Abstract: The present invention concerns a method for treating textile material, such as fabrics and yarns. In particular, the invention concerns a method for obtaining a fabric with a "denim look" and the fabric thus obtained. In particular, the present invention concerns a method for treating textile materials, comprising the following operating steps in succession: f) providing a warp made up of a raw yarn; g) coating said raw yarn of the warp with a composition containing a cationizing agent; h) drying and polymerizing said cationizing agent on said raw yarn of the warp; i) sizing said raw yarn of the warp obtained according to step c) through passage in a size composition; j) drying said raw yarn of the warp obtained according to step d).
DESCRIPTION

"METHOD FOR TREATING TEXTILE MATERIALS"

The invention concerns a method for treating textile material, such as fabrics and yarns. In particular, the invention concerns a method for obtaining a fabric with a "denim look" and the fabric thus obtained.

By the term "denim look" we mean an appearance similar to that of denim fabric used in making jeans and that behaves similarly with regard to wear.

Denim fabric is normally woven using a warp dyed with the typical colours of jeans, in particular the colour indigo, and a weft of raw yarn. This weaving process gives the typical denim look, as well as the known properties of discoloration that give garments made from such a fabric a worn look.

Although this fabric is among the most common in the world, the method for obtaining it has various drawbacks.

Firstly, it foresees the use of totally dedicated plants, essentially due to the predyeing of the warp that limits the subsequent use of conventional production lines. Therefore, dyeing batches must necessarily be very large (about 10,000 metres). An indirect consequence is low flexibility
of the method, so that the colours normally used are limited to indigo and sulphur. Indeed, every colour variant requires the repetition of all of the steps of the method, since the dyeing happens at the start of the process.

Due to the above, it is clear that plants for producing denim fabric are located in large dedicated factories, with substantial energy consumption and great environmental impact. Indeed, large quantities of polluting chemical compounds (caustic soda, hydrosulphites, sodium chloride) are used, which remain in the wastewater, together with the hydrolysed dye of the dyeing baths. Also in the washing step after dyeing, large volumes of water polluted by the high discharge of dye are created.

The energy consumption of these plants is very high, due to the large volumes involved and the high temperatures (90°C for pretreatment and 70°C for dyeing).

Moreover, during weaving the indigo powder released by the warp represents a great risk factor to the health of workers, due to the generation of micro-powders.

Further drawbacks are due on the one hand to the weakening of the denim that occurs following
treatments on the garment (washings, stone washing, chlorination, etc.), and on the other hand to the low solidity of dyeing by wet rubbing, which runs the risk of dirtying the surfaces that come into contact with a denim garment worn while wet.

Moreover, the method described above can only be used for cotton yarns, thus excluding all other fibres that could offer interesting functional and aesthetic characteristics.

An object of the present invention is therefore to provide a method that is more flexible, economically advantageous and environmentally sustainable, which makes it possible to obtain fabrics with a denim look in a vast range of colours.

A further object is to provide a fabric with a denim look that provides the typical effect of discolouration by wearing of the garment, but not by wet rubbing and that at the same time has high resistance to structural weakening of the fabric itself.

The method of the invention foresees a cationizing treatment of the surface of the yarn that constitutes the warp, as will be better described hereafter.
The idea of chemically modifying the surface of a thread or of a fabric by means of cationic charges able to bind with anionic charges typical of acidic and/or reactive dyes is known and has been applied mainly in the following processes:

i) Application of cationic resins on flock fibres with exhaustion processes in an autoclave; this method makes it possible to impregnate the entire fibre and therefore to subsequently dye the entire mass of yarn;

ii) Cationization of unwoven fabric by "colour catcher" effects with exhaustion and/or foulard applications: this method is dedicated to the production of sheets with "dye-scavenger" properties that can be used when washing dyed and dye-free garments in washing machines;

iii) Application of cationic resins carried out directly on the garment: in this case the cationization of the entire fabric is obtained, with consequent homogeneous dyeing of the garment.

