(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 0 911 441 A1

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
28.04.1999 Bulletin 1999/17

(51) Int Cl.[6]: **D06M 16/00**

(21) Application number: **98660109.4**

(22) Date of filing: **27.10.1998**

(84) Designated Contracting States:
AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE
Designated Extension States:
AL LT LV MK RO SI

(30) Priority: **27.10.1997 FI 974067**

(71) Applicant: **Röhm Enzyme Finland Oy**
05201 Rajamäki (FI)

(72) Inventors:
- **Miettinen-Oinonen, Arja**
  01800 Klaukkala (FI)
- **Puolakka, Arja**
  33250 Tampere (FI)

(74) Representative: **Knuth-Lehtola, Sisko et al**
Seppo Laine Oy,
Itämerenkatu 3 B
00180 Helsinki (FI)

(54) Process for cellulase treatment

(57) The present invention relates to cellulase-treated spun-yarn, which has reduced hairiness, decreased yarn number and/or improved evenness. This invention relates also to fabric or knit prepared from the cellulase-treated spun yarn. According to the invention the cellulase treatment step is carried out after spinning, pretreatment, bleaching or dyeing.

EP 0 911 441 A1

Description

[0001] The present invention relates to a process for treating yarn by cellulase enzymes, to the treated yarn and to the treated fabric prepared from said yarn. The present invention also relates to a method for reducing hairiness, decreasing the yarn number, improving the evenness of yarn and/ or decreasing the dustiness of yarn.

[0002] Cotton is mainly spun by two technically different methods: ring spinning and open-end spinning (rotor spinning), Open-end spinning is more common in Europe, whereas ring spinning is more common in Asia, Ring-spun yarn can be combed yarn or carded yarn, open-end spun yarn can only be carded yarn, Other spinning methods are available, but they are not used as often as the above-mentioned spinning methods.

[0003] Cotton yarn production usually begins with a cotton card, The card slivers are stretched in stretching machines in several steps, If combed yarn is produced, the slivers are combed to remove short hairs protruding from the yarn and to straighten the fibers.

[0004] If yarn is produced by using open-end spinning, stretch slivers are spun after the stretching steps by using open-end spinning, Open-end spinning does not contain any rowing phase.

[0005] In open-end spinning shorter cotton fibers can be used than in ring spinning, The structure and form of open-end spun yarn is different from ring-spun yarn, The twist is not as clear as that of ring-spun yarn, Because of its structure the open-end spun yarn is hairier and visually dull, Short-fibered cotton cannot be used for textile products having a glossy surface, In dyed yarn products the tone of the colours is not "rich enough" for the eye, The surface of the fabric easily becomes pilled and fuzzy, The yarn is stiff and lacks elasticity, Products made of open-end spun yarn easily become wrinkled, The surface of pile products such as velvet is fuzzy, Open-end spun yarn is 15 to 25 % cheaper, but its scope of application is restricted, In knitting it can merely be used as fleecy yarn, Open-end spun yarn is too stiff and hard to be used in preparing high quality knitwear.

[0006] The evenness of cellulosic yarn is a problem that depends on the raw material of the yarn and on the spinning method as discussed above, The unevenness causing hairs and neps on the surface of ring-spun yarn is usually removed by combing the slivers before spinning, Combing is not used to improve the evenness of open-end spun yarn, The scope of application is merely chosen to be suitable for the quality of the yarn.

[0007] Cellulase treatment of cellulose-containing fabric during their manufacture or finishing is known *per se* in the art, The enzymatic treatment for the finishing of cellulose-containing fabric is called biofinishing, Biofinishing has been used e.g, to remove all kinds of impurities and individual loose fibre ends that protrude from the textile surface, The key benefits offered by biofinishing with cellulases lie in the permanent improval of depilling, a cleared surface structure due to reduced fuzz, improved textile handle, such as softness, smoothness and a silkier feel, improved drapability of the textile and improved moisture absorbability.

[0008] Cellulases have been used to impart a stonewashed appearance to denims, Stone wash has traditionally been performed using so-called pumice stones, However, the use of pumice stones causes laundries several problems, such as the heaviness of the stones when handled, the laborious picking by hand of the stones from among the garments, significant machine wear with resulting high repair and investment costs, the growing amounts of waste caused by broken stones and, additionally, the complicated access to pumice stones, as the mining of pumice stones is forbidden in certain countries due to environmental reasons.

[0009] The complete biodegradation of cellulases is an advantage of the cellulase treatment which consequently stands out as an environmentally friendly alternative to chemical treatment.

[0010] There are several publications which suggest and disclose methods for treating cotton fabrics, prior to dyeing and finishing, with a cellulase solution in order to remove lint and loose surface fibres to impart a better appearance, for example US Patent No, 5,232,851. The employed cellulase can be produced, for example, by the species *Trichoderma reesei, T. koningii, Penicillium sp* or *Humicola insolens.*

[0011] A method for imparting a stone-washed appearance to denim garments by cellulase enzymes is disclosed, for example, in the US Patent No, 4,832,864.

[0012] Some publications have suggested biofinishing of textile materials in a broad sense meaning fabrics, knits, ready-made garments and their raw materials, such as fibres and yarn (WO 94/12578, WO 96/34945, WO 97/14804). However, in the prior art there has been no teaching of how and in which phase of the process the cellulase treatment of yarn should be performed, nor has there been any data about the effect of cellulase treatment of yarns on fabrics and knits, on the processing of yarns or on the yarn properties. On the basis of experience of fabric treatment with cellulases it is not possible to predict whether cellulase enzymes will cut hairs and neps on the yarn surface or whether they will cut the narrow areas of the yarn cutting the yarn into short pieces.

[0013] The present invention aims at eliminating the problems relating to the prior art and it is an object of the present invention to provide a cellulase-treated spun yarn having reduced hairiness, a decreased yarn number, improved evenness and/or reduced dustiness.

[0014] It is another object of this invention to provide a fabric or knit prepared from the cellulase-treated spun yarn.

[0015] It is a further object of this invention to provide a process for treating yarn, which comprises a cellulase treatment step.

[0016] It is also an object of this invention to provide

a method for reducing the hairiness, decreasing the yarn number, improving the evenness of yarn and/or reducing the dustiness, the method comprising treating spun yarn by a cellulase solution.

[0017] It is still a further object of this invention to provide a method for treating especially carded yarn. It has surprisingly been found that by cellulase treatment open-end spun yarn becomes usable in applications where higher-quality longer-fibered cotton such as ring-spun yarn or combed yarn is normally used.

[0018] According to one embodiment of this invention the cellulase treatment step is carried out after spinning and reconing of the yarn onto yarn cones, but before the pretreatment of the yarn. If needed, the yarn is then bleached and dyed and other finishing steps such as waxing and sizing steps are performed. Waxing is specifically used for improving the running of yarn used in knitting machines. The term sizing is used to refer to the treatment of yarns for example by starch before weaving in order to make warp yarn firm at loom.

[0019] According to a second embodiment of this invention the cellulase treatment step is carried out after the pretreatment. As above, the yarn is then bleached and dyed and other finishing steps such as waxing and sizing are performed, if needed.

[0020] According to a third embodiment of this invention the cellulase treatment step is carried out after bleaching. If needed, the yarn is then dyed and other finishing steps such as waxing and sizing steps are performed.

[0021] According to a fourth embodiment of this invention the cellulase treatment step is carried out after dyeing. After the cellulase treatment other finishing steps such as waxing and sizing steps may be performed.

[0022] According to a fifth embodiment of this invention the cellulase treatment step is carried out in connection with the dyeing step.

[0023] This invention provides various advantages. Cellulase enzymes cut the hairs protruding from the raw yarn surface and they are able to remove the thicker areas and neps from the surface of yarn. This improves the evenness of the yarn, resulting in a more even fabric or knit with a cleared surface structure (e.g. in knittings the stitch structure is clearer), reduced pilling and fuzz. The hairiness of cellulase-treated yarn may be at least 10 % , preferably 20 %, most preferably 50 % smaller than the hairiness of untreated yarn.The pilling and fuzz is reduced by at least 20%, preferably 50 % and most preferably 100 % compared to the pilling and fuzz in fabric prepared from untreated yarn. The following parameters: thick places, thin places and/or neps, which have effect on the yarn evenness, have decreased at least 10 %, preferably, 20 %, most preferably 30 % compared to untreated yarn.

[0024] For fabric manufacturers these are remarkable advantages, because they can use yarn of better quality and they do not need to finish the fabric or knit or ready-made garment by cellulase enzymes, chemicals or by technical means like burning.

