing, per liter of water,

10 grams of ammonium sulfate,  
5 grams of a non-ionic surface-active wetting agent,  
20 grams of sodium chlorite, and  
10 grams of bacetria amylase corresponding to 4 amylase units according to Willstätter.

The thus treated fabric is squeezed until its humidity content is reduced to 100%, and then heated first for 20 minutes to 60° C. and then for 60 minutes to 95° C. The fabric is then boiled in a dilute soda solution (2 grams of soda per 1 liter of water) and rinsed hot and cold. Complete desizing and practically complete bleaching effect are the results.

*Example VI*

A cotton fabric of relief texture, which has been sized with potato starch and weighs about 200 grams per square meter, is treated at 30° C. with a padding liquor containing, per liter of water,

10 grams of ammonium sulfate,  
5 grams of a non-ionic surface-active wetting agent,  
20 grams of sodium chlorite,  
5 grams of bacteria amylase corresponding to 2 amylase units according to Willstätter, and  
5 grams of pancreas amylase corresponding to 10 amylase units according to Willstätter.

The thus treated fabric is squeezed until its humidity content is reduced to 100%, whereupon it is heated first for 20 minutes to 50° C. and then for 60 minutes to 95° C. Subsequently, the fabric is washed neutral with a dilute soda solution (1–2 grams of soda per 1 liter of water) of 90° C., and rinsed hot and cold. Complete desizing and practically complete bleaching of the fabric are the results.

*Example VII*

A cotton fabric of about 170 grams per square meter sized with potato starch is impregnated with a bleaching and desizing liquor heated to a temperature of 45–50° C. and containing, per liter of water,

10 grams of ammonium sulfate,  
5 grams of a wetting agent,  
15 grams of sodium chlorite, and  
10 grams of a pancreas amylase corresponding to 20 amylase units according to Willstätter.

The impregnated fabric is squeezed until its humidity content is reduced to 120% of its weight, kept for 30 minutes at said temperature of 45–50° C., and finally heated for one hour to 95–100° C. Subsequently, the fabric is boiled in a soda solution containing 2 grams of sodium carbonate per liter of water and rinsed hot and cold. Complete desizing and almost hundred percent bleaching are the result.

Instead of the cotton fabric referred to in the above examples there may be used for the process of the present

invention any fabric consisting of cellulose or regenerated cellulose, or of mixtures of these with each other or with synthetic fibres, equally good results being obtained.

Instead of potato starch there may be used for sizing these fabrics any other commercial sizing agent, e.g. rice starch, maize starch, or tapioca starch (manioc starch).

What we claim is:

1. In the process of bleaching starch-sized textile fabric with aqueous sodium chlorite liquor, the improvement which comprises supplying the chlorite liquor in neutral condition and containing a concentration of amylase desizing agent sufficient to effectively desize the fabric in a period of from 10 to 60 minutes, the liquor also containing a compound that liberates acid upon being heated to at least 60° C., the acid-liberating compound being in a concentration sufficient to cause the liquor with the liberated acid to effectively bleach the fabric, and first contacting the fabric with the liquor at a temperature between 20° and 60° C. for between 10 and 60 minutes to effect desizing, and then raising the temperature of treatment to above 60° C. to effect bleaching.

2. The process of claim 1 in which the bleaching step is carried out at a temperature of between 75° and 100° C.

3. The process of claim 1 in which the desizing step is carried out at a temperature of between 30° and 55° C.

4. The process of claim 1 in which the desizing step is carried out by gradually raising the temperature of the liquor treatment till it reaches the bleaching temperature.

5. A bleaching liquor in the form of a 1.5 to 2.0% neutral aqueous sodium chlorite solution containing an amylase desizing agent which is effective to desize fabrics in from 10 to 60 minutes at temperatures between 20° and 60° C., said solution also containing in a concentration of approximately 10 grams per liter of water, a member of the class consisting of ethyl lactate, ethyl tartrate, ethyl phthalate and glucose.

6. A bleaching liquor in the form of a neutral aqueous sodium chlorite solution containing an amylase desizing agent in a concentration which is effective to desize fabrics in from 10 to 60 minutes at a temperature range between 20 and 60° C. and also containing an agent that under the influence of temperatures above 60° C. liberates sufficient acid to activate the liquor for bleaching textile fabric.

7. A bleaching liquor in the form of a 1.5 to 2.0% neutral aqueous sodium chlorite solution containing an amylase desizing agent selected from the group consisting of pancreas amylases, the amylases obtained from *Bacillus subtilis*, *Bacillus mesentericus*, *Bacillus thermophilus*, the enzymatic products prepared from *Aspergillus oryzae* and *Aspergillus niger*, as well as an agent that liberates acid under the influence of temperatures above 60° C. the desizing agent being present in a concentration that effectively desizes fabrics in from 10 to 60 minutes at from 20° to 60° C., and the acid liberating agent being present in a concentration that liberates sufficient acid to activate the liquor for bleaching textile fabric.

8. A bleaching liquor according to claim 7 in which the desizing agent is pancreas amylase.

9. A bleaching liquor according to claim 7 in which the desizing agent is amylase obtained from *Bacillus subtilis*.

10. A bleaching liquor according to claim 7 in which the desizing agent is amylase obtained from *Bacillus mesentericus*.

11. A bleaching liquor according to claim 7 in which the desizing agent is amylase obtained from *Bacillus thermophilus*.

12. A bleaching liquor according to claim 7 in which the desizing agent is the enzymatic product prepared from *Aspergillus oryzae*.

13. A bleaching liquor according to claim 7 in which the desizing agent is an enzymatic product prepared from *Aspergillus niger*.

14. A bleaching liquor in the form of a 1.5 to 2.0% neutral aqueous sodium chlorite solution containing an amylase desizing agent which is effective to desize fabrics in from 10 to 60 minutes at temperatures between 20 and 60° C., said solution also containing an ammonium salt of a strong acid in a concentration of approximately 10 grams per liter of water.

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