The international application published with number WO 20087115685 describes the personalised dyeing of a garment obtained from a fabric totally or partially made up of a cationic cotton yarn. However, the method for manufacturing said cationic
cotton is not described, which leads us to think of using the conventional cationization of the flock fibre.

The problem forming the basis of the present invention is therefore that of overcoming entirely or partially the drawbacks discussed above of the conventional process for manufacturing denim fabric.

Such a problem is solved by a process and by a yarn and a fabric obtainable by such a process, as outlined in the attached claims, the definitions of which form an integral part of the present description.

Further characteristics and advantages of the invention will become clearer from the description of some example embodiments, given hereafter for indicating and not limiting purposes, with reference to the following figures:

Figure 1 represents a schematic view of a plant for implementing the method of the invention;

Figure 2 represents a schematic cross section view of the yarn for warp obtainable with the method of the invention.

As shown in figure 1, the plant, wholly indicated with reference numeral 1, is totally conventional and comprises:
- a first treatment (cationization) section 2, for example consisting of a foulard in which the warp is made to pass at a predetermined temperature for a predetermined time,

- a subsequent drying and polymerisation section 3, in which the warp in outlet from the first treatment section is made to pass between cylinders 6, preferably Teflon-coated cylinders at a first predetermined temperature and pressure,

- a second treatment (sizing) section 4, for example consisting of a foulard in which the cationized and polymerised warp is made to pass at a predetermined temperature for a predetermined time,

- a subsequent drying section 5, in which the warp in outlet from the second treatment section is made to pass between cylinders 7, preferably Teflon-coated cylinders, at a second predetermined temperature and pressure.

An analogous plant (not shown) can comprise the drying sections 3, 5 in parallel instead of in series. In this case, both of the treatment sections 2, 4 will be used alternately for cationizing or for sizing, thus carrying out a double passage of the warp in the plant or the arrangement of a second twin plant downstream of the first. This solution is
preferable when the warp is made up of a number of threads greater than 10,000-15,000.

The method of the invention comprises the following operating steps in succession:

a) providing a warp made up of a raw yarn;

b) coating said raw yarn of the warp with a composition containing a cationizing agent;

c) drying and polymerising said cationizing agent on said raw yarn of the warp;

d) sizing said raw yarn of the warp obtained according to step c);

e) drying said raw yarn of the warp obtained according to step d).

Step a) of providing a warp of a raw yarn comprises the arrangement of said raw yarn on conventional beams 8 with from 4000 to 15,000 threads.

Said raw yarn is selected from a yarn of cotton, wool, flax, synthetic or mixed fibres. The raw yarn can be obtained according to conventional techniques, which will not therefore be described here in detail.

Step b) of coating the raw yarn with a cationizing agent comprises passing the raw yarn of the warp in said first treatment section 2. As
shown in figure 1, such a passage is advantageously carried out in a conventional foulard containing an aqueous composition of a cationizing agent.

The aqueous composition of a cationizing agent according to the invention comprises:

1) a cationizing agent,
2) a hot-polymerisable resin,
3) ammonium sulfate,
4) a non-ionic wetting agent.

In a preferred embodiment of the method, the aqueous composition of the invention has the following composition: 1) cationizing agent 80-120 g/L; 2) hot-polymerizable resin 50-70 g/L; 3) ammonium sulphate 7-9 g/L; 4) non-ionic wetting agent 2-4 g/L.

In an embodiment, the cationizing agent is an aliphatic amine quaternary ammonium salt, for example Reapret MDZ from the company Giovanni Bozzetto S.p.A..

In an embodiment, the hot-polymerizable resin is a melamine/formaldehyde based resin, for example Reacel MFC from the company Giovanni Bozzetto S.p.A..