[0025] When yarn is subjected to a cellulase treatment, the fabric prepared from the yarn is not dusty, because mechanical action removes dust from the yarn during the yarn processing steps. The fabric prepared from the cellulase-treated yarn may carry at least 50 % less dust than a fabric treated by a substantially similar cellulase treatment. If the fabric or ready-made garment is subjected to a cellulase treatment the garment may carry some dust during the first few washes and initial use. The undustiness of the yarn is also very important for employees, rooms and facilities during knitting or weaving processes. The dustiness of the yarn is 50 %, preferably 80% decreased compared to untreated yarn.

[0026] One further advantage of the present invention is that it can be difficult to find a suitable cellulase treatment for a fabric which is a blend of different cellulosic fibers. The cellulase dosage and treatment time must be suitable for the weak as well as for the strong component of the fabric, e.g. linen/lyocell. This results in a treatment which is a compromise and in fact not ideal for either of the textile materials. If different cellulosic yarns are treated separately", the cellulase treatment time, dosage and other parameters can be optimized especially for each textile material.

[0027] One advantage of the present invention is that by means of a cellulase treatment of yarn the yarn number can be decreased. The yarn number of cellulase-treated yarn may be 3 to 7 % smaller than the yarn number of untreated yarn. This is an advantage especially for open-end spun yarn. By open-end spinning it is not possible to prepare a yarn under 20 tex. Consequently, in applications where thin yarn is needed, the use of more expensive ring-spun yarn has been necessary. If open-end spun yarn is treated by the method of this invention the resulting yarn can be used instead of ring-spun yarn.

[0028] In ring-spinning of yarn combing is used to reduce the hairiness of the yarn. The combing step is not needed at all if the yarn is finished by a cellulase treatment.

[0029] In open-end spinning shorter cotton fibers can be used than in ring spinning. However the open-end spun yarn is hairier and visually dull because of its structure and in dyed yarn products the tone of the colours is not "rich enough" for the eye. The yarn is stiff and lacks elasticity. Although open-end spun yarn is 15 to 25 % cheaper, its scope of application is restricted. One significant advantage of treating open-end spun yarn with cellulases is that from cheaper raw material it is possible to prepare high quality fabrics and knitwear. No combing of the yarn is needed. The tone of colours looks better, the yarn is bending and elastic and the products prepared from cellulase treated open-end spun yarn do not wrinkle easily. Hence the scope of application of open-end spun yarn can be remarkable widened.

[0030] There are several advantages in treating flax

yarn by cellulases. The hairiness and stiffness of the yarn is decreased and bendiness increased, which improves the feel and drapability of linen fabrics.

[0031] The results of treating yarn by cellulase enzymes are illustrated in Figures 1 to 40.

[0032] Figure 1. Yarn number of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0033] Figure 2. Hairiness, length 1 mm, of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0034] Figure 3. Hairiness, length 2 mm, of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0035] Figure 4. Hairiness, length 3 mm, of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0036] Figure 5. Hairiness, length 4 mm, of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0037] Figure 6. Hairiness, length 6 mm, of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0038] Figure 7. Hairiness, length 8 mm, of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0039] Figure 8. Hairiness, length 10 mm, of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0040] Figure 9. Yarn breaking load of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0041] Figure 10. Yarn elongation of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0042] Figure 11. The thin places of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0043] Figure 12. The thick places of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0044] Figure 13. The neps of the reference cotton yarn and the yarns after treatment with cellulase in an ILMA dyeing machine.

[0045] Figure 14. Yarn number of the reference cotton yarn, the yarn after treatment with cellulase in a Thies dyeing machine and after treatment with cellulase and dyeing.

[0046] Figure 15. Hairiness, length 1 mm, of the reference cotton yarn, the yarn after treatment with cellulase in a Thies dyeing machine and after treatment with cellulase and dyeing.

[0047] Figure 16. Hairiness, length 2 mm, of the reference cotton yarn, the yarn after treatment with cellulase in a Thies dyeing machine and after treatment with cellulase and dyeing.

[0048] Figure 17. Hairiness, length 3 mm, of the reference cotton yarn, the yarn after treatment with cellulase in a Thies dyeing machine and after treatment with cellulase and dyeing.

[0049] Figure 18. Hairiness, length 4 mm, of the reference cotton yarn, the yarn after treatment with cellulase in a Thies dyeing machine and after treatment with cellulase and dyeing.

[0050] Figure 19. Hairiness, length 6 mm, of the reference cotton yarn, the yarn after treatment with cellulase in a Thies dyeing machine and after treatment with cellulase and dyeing.

[0051] Figure 20. Hairiness, length 8 mm, of the reference cotton yarn, the yarn after treatment with cellulase in a Thies dyeing machine and after treatment with cellulase and dyeing.

[0052] Figure 21. Hairiness, length 10 mm, of the reference cotton yarn, the yarn after treatment with cellulase in a Thies dyeing machine and after treatment with cellulase and dyeing.

[0053] Figure 22. Yarn breaking load of the reference cotton yarn, the yarn after treatment with cellulase in a Thies dyeing machine and after treatment with cellulase and dyeing.

[0054] Figure 23. Yarn elongation of the reference cotton yarn, the yarn after treatment with cellulase in a Thies dyeing machine and after treatment with cellulase and dyeing.

[0055] Figure 24. Pilling of knitted reference cotton yarn, enzyme-treated cotton yarn and enzyme-treated and dyed cotton yarn after 125 rubs.

[0056] Figure 25. Pilling of knitted reference cotton yarn, enzyme-treated cotton yarn and enzyme-treated and dyed cotton yarn after 500 rubs.

[0057] Figure 26. Pilling of knitted reference cotton yarn, enzyme-treated cotton yarn and enzyme-treated and dyed cotton yarn after 2000 rubs.

[0058] Figure 27. The thin places of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference).

[0059] Figure 28. The thick places of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference).

[0060] Figure 29. The neps of the reference cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference).

[0061] Figure 30. Yarn number of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference).

[0062] Figure 31. Hairiness, length 1 mm, of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn

(reference).

[0063] Figure 32. Hairiness, length 2 mm, of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference).

[0064] Figure 33. Hairiness, length 3 mm, of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference).

[0065] Figure 34. Hairiness, length 4 mm, of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference).

[0066] Figure 35. Hairiness, length 6 mm, of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference).

[0067] Figure 36. Hairiness, length 8 mm, of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference).

[0068] Figure 37. Hairiness, length 10 mm, of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference).

[0069] Figure 38. Yarn breaking load of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference).

[0070] Figure 39. Yarn elongation of the of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference).

[0071] Figure 40. Pilling of the knitting made of the raw cotton yarn (reference), the scoured yarn (reference), the scoured enzyme-treated yarn, the scoured enzyme-treated and dyed yarn and the scoured and dyed yarn (reference) after 125 rubs, 500 and 2000 rubs.

[0072] The present invention will be discussed in detail in the following description and examples.

[0073] According to a first embodiment of this invention yarn is treated by cellulase enzymes after spinning and after reconing in a raw yarn phase. The cellulase treatment is followed by a pretreatment.

[0074] There are still pectins and other undesirable plant subtances left in raw yarn. These must be removed by a pretreatment. The pretreatment may be an alkalic treatment in the presence of caustic soda.

[0075] The pretreatment step may be followed by a bleaching step especially if light colours or white yarn are/is desired. The bleaching may be done by well known bleaching chemicals such as hydrogen peroxide. After the bleaching step the yarn is dyed, if coloured yarn is produced. The yarn may be then finished by well-known finishing steps such as waxing (for knit machines) or sizing. Waxing is specifically used for improving the running of yarn used in knitting machines.

[0076] The raw yarn is reconed on a perforated yarn cone before the alkalic pretreatment. The cellulase treatment step may be performed on the same cones in the same machine as the pretreatment or bleaching or dyeing. The cellulase treatment step must be carried out in a solution wherefore it should be performed within a wet processing step in the yarn processing procedure.

[0077] The cones are subjected to a cellulase solution circulating the yarn on the cones from different directions. The solution may be pumped through the cone preferebly changing the direction of the liquid flow preferably in both directions, because the treatment should be as consistent as possible over the entire cone. It is advantageous to change the direction of the liquid flow at least once, preferable 5 to 10 times.

[0078] According to a second embodiment of this invention the cellulase treatment is carried out after the pretreatment step.

[0079] According to a third embodiment of this invention the cellulase treatment is carried out after the bleaching step, but before the dyeing step. The cellulase treatment can be performed in the dyeing machine on the same cones as the dyeing.

[0080] According to a fourth embodiment of this invention the cellulase treatment step is performed after the dyeing step. Here again the cellulase treatment can be performed in the dyeing machine on the same cones as the dyeing.

[0081] According to a fifth embodiment of this invention the cellulase treatment step is performed in connection with the dyeing step. The enzyme is added first, then the dye and there is no rinsing step with water between the steps, which saves time.