In an embodiment, the non-ionic wetting agent is
a mixture of polyglycol ethers of fatty acids, for example Finbil 3001 from the company Giovanni Bozzetto S.p.A.
The ammonium sulphate has the function of catalysing the polymerisation of the resin and of regulating the pH of the solution.
The hot-polymerizable resin coats and homogeneously forms a film over the surface of every single thread of warp and prevents the cationizing agent from penetrating deep. The non-ionic wetting agent regulates the wettability of the raw yarn and, when used in the quantities indicated above, it allows the surface coating of the yarn, preventing total impregnation thereof. This effect is also obtained thanks to the viscosity of the solution thus obtained and to the process parameters as will be defined herebelow.

The fact that the cationizing agent is only present on the surface of the raw yarn is essential for the purposes of the present invention, since, in the subsequent dyeing step, it results in dyeing of just the surface.

According to an embodiment, step b) of coating the raw yarn of the warp with said composition of
cationizing agent is carried out at temperatures of between 30°C and 40°C and at a pH of about 5, at a speed of 20-50 m/min and with a degree of pressing such as to obtain a pick-up of 60-80%.

The concentration of the reactants, the viscosity of the solution and the pressing pressure that eliminates the excess of cationizing composition on the yarn make it possible to not exceed the physical saturation limit on the treated surface. A possible excess of resin, indeed, could complex the dye in the bath of the subsequent dyeing step, thus obtaining an opposite effect to the desired one of fixing the anionic dye.

The subsequent step c) of drying and polymerising foresees a treatment temperature of about 140°C and a contact time of about 30-50 seconds, with speeds within the range of 20-36 m/min, which leads to the formation of a thin and uniform film of polymerised resin on the surface of the yarn, which incorporates the cationizing agent. The temperature can nevertheless be regulated as a function of the polymerisation temperature of the resin used and therefore it can vary with respect to the temperature given here. In general, temperatures over 100°C will be necessary to
obtain the polymerisation of the resin.
The yarn thus obtained preferably has a residual humidity equal to 3-4%.
The surface layer of resin also gives the yarn strength and elasticity.

Step d) of sizing makes it possible to obtain high frame yields (80-90%) and to avoid pilling effects. The sizing is obtained by making the yarn of the warp, previously treated according to steps b) and c), pass in the second treatment section 4, in particular in a foulard in which there is a size composition.
The size composition is selected so as to be compatible with the cationizing agent previously deposited on the yarn and it is preferably devoid of anti-static agents.

According to an embodiment of the invention, a sizing composition comprises a mixture of polyvinyl alcohols (PVA) and of polysaccharides.
For example, the commercial product Larotex OL S from the firm Laros can be used. The percentage in weight of the size in the composition is between 8 and 10% of the bath volume necessary.
The composition of the size does not vary depending on the characteristics of the warp,
such as to variable count of between Ne 9/1 and Ne 40/1 and a number of threads of between 4000 and 10,000, as does happen for conventional sizes.

This composition of the size makes it possible to combine the advantages typical of PVA such as the formation of an elastic film, a good hand, a low influence of humidity, with the advantages of polysaccharides, such as good sizing properties, good behaviour with regard to melting and low sensitivity to alkaline conditions.

The size is firstly cooked at 90°C according to a conventional method, then is cooled to less than 70°C and preferably between 55 and 65°C or to about 60°C and kept at this temperature during the passage of the yarn to be treated. Indeed, higher temperatures could lead to the removal at least of part of the cationized resin.

The yarn is passed through the sizing section at speeds of 20-50 m/min and is pressed with an application refractometric degree of about 12 and residual load on the yarn of 6-8% by weight.

The warp yarn thus treated is then dried (step d) in the drying section 5 and then wound in a yarn beam 9 of the conventional type.
In an embodiment, the method of the invention also comprises at least one of the following steps in succession:

f) loom weaving the yarn of the warp obtained from step e) with a raw yarn weft;

g) dyeing the yarn obtained from step f) with anionic dyes;

h) after-treatment of the dyed fabric obtained from step g).

Step f) of weaving can be carried out using conventional wefts, according to the aesthetic effect that it is wished to give to the fabric. Step g) of dyeing is preferably carried out without removal of the size and without pre-dyeing scouring.

In an embodiment, step g) of dyeing foresees an exhaust dyeing or a pad-batch using acid, preferably pre-metalized, or reactive in acid bath.