[0082] Fungi and bacteria can be used as source material for the cellulase composition of the present invention, whereby the cellulase compositions of this invention are preferebly derived from fungi, for example from species of the genera *Trichoderma, Penicillium, Humicola, Fusarium* or *Melanocarpus,* whereby *Trichoderma reesei* is preferably employed as the source material. As for *Trichoderma reesei,* suitable strains include, for example, the wild type strain of *T. reesei* QM6a and the derived mutant strains, e.g. QM9414 and RutC-30, developed for cellulase production, and strains further developed from these, in which e.g.the cellulase level has been further raised, and/or in which the level of produced protease has been lowered. If the resulting cellulase composition comprising CBH components and various EG components is not as such applicable to the treatment of cellulose-containing textile materials for

producing the desired end result, the composition and the ratios between the CBH and EG components can be modified so as to obtain a desired composition. When a strain other than *Trichoderma* is used as the source material, it is possible, in a similar fashion, to modify the ratios of the types of cellulase produced by the employed strain and functionally corresponding to *Trichoderma* cellulases. The modification of cellulase compositions can be done by biochemical fractionation methods or by genetically engineering the cellulase producing hosts as described in US 5,298,405.

[0083] Suitable enzyme dosages are 0.1 to 100 mg cellulase per gram of yarn, advantageously 1 to 50 and preferably 15 to 40 mg per gram of yarn. Examples of typical commercial liquid cellulases that can be used in this invention are Biotouch® L, Biotouch® C 601, Biotouch® C25, Biotouch® NC40, Ecostone® L 20, Ecostone® L 300, Ecostone® C80, Ecostone® N400, Ecostone® N500, (Röhm Enzyme Finland Oy, Nurmijärvi, Finland). Other corresponding cellulases imparting the same effects may be used. The suitable enzyme dosages for imparting the desired effects to yarn depend on the desired result, on the treatment method and the activity of the enzyme product.

[0084] Hence a suitable enzyme dosage can be calculated on the basis of the weight of the yarn, the amount of the circulating solution, cellulase treatment time and concentration of the enzyme, and the type of the treatment (dyeing) machine.

[0085] Treatment media of yarn may contain, in addition to enzymes, e.g. surfactants, polymers as for example PVA and PVP polymers, buffers as for example citrates, acetates and phosphates for regulating the acidity of the solutions, possibly bulk agents, conventional preserving agents, stabilisers and abrasion agents.

[0086] The yarn liquid ratio may be from 1:5 up to 1:80, preferably 1:10 to 1:40, most preferably 1:10 to 1:20.

[0087] The treatment time may be 0.5 h to 2 h, preferably 1 to 1.5 h.

[0088] The pH range for applying the cellulase composition of this invention is dependent on the pH activity profile of the enzyme. The pH may be adjusted to pH 3 to 10. preferably to pH 4.5 to 8 and most preferably to pH 5 to 6.

[0089] The enzyme treatment is stopped by raising the pH or temperature to such a high level that the cellulase enzyme becomes inactive. The pH may be raised to a value above pH 9 and/or the temperature may be raised to a temperature above 85°C.

[0090] Enzyme activity is one concept applied in determining the properties of enzymes. The enzyme activities used in the present patent application are ECU, CMC, FPU and MUL, which are defined as follows:

ECU

[0091] The endo-1,4-beta-glucanase in the sample hydrolyses the hydroxyethyl cellulose substrate, and the resulting reducing sugars are assayed spectrophotometrically using a dinitrosalicylic acid reagent (DNS). One unit of endo-1,4-beta-glucanase is defined as the amount of enzyme producing one nmole of reducing sugars as glucose in one second (1 ECU = 1 nkat), (Bailey, M. and Nevalainen, H., 1981, Enzyme Microb. Technol. 3:153-157).

FPU

[0092] The cellulase in the sample hydrolyses the filter paper used as a substrate, and the resulting reducing sugars are assaved spectrophotometrically using a DNS reagent. The filter paper degrading activity is described as FPU units. The calculation is based on the definition of the International Unit (IU). 1 IU = 1 micromol min^{-1} of product formed (reducing sugars as glucose) (Commission on Biotechnology, International Union of Pure and Applied Chemistry, in "Measurement of Cellulase Activities" (T.K. Chose. Ed.) Biochemical Engineering Research Centre. Indian Institute of Technology, New Delhi, India, 1984).

MUL

[0093] The cellobiohydrolase (CBHI) and endoglucanase (EGI) of the sample hydrolyse the 4-methylumbelliferyl-beta-D-lactoside that acts as a substrate, whereby methylumbelliferone is released that can be measured spectrophotometrically. The method can be applied in the determination of cellobiohydrolase I (CBHI) activity. The method also measures the endoglucanase I (EGI) activity, whose proportion can be determined by inhibiting the activity of cellobiohydrolase using 5 mM of cellobiose. One MUL unit is the amount of enzyme activity that in one second under the determination conditions, releases 1 nmol of methylumbelliferone from 4-methylumbelliferyl-beta-D-lactoside (van Tilbeurgh *et al.* Meth. Enzymol. 160:45-59, 1988).

CMC

[0094] The endo-1,4-beta-glucanase in the sample hydrolyses the carboxymethyl cellulose substrate, and the resulting reducing sugars are assayed spectrophotometrically using a dinitrosalicylic acid reagent (DNS). One unit of endo-1,4-beta-glucanase is defined as the amount of enzyme producing one nmole of reducing sugars as glucose in one second (Measurement of cellulase activities, Pure & Appl.Chem., Vol. 59, No. 2. pp. 257-268, 1987).

[0095] The material of the treated yarn may be 100% cotton or 100% non-cotton cellulosic fibre or it may be a blend of different raw materials such as cotton, flax, ramie, jute or man-made cellulosic fibres, for example, viscose, modal, lyocell (e.g. Tencel®) or cupro. It may be a blend of cellulosic fibres and synthetic man-made

fibres. In this invention the amount of cellulosic fibre in the yarn is advantageously at least 30 percent, preferably over 50 percent.

[0096] By the "evenness" of the yarn is meant here that the yarn does not contain any significant amount of thin places, which here means a 40% deviation from the mean value, or thick places, which here means a 50% deviation from the mean value. Neps are thicker places which differ by 200% from the mean value. Evenness was measured here by a Uster evenness tester (see example 1).

[0097] By the "hairiness" of the yarn is meant here that hairs of differing lengths protrude from the surface of the yarn. Hairiness was measured here by a Zweigle G565 hairiness tester (see example 1).

[0098] The yarn strength was measured here as breaking load and elongation according to SFS Standard 2810 by using an Alwetron drawing machine (see example 1).

[0099] The yarn number was measured according to the "tex" system. The tex value indicates the mass of yarn having a length of 1 km in grams.

[0100] Yarn which is less than 20 tex is usually ring-spun yarn. When in a certain application an over 25 tex yarn is usable, open-end spun yarn may be used. The present invention decreases the yarn number and makes open-end spun yarn usable for several applications, where ring-spun yarn has previosly been used.

[0101] Normally the open-end spinning area varies between 17 and 200 tex.

[0102] Here the yarn number was measured according to SFS Standard 2780.

[0103] The term "fabric" is used here to refer to a woven fabric or a knitting.

[0104] "Pilling" means here small accumulations of fibre on the surface of the fabric. Pilling was tested here by a Martindale abrasion tester. The knittings were rubbed against standard wool fabric using various numbers of cycles (here 125, 500 and 2000 cycles) and after each number of cycles the degree of pilling was evaluated in artificial day light by test persons.

[0105] The following examples and figures provide further details of the invention.

Example 1

[0106] Rotor spun cotton raw yarn whose number was 50 tex, was treated with a cellulase enzyme in a pressure dyeing machine (ILMA, Italy). One yarn cone of about 1.2 kg was treated at a time. The treatment was performed at pH 5, 50°C for one hour with a liquid ratio of 1:67. The cellulase preparation was Ecostone® L (Röhm Enzyme Finland Oy, Nurmijärvi, Finland). Enzyme doses of 12 and 40 mg of total protein per gram of yarn were used. After treatment the enzyme was inactivated by raising the pH to a value over 9. Then the yarn was rinsed with water three times 5 minutes. A reference cone was treated under the same conditions as the cellulase-treated cones but without the cellulase.

[0107] The enzyme treated yarn was examined by the following methods:

Yarn number

[0108] According to SFS Standard 2780

Yarn evenness

[0109] A Uster evenness tester (Zellweger Uster, Switzerland) was used following the instructions of the manufacturer. The speed of the yarn was 400 m/min and the testing time was 2.5 minutes. A thin place was a place, which deviated by 40% from the mean value of the yarn and a thick place deviated by 50% and neps by 200% from the mean value.

Hairiness

[0110] A Zweigle G 565 hairiness tester (Zweigle Textilpruefmaschinen GMBH, Germany) was used following the instructions of the manufacturer. The driving length was 100 m, speed 50 m/min. The test was done three times in a row.

Strength measurement

[0111] Breaking load and elongation by an Alwetron drawing machine in accordance with SFS Standard 2810.

[0112] All tests were performed 9 times on one yarn package.

[0113] The results are given in Figures 1 - 13.

[0114] The results show that compared to the reference sample the yarn number decreased with the higher enzyme dosage. Also the hairiness decreased with all length of hair and generally speaking the decrease was bigger with a higher dosage. Compared to the reference sample the thin and thick places and neps were decreased in the enzyme treated yarn samples The enzyme treatment did not have any effect on the breaking load and elongation of the yarn when compared to the reference sample.

Example 2

[0115] Rotor spun cotton yarn whose number was 50 tex was treated with a cellulase enzyme in an industrial-scale machine (Thies, open yarn dyeing machine, Germany). There was 26 kg of yarn in 21 cones (7 cones in one tube) in the machine. Before the cellulase treatment the cones were scoured with 3 g/l of 50% caustic soda at 95 °C for 30 minutes. The cellulase treatment was performed at pH 5 and 50 °C for one hour with a liquid ratio of 1:40. The cellulase preparation was Biotouch® C25 (Röhm Enzyme Finland Oy, Nurmijärvi. Finland). An enzyme dose of 38 mg of total protein per gram of

yarn was used. After the treatment the enzyme was inactivated by raising the temperature to 85 °C for 10 minutes. Some of the yarn cones were dyed by a reactive dye according to the dye manufacturer's instructions.

[0116] Scoured untreated (reference), enzyme-treated. and enzyme-treated and dyed specimens were tested by the same methods as described in example 1. All cones were tested 12 times, from the surface to the bottom of the cone.

[0117] The pilling was tested of the knitted reference cotton yarn, enzyme-treated cotton yarn and enzyme-treated and dyed cotton yarn by a Martindale abrasion tester. The knittings were rubbed against standard wool fabric 125, 500 and 2000 times and they were evaluated in artificial day light by three persons. The evaluation scale was 1 - 5 where 5 is the best grade.

[0118] The results are given in Figures 11 - 26.

[0119] The results show that compared to the reference sample the yarn number was decreased as well as the hairiness with hair lengths of 4, 6, 8 and 10 mm after enzyme treatment. Also the breaking load and elongation of the yarn were decreased. Compared to the reference sample the thin and thick places and neps were decreased in the enzyme-treated yarn samples. After 125 rubs the pilling has decreased at the beginning of the enzyme-treated cone, but inside the cone the results are the same with all specimens. After 500 rubs the reference knitting is the worst and the enzyme-treated and dyed the best, after 2000 rubs the results are the same.

Example 3

[0120] Rotor-span cotton yarn whose number was 50 tex was treated with a cellulase enzyme in an industrial-scale machine, where the solution was pumped through the cone surface from inside out and from outside in of the cone (Thies, yarn dyeing machine, Germany). The direction of the solution was changed every 10 minutes. There was 25 kg of yarn in 21 cones (7 cones in one tube) in the machine. Before cellulase treatment the cones were scoured with 3 g/l of 50% caustic soda at 95 °C for 30 minutes. The cellulase treatment was performed at pH 5 and 50 °C for one hour with liquid ratio of 1:40. The cellulase preparation was commercial cellulase preparation Biotouch C25 (Röhm Enzyme Finland Oy, Nurmijärvi, Finland). Enzyme dose of 38 mg of total protein per gram of yarn was used. After treatment the enzyme was inactivated by raising the temperature to 85 °C for 10 minutes. Some of the yarn cones was dyed by reactive dye according to the dye manufacturer's instructions.

[0121] The following specimens were tested with the same methods as described in the examples 1 and 2:

raw yarn (reference)
scoured untreated yarn (reference)
scoured untreated dyed yarn (reference)
scoured enzyme-treated yarn
scoured enzyme-treated dyed yarn

[0122] All cones were tested 6 times, from the surface to the bottom of the cone.

[0123] The results are given in Figures 30 - 40.

[0124] The results show that compared to the reference samples (raw yarn and scoured untreated yarn) the yarn number as well as hairiness with hair lengths of 1, 2, 3, 4, 6 and 8 mm were decreased after enzyme treatment. After dyeing, however, the yarn number and hairiness were increased to about the same level with both enzyme-treated and untreated samples. The breaking load of the yarn was decreased about 20% and the elongation about 10% when comparing the scoured untreated yarn and the scoured enzyme-treated yarn. After dyeing the breaking load and elongation were decreased about 30% when comparing the scoured untreated dyed yarn and the scoured enzyme-treated dyed yarn.

[0125] The pilling of the knits was decreased after 125, 500 and 2000 rubs in the scoured enzyme-treated cone compared to the scoured untreated cone. When comparing the dyed specimens the pilling was clearly decreased after 2000 rubs in the enzyme-treated sample compared to the untreated sample, but after 125 and 500 rubs the pilling was the same with both enzyme-treated sample and untreated reference sample.

[0126] It was also noticed that the stitches and the structure of the knitting was more even in the knittings made of enzyme-treated yarns compared to the reference knittings.

Claims

1. Cellulase-treated cellulosic spun yarn, wherein the hairiness of the yarn is at least 10 % smaller than that of untreated yarn.

2. The yarn according to claim 1, wherein the hairiness of the yarn is 20 %, preferably 50 % smaller than that of untreated yarn.

3. Cellulase-treated cellulosic spun yarn, wherein the yarn number is 3 to 7 % smaller than that of untreated yarn.

4. Cellulase-treated cellulosic spun yarn, wherein one of the following parameters: thick places, thin places and/or neps, which have effect on the yarn evenness, have decreased at least 10 % compared to untreated yarn.

5. The yarn according to claim 4, wherein one of the following parameters: thick places, thin places and/or neps, have decreased 20%, preferably 30 % compared to untreated yarn.

6. Cellulase-treated cellulosic spun yarn, wherein the dustiness of the yarn is decreased 50 %, preferable 80 % compared to untreated yarn.

7. The yarn according to any one of claims 1 to 6, wherein the yarn is open-end spun yarn.

8. The yarn according to any one of claims 1 to 6, wherein the yarn is ring-spun yarn.

9. The yarn according to any one of claims 1 to 8, wherein the yarn is cotton.

10. The yarn according to any one of claims 1 to 8, wherein the yarn is flax, ramie, jute or man-made cellulosic yarn such as viscose, modal, lyocell, cupro.

11. The yarn according to any one of claims 1 to 10, wherein the yarn is a blend of cellulosic cotton and non-cotton fibres or cellulosic fibres and synthetic man-made fibres.

12. Fabric made from the yarn of claims 1 to 11, wherein the pilling and fuzz of the fabric is 20 %, preferably 50 % and most preferably 100 % smaller than that of untreated fabric and wherein the dustiness of the fabric is at least 50 % smaller than that of cellulase-treated fabric.

13. The fabric according to claim 12, wherein the fabric is a woven fabric or a knitting.

14. A process for treating cellulosic yarn, comprising treating spun yarn with a cellulase solution after one of the following treatment steps: spinning, pretreatment, bleaching or dyeing.

15. The process according to claim 14, wherein the cellulase treatment step is carried out before said pretreatment step.

16. The process according to claim 14, wherein the cellulase treatment step is carried out before said bleaching step.

17. The process according to claim 14, wherein said cellulase treatment step is carried out before said dyeing step.

18. The process according to claim 14, wherein said cellulase treatment step is carried out after said dyeing step.

19. The process according to claim 14, wherein the cellulase treatment step is carried out in connection with said dyeing step.

20. The process according to any one of claims 14 to 19, wherein said spun yarn is ring-spun yarn.

21. The process according to any one of claims 14 to 19, wherein said spun yarn is open-end spun yarn.

22. The process according to any one of claims 14 to 21, wherein said cellulosic yarn is cotton.

23. The process according to any one of claims 14 to 22, wherein said cellulosic yarn is non-cotton e.g., flax, ramie, jute or man-made cellulosic yarn such as viscose, modal, lyocell, cupro.

24. The process according to claims 22 and 23, wherein said cellulosic yarn is a blend of cotton and non-cotton cellulosic materials.

25. The process according to any one of claims 14 to 24, wherein said cellulosic yarn is a blend of cellulosic fibres and synthetic man-made fibres and wherein the amount of cellulosic fibres in the blend is at least 30 %.

26. The process according to any one of claims 14 to 24, wherein said cellulosic yarn is a blend of cellulosic fibres and synthetic man-made fibres and wherein the amount of cellulosic fibre in the blend is at least 50 %.