The dyes must be pre-selected as a function both of the solidity to light of the dye-resin substrate, and of the dimensions of the dye molecule.

The dyeing is carried out through the salification by the negative ions of the dye of
the cationic groups of the cationizing agent that coats the yarn of the warp.
Step g) of dyeing is carried out in absence of electrolytes (for example sodium chloride) and in absence of fixing with alkali. In this way, there is just dyeing of the warp, whereas the weft does not catch the dye and stays raw. The dye remains fixed just one the warp, thanks to the presence of the cationizing agent.

Step h) of after-treatment can comprise singeing and/or mild bleaching with hydrogen peroxide. It is suitable for such operations not to be carried out before step g) of dyeing. All of the conventional cotton finishing treatments are also possible, since the fabric obtained according to the method of the invention is stable in high temperature treatments (boiling) and in highly alkaline treatments (mercerization).

The treatments on the garment obtained with the fabric according to the invention, such as "chemical polishing" with enzymes, "stone washing" or treatments with chlorine or permanganates, lead to the desired effects of discolouration by progressive removal of the colour on the surface of the fibre that degrades
to the natural raw colour of the inner mass of
the fibre, demonstrating that the method of the
invention actually results in just surface dyeing
of the yarn of the warp.

A further object of the present invention is a
yarn 100, the structure of which is shown in
figure 2, which comprises:
i) a core 101 of raw yarn;
ii) a layer 102 comprising a resin incorporating
a cationizing agent;
iii) an outer layer 103 comprising a size;
iv) optionally, a dye applied to the outer
surface of the yarn 100 through said cationizing
agent.

In an embodiment, the core 101 of raw yarn is
made from cotton, wool, flax, synthetic or mixed
yarns.

In an embodiment, the cationizing agent is an
aliphatic amine quaternary ammonium salt, for
example Reapret MDZ from the company Giovanni
Bozzetto S.p.A..

In an embodiment, the hot-polymerizable resin is
a melamine/formaldehyde based resin, for example
Reacel MFC from the company Giovanni Bozzetto
S.p.A.
According to an embodiment of the invention, the size comprises a mixture of polyvinyl alcohols (PVA) and of polysaccharides. In an embodiment, the dye is an acidic anionic dye.

A further object of the invention is a fabric with orthogonal structure, comprising a warp and a weft, in which said warp consists of a yarn 100 comprising:

1) a core 101 of raw yarn;
2) a layer 102 comprising a resin incorporating a cationizing agent;
3) an outer layer 103 comprising a size, and said weft consists of a raw yarn, and wherein said warp comprises a surface dye with an anionic dye.

A further object of the invention are the manufactured products obtained with the fabric of the invention or comprising a yarn as defined above. For example, such manufactured products can be garments, accessories (bags, wallets, belts, etc.), furnishing elements or other.

The advantages of the present invention are many. Firstly, the fact that the dye is applied to the raw fabric instead of to the warp — as in the
conventional method for a denim fabric - means that conventional plants can be used. It is possible to use dyeing batches typical of a pad batch or exhaust dye (from 50 to 4000 m), without the need to work with large batches (10,000 m) like in the conventional denim method.

A further consequence is that a wide range of variants and colour tones can be obtained, as a consequence of the use of reactive dyes and/or brilliant acids, which makes the inventive method very flexible. This also makes it simpler to carry out sampling with small amounts of fabric. The environmental and energy advantages should also not be forgotten. The process of the invention avoids the use, in the dyeing step, of caustic soda and sodium chloride, which normally require a treatment of the waste water. Moreover, for exhaust dyes the process exhausts the dye in the dyeing bath and therefore the polluting load is minimal. Also for pad batch dyes the amounts of dye are less than 15-25 g/L. The first washing water is also almost dye-free, since it is strongly bound to the cationic groups of the yarn. The volumes of treatment water are in any case much smaller with respect to the
conventional denim process. The process times are reduced by about 50-60%, at room temperature, with respect to the conventional method and by about 40% at a temperature below 70°C.