27. The process according to any one of claims 14 to 26, wherein the cellulase dosage in said cellulase treatment is 0.1 to 100 mg cellulase enzyme per gram of yarn, preferably 1 to 50 and most preferably 15 to 40 mg cellulase per gram of yarn.

28. The process according to any one of claims 14 to 27, wherein said cellulase treatment is performed at a yarn: liquid ratio of 1:5 to 1:80, preferably 1:10 to 1:40, most preferably 1:10 to 1:20.

29. The process according to any one of claims 14 to 28, wherein said cellulase treatment time is 0.5 to 2 h, preferably 1 to 1.5 h.

30. The process according to any one of claims 14 to 29, wherein said cellulase treatment pH is adjusted to pH 3 to 10, preferably 4.5 to 8, most preferably to pH 5 to 6.

31. A process for treating cellulosic yarn, which comprises the following steps:

- reconing spun yarn onto perforated yarn cones,
- subjecting said cones to at least one of the following treatment steps: pretreatment, bleaching and/ or dyeing,
- subjecting said cones to a cellulase treatment,

said treatment being carried out by pumping the cellulase solution through the cones.

32. The process according to claim 31, wherein said cellulase treatment is carried out on reconed yarn before or after at least one of the following steps: pretreatment, bleaching or dyeing.

33. The process according to claim 31 or 32, wherein said cellulase treatment is carried out in connection with dyeing.

34. The process according to any one of claims 31 to 33, wherein said cellulase treatment is carried out by pumping the cellulase solution through the cones and changing the direction of the liquid flow at least once, preferably 5 to 10 times.

35. A method for reducing the hairiness, decreasing the yarn number, improving the evenness and/or reducing the dustiness of yarn, comprising treating spun yarn by a cellulase solution after one of the following treatment steps: spinning, pretreatment, bleaching or dyeing.

36. A method for reducing the hairiness and/or reducing the pilling and fuzz and/or reducing the dustiness of a fabric or knit, comprising treating the spun yarn from which the fabric or knit is prepared by a cellulase solution after one of the following treatment steps: spinning, pretreatment, bleaching or dyeing.

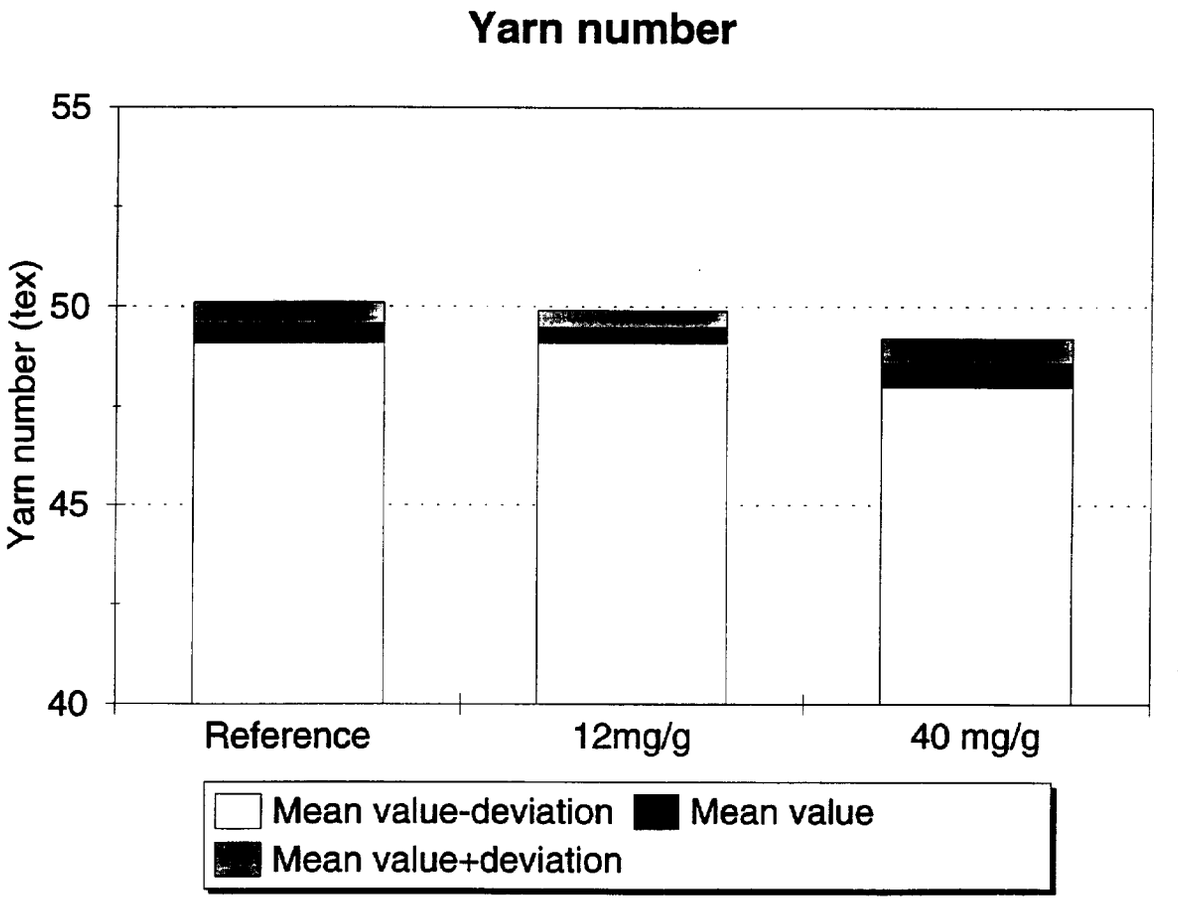
Yarn number
55
50
45
40
Yarn number (tex)
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG. 1

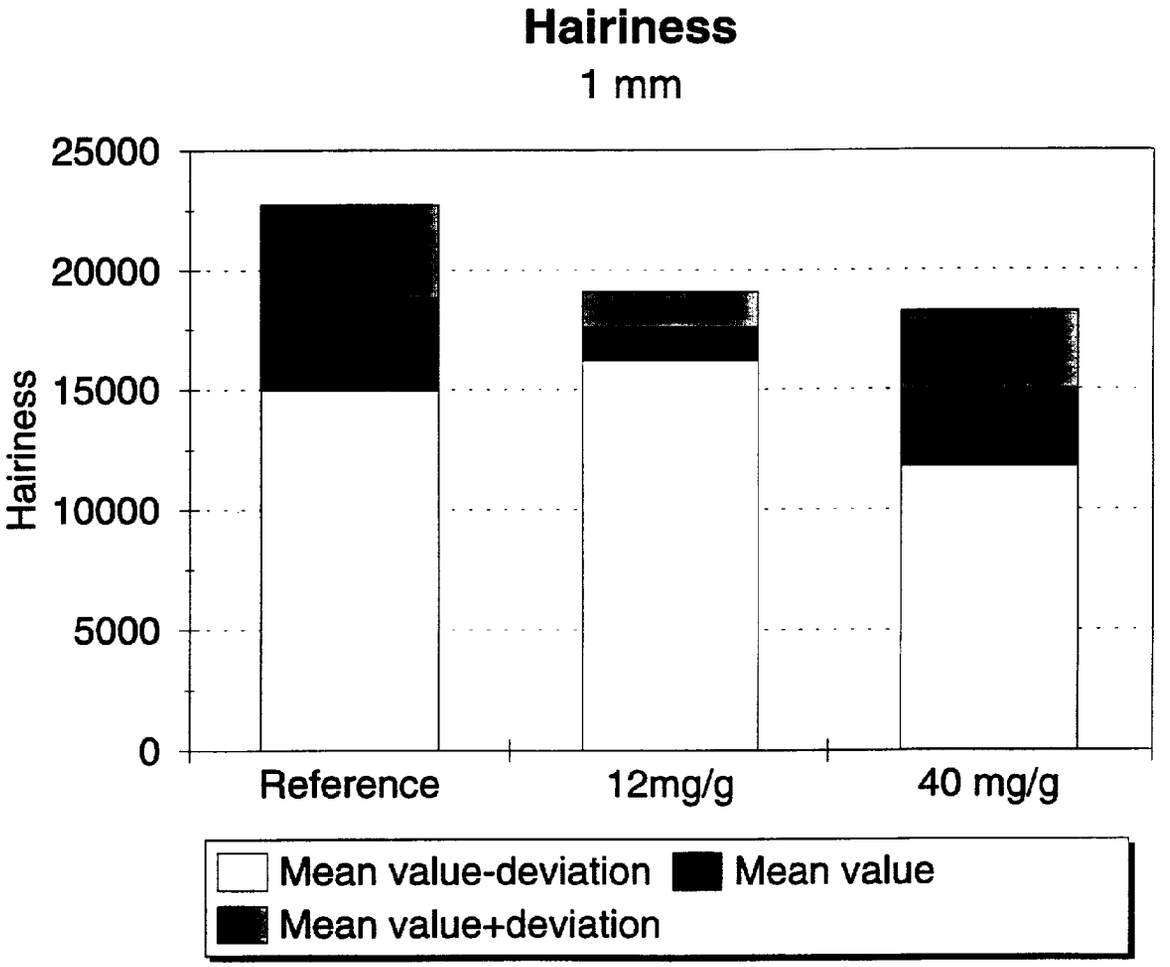
Hairiness
1 mm
25000
20000
15000
10000
5000
0
Hairiness
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG. 2

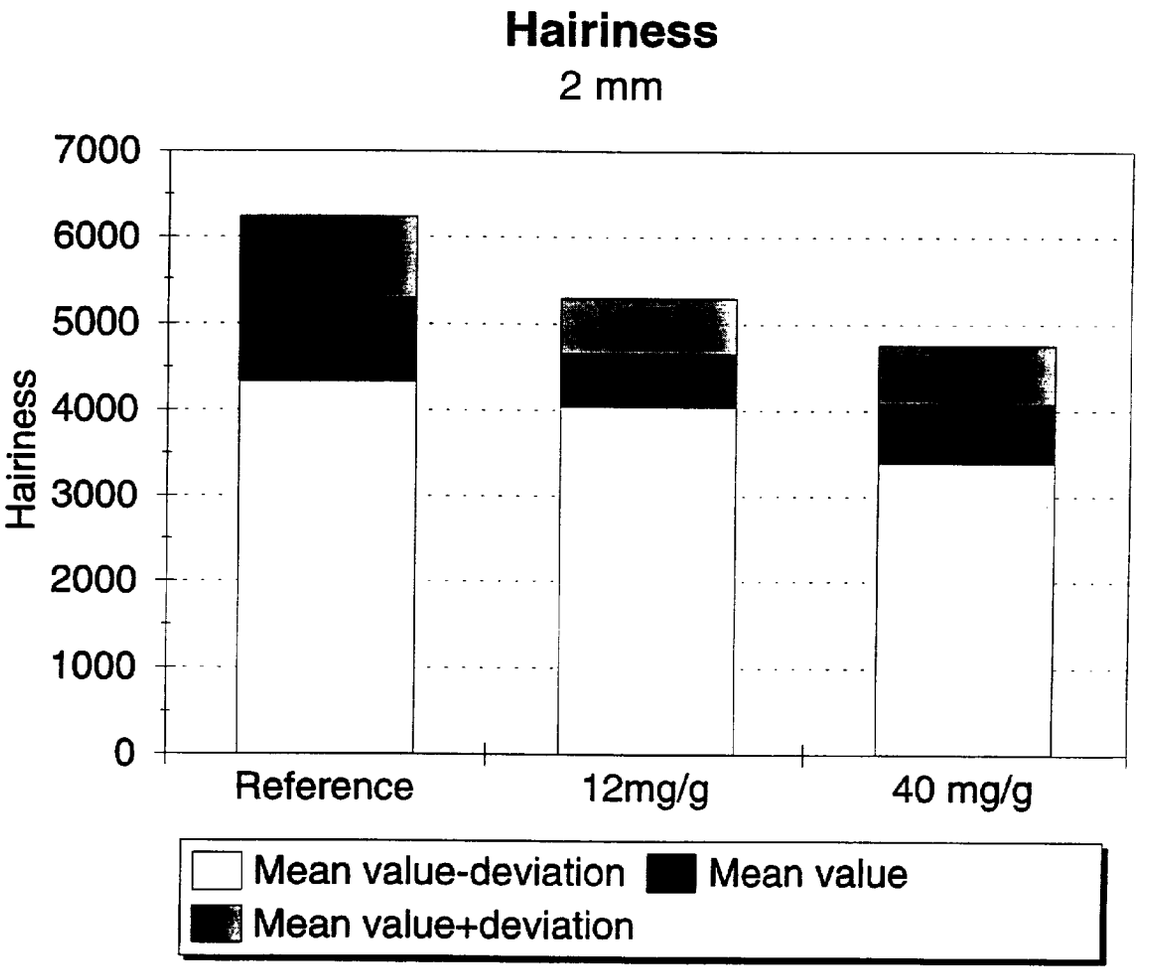
Hairiness
2 mm
Hairiness
7000
6000
5000
4000
3000
2000
1000
0
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG. 3

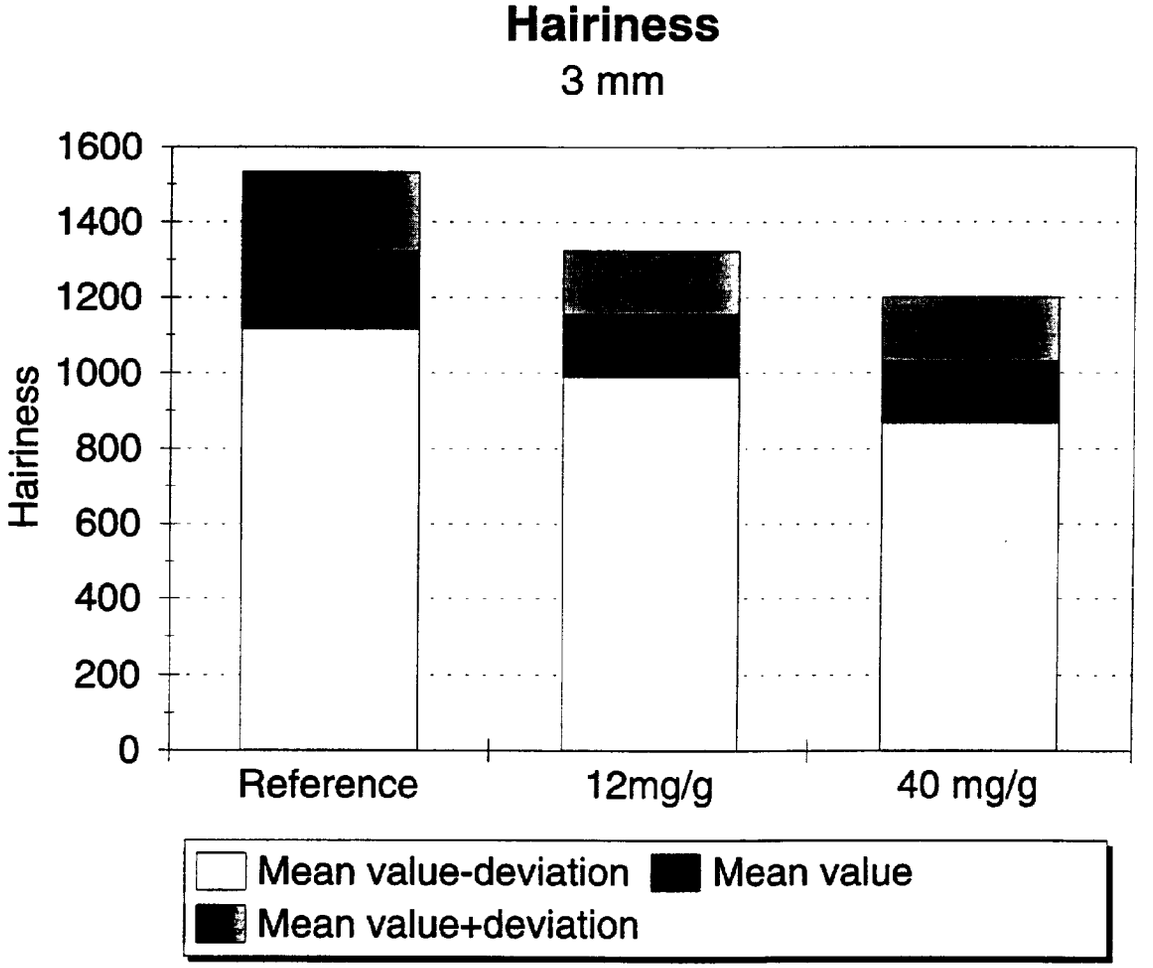
Hairiness
3 mm
1600
1400
1200
1000
800
600
400
200
0
Hairiness
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG. 4