The weaving takes place without the presence of dyes and the powder is generated by just the sizing products used in conventional low environmental-impact weaving.

With the method of the invention, the treatments on the garment provide the same effects of discolouration of the conventional denim fabric, but the resistance of the garment over time is greater thanks to the presence of the dyed cationized resin that is progressively removed in the mechanical treatments, preserving the yarn beneath.

The binding of the dye to the cationic groups of the warp is strong, which means high solidity against rubbing and reduces the release of dye while wet. The resistance to alkali is also very high.

The use of yarns made from various raw materials, both natural and synthetic, increases the flexibility of the method.
It is clear that only some particular embodiments of the present invention have been described, to which the man skilled in the art will be able to make all those modifications that are necessary to adapt it to particular applications, without however departing from the scope of protection of the present invention.
CLAIMS

1. Method for treating textile materials, comprising the following operating steps in succession:
   a) providing a warp made up of a raw yarn;
   b) coating said raw yarn of the warp with a composition containing a cationizing agent;
   c) drying and polymerising said cationizing agent on said raw yarn of the warp;
   d) sizing said raw yarn of the warp obtained according to step c) through passage in a size composition;
   e) drying said raw yarn of the warp obtained according to step d).

2. Method according to claim 1, wherein said step a) of providing a warp of raw yarn comprises the arrangement of said raw yarn on 4000 to 15000 yarn beams.

3. Method according to claim 1 or 2, wherein said raw yarn is selected from among a cotton, wool, flax, synthetic or mixed fibres yarn.

4. Method according to any one of claims 1 to 3, wherein said method is carried out in a plant (1) comprising:
   - a first treatment section (2), preferably
constituted by a foulard wherein the warp is passed at a predetermined temperature and period of time,
- a subsequent section for drying and polymerizing (3), wherein the warp exiting from the first treatment section is passed between cylinders (6), preferably teflon-coated cylinders, at first predetermined temperature and pressure,
- a second treatment section (4), preferably constituted by a foulard wherein the cationized and polymerized warp is passed at a predetermined temperature and period of time,
- a subsequent drying section (5), wherein the warp exiting from the second treatment section is passed between cylinders (7), preferably teflon-coated cylinders, at second predetermined temperature and pressure.

5. Method according to claim 4, wherein said step b) of coating the raw yarn with a cationizing agent comprises the passage of the raw yarn of the warp in said first treatment section (2) containing an aqueous composition of a cationizing agent.

6. Method according to any one of claims 1 to 5, wherein said aqueous composition of cationizing agent comprises:

1) a cationizing agent,
2) a hot-polymerizable resin,
3) ammonium sulfate,
4) a non-ionic wetting agent.

7. Method according to claim 6, wherein said aqueous composition has the following composition: 1) cationizing agent 80-120 g/L; 2) hot-polymerizable resin 50-70 g/L; 3) ammonium sulphate 7-9 g/L; 4) non-ionic wetting agent 2-4 g/L.

8. Method according to claim 6 or 7, wherein said cationizing agent is an aliphatic amine quaternary ammonium salt.

9. Method according to any one of claims 6 to 8, wherein said hot-polymerisable resin is a melamine/formaldehyde based resin.

10. Method according to any one of claims 6 to 9, wherein said non-ionic wetting agent is a mixture of polyglycol ethers of fatty acids.

11. Method according to any one of claims 1 to 10, wherein said step b) of coating the raw yarn of the warp with said composition of cationizing agent is carried out at temperatures comprised between 30°C and 40°C and a pH of about 5, at a speed of 20-50 m/min and with a pressing degree such as to obtain a 60-80% pick-up.

12. Method according to any one of claims 1 to 11,
wherein said step c) of drying and polymerising provides for a treating temperature of about 140°C and a contact period of about 30-50 seconds with speed in the range of 20-36 m/min.

13. Method according to claim 12, wherein the yarn obtained according to step c) has a residue moisture equivalent to 3-4%.

14. Method according to any one of claims 4 to 13, wherein said sizing step d) comprises the passage of said yarn of the warp, previously treated according to steps b) and c), in said second treatment section (4) wherein there is a size composition.

15. Method according to any one of claims 1 to 14, wherein said size composition comprises a mixture of polyvinyl alcohols (PVA) and polysaccharides and it is preferably free of antistatic agents.

16. Method according to claim 15, wherein the percentage in weight of the size in the composition is comprised between 8% and 10% of the required bath volume.

17. Method according to any one of claims 1 to 16, wherein said size is first baked at 90°C, then it is cooled to below 70°C or between 55 and 65°C or to about 60°C and maintained at this temperature during the passage of the yarn to be treated.
18. Method according to any one of claims 1 to 17, wherein said yarn is passed through the size composition at a speed of 20-50 m/min and it is pressed with an application refractometric degree of about 12 and residual load on the yarn of 6-8% in weight.

19. Method according to any one of claims 1 to 18, further comprising at least one of the following steps in succession:

f) loom weaving the yarn of the warp obtained from step e) with a raw yarn weft;

g) dyeing the fabric obtained from step f) using anionic dyes;

h) after-treatment of the dyed fabric obtained from step g).

20. Method according to claim 19, wherein said dyeing step g) is conducted without removing size and without pre-dyeing scouring.

21. Method according to claim 19 or 20, wherein said dyeing step g) provides an exhaust dyeing or a pad-batch using acid, preferably pre-metalized or reactive in acid bath, dyes, in absence of electrolytes and application with alkali.

22. Method according to any one of claims 19 to 21, wherein said after-treatment step h) comprises one
or more of the following operations: singeing, mild bleaching with hydrogen peroxide, mercerization, chemical polishing using enzymes, stone washing, treatment with chlorine or permanganates.

23. Yarn (100), comprising:
   i) a core (101) of raw yarn;
   ii) a layer (102) comprising a resin incorporating a cationizing agent;
   iii) an outer layer (103) comprising a size;
   iv) optionally, a dye applied to the outer surface of said yarn (100) through said cationizing agent.

24. Yarn (100) according to claim 23, wherein said core (101) of raw yarn is made of cotton, wool, flax, synthetic or mixed yarns.

25. Yarn (100) according to claim 23 or 24, wherein said cationizing agent is an aliphatic amine quaternary ammonium salt.

26. Yarn (100) according to any one of claims 23 to 25, wherein said hot-polymerisable resin is a melamine/formaldehyde based resin.

27. Yarn (100) according to any one of claims 23 to 26, wherein said size comprises a mixture of polyvinyl alcohols (PVA) and polysaccharides.

28. Yarn (100) according to any one of claims 23 to 27, wherein said dye is an acid anionic dye.
29. Fabric with orthogonal structure, comprising a warp and weft, wherein said warp is constituted by a yarn (100) as defined in any one of claims 23 to 28, which comprises:

i) a core (101) of raw yarn;

ii) a layer (102) comprising a resin incorporating a cationizing agent;

iii) an outer layer (103) comprising a size, wherein said weft is constituted by a raw yarn, and wherein said warp comprises a surface dyeing with an anionic dye.

30. Products obtained using a textile as defined in claim 29 or comprising a yarn as defined in any one of claims 23 to 28.
# INTERNATIONAL SEARCH REPORT

## A. CLASSIFICATION OF SUBJECT MATTER

- INV. D06P1/56
- D06P1/66
- D06P3/854
- D06P5/22
- D06B21/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

- Minimum documentation searched (classification system followed by classification symbols)
  - D06P
  - D06B
  - D06M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Further documents are listed in the continuation of Box C. See patent family annex.

* Special categories of cited documents:
  - "A" document defining the general state of the art which is not considered to be of particular relevance
  - "E" earlier document but published on or after the international filing date
  - "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
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### Date of the actual completion of the international search
10 April 2012

### Date of mailing of the international search report
16/04/2012

Name and mailing address of the ISA/
- European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk
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