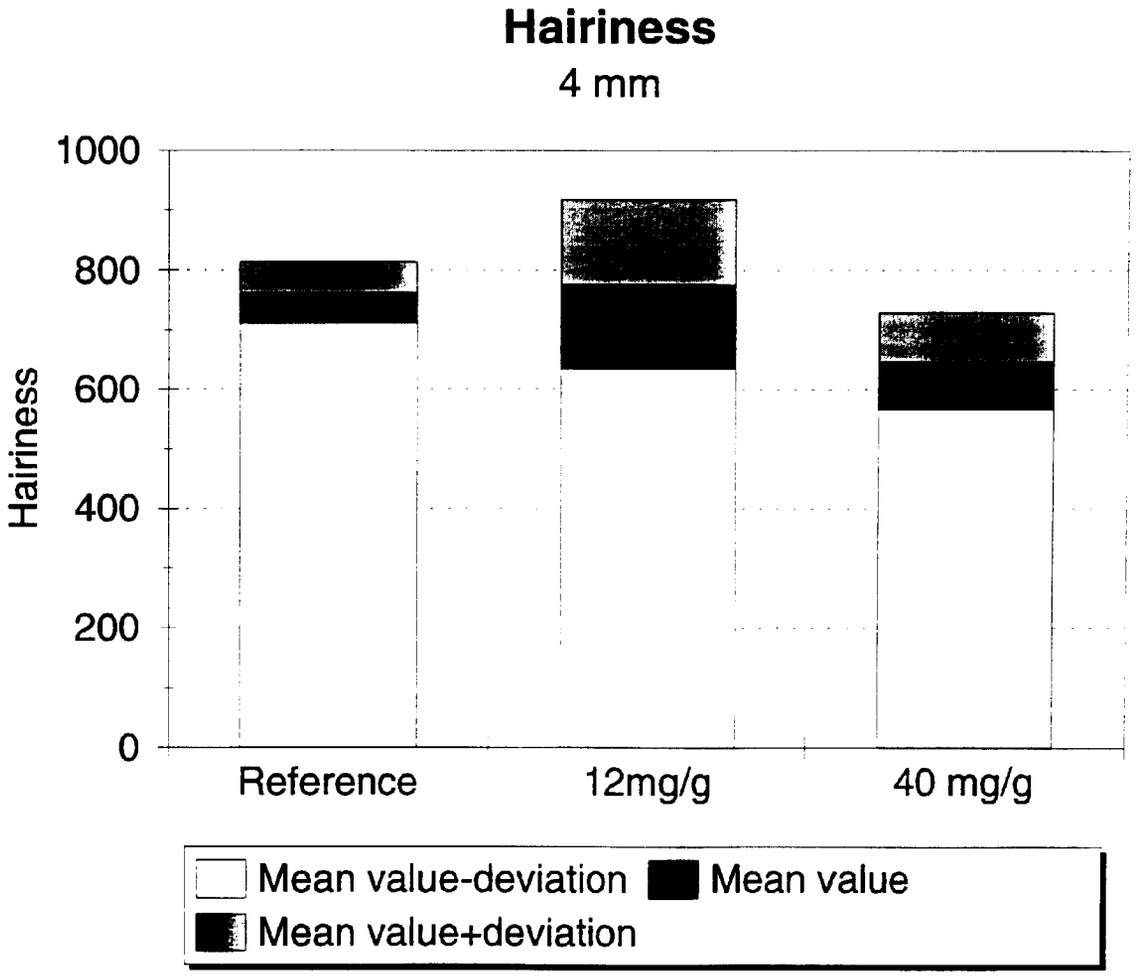
Hairiness
4 mm
1000
800
600
400
200
0
Hairiness
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG. 5

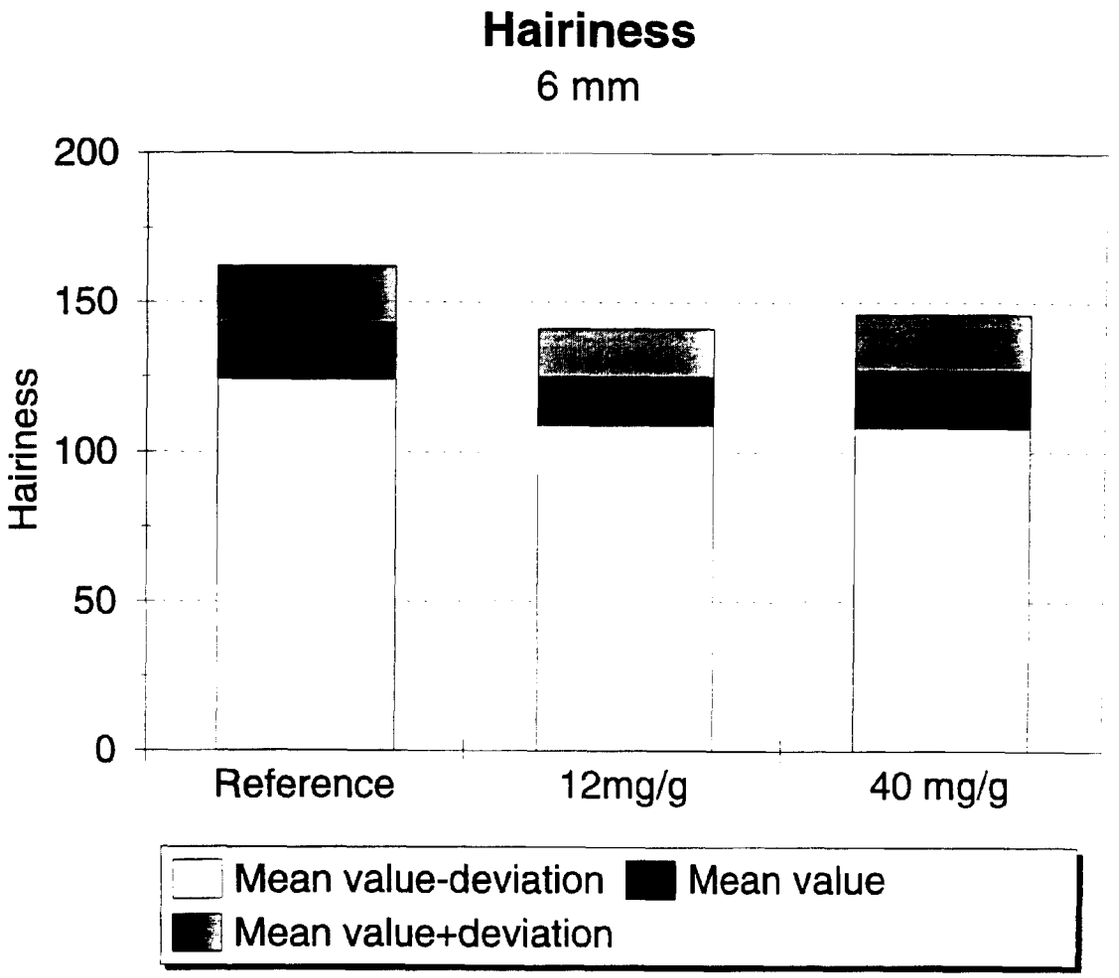
Hairiness
6 mm
200
150
100
50
0
Hairiness
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG. 6

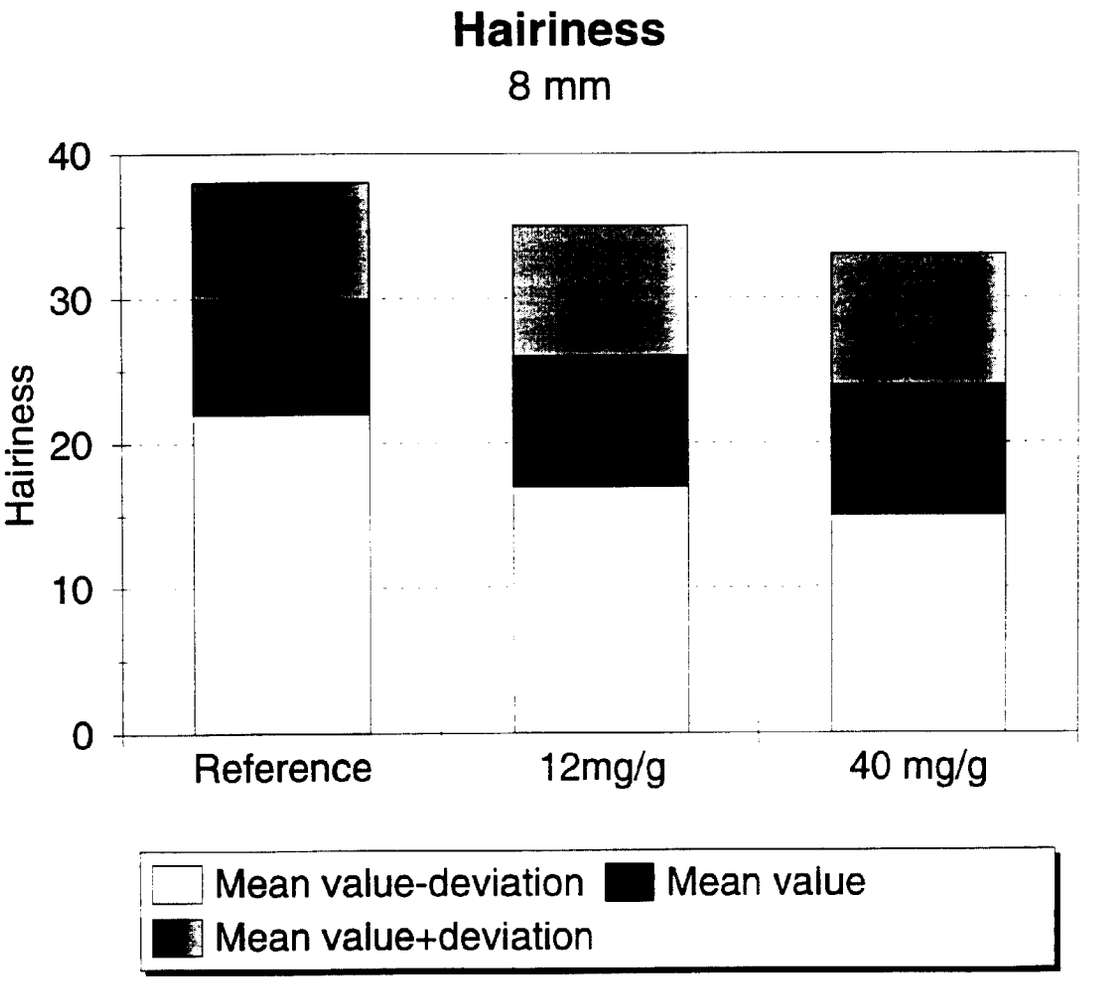
Hairiness
8 mm
40
30
20
10
0
Hairiness
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG. 7

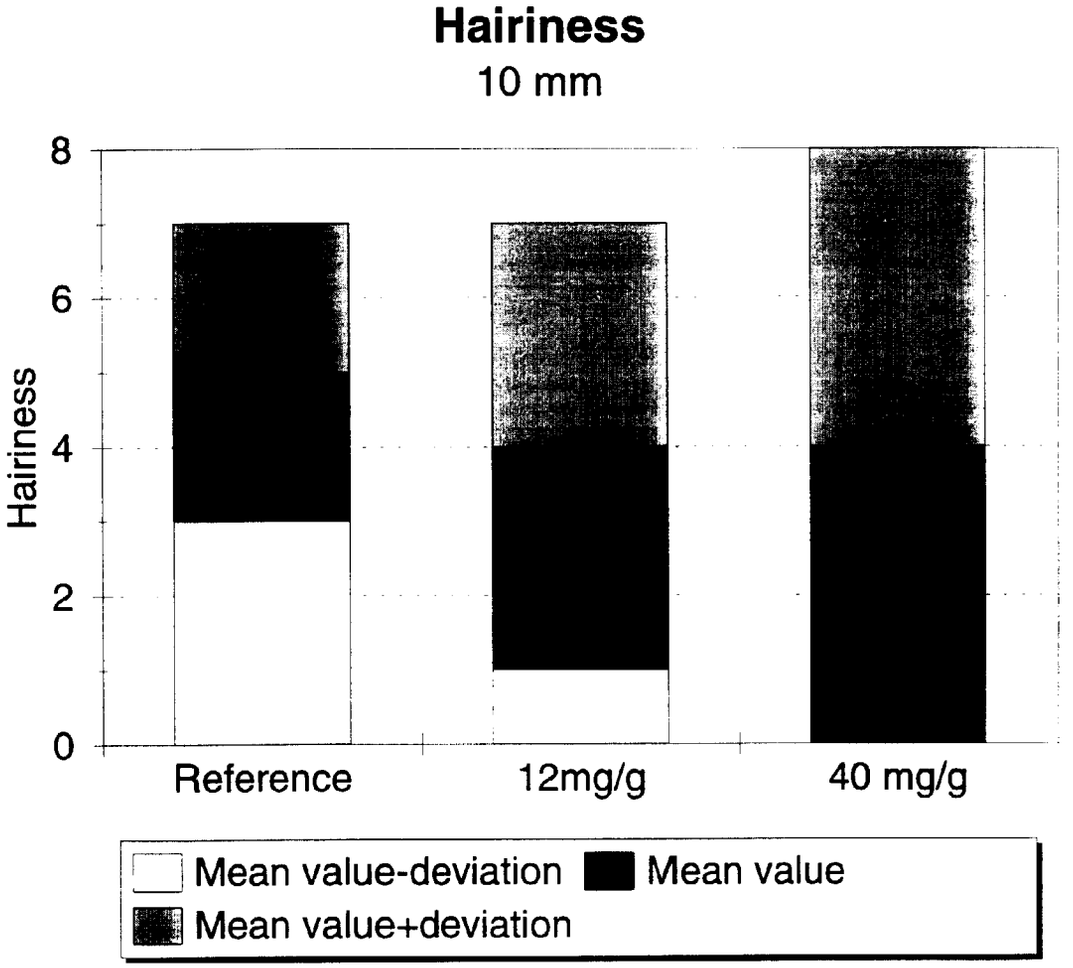
Hairiness
10 mm
Hairiness
8
6
4
2
0
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG. 8

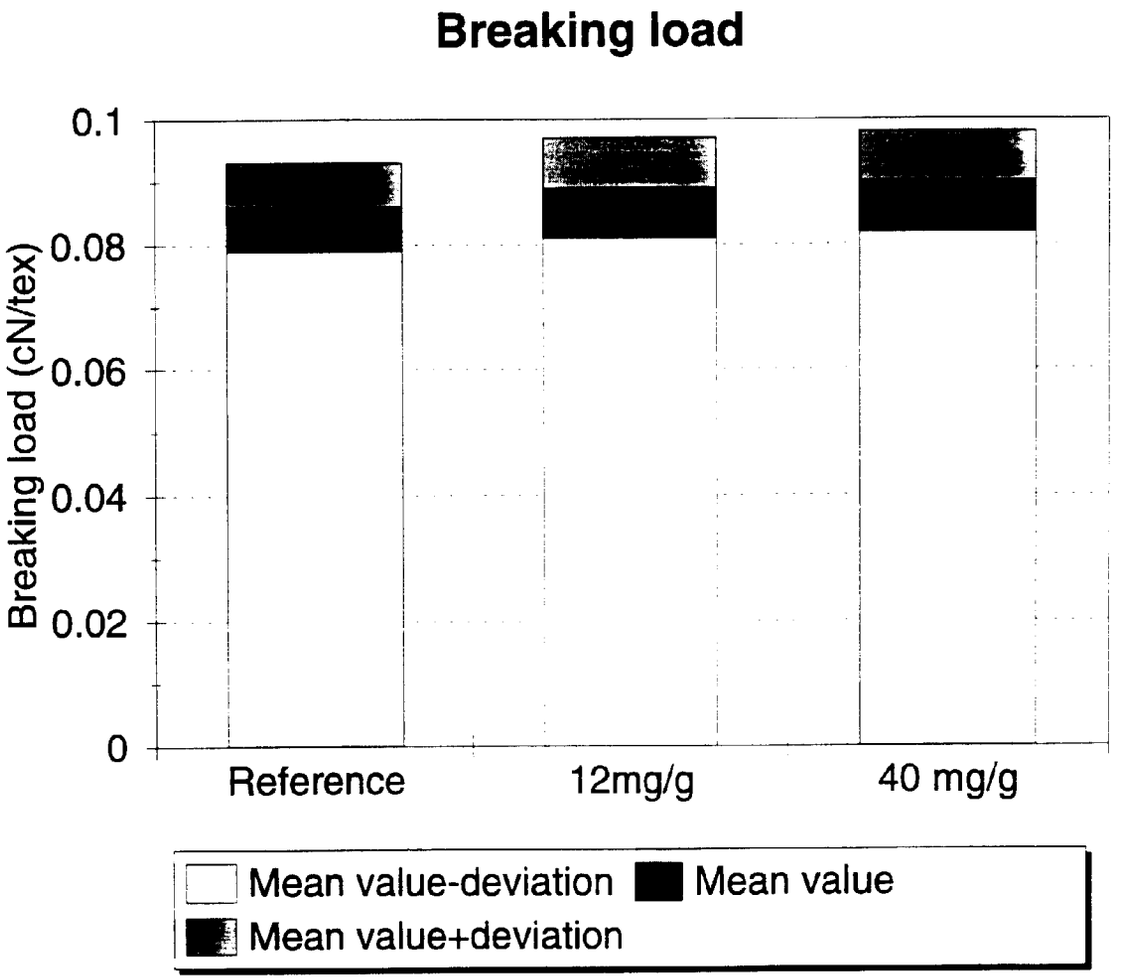
Breaking load
Breaking load (cN/tex)
0.1
0.08
0.06
0.04
0.02
0
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG. 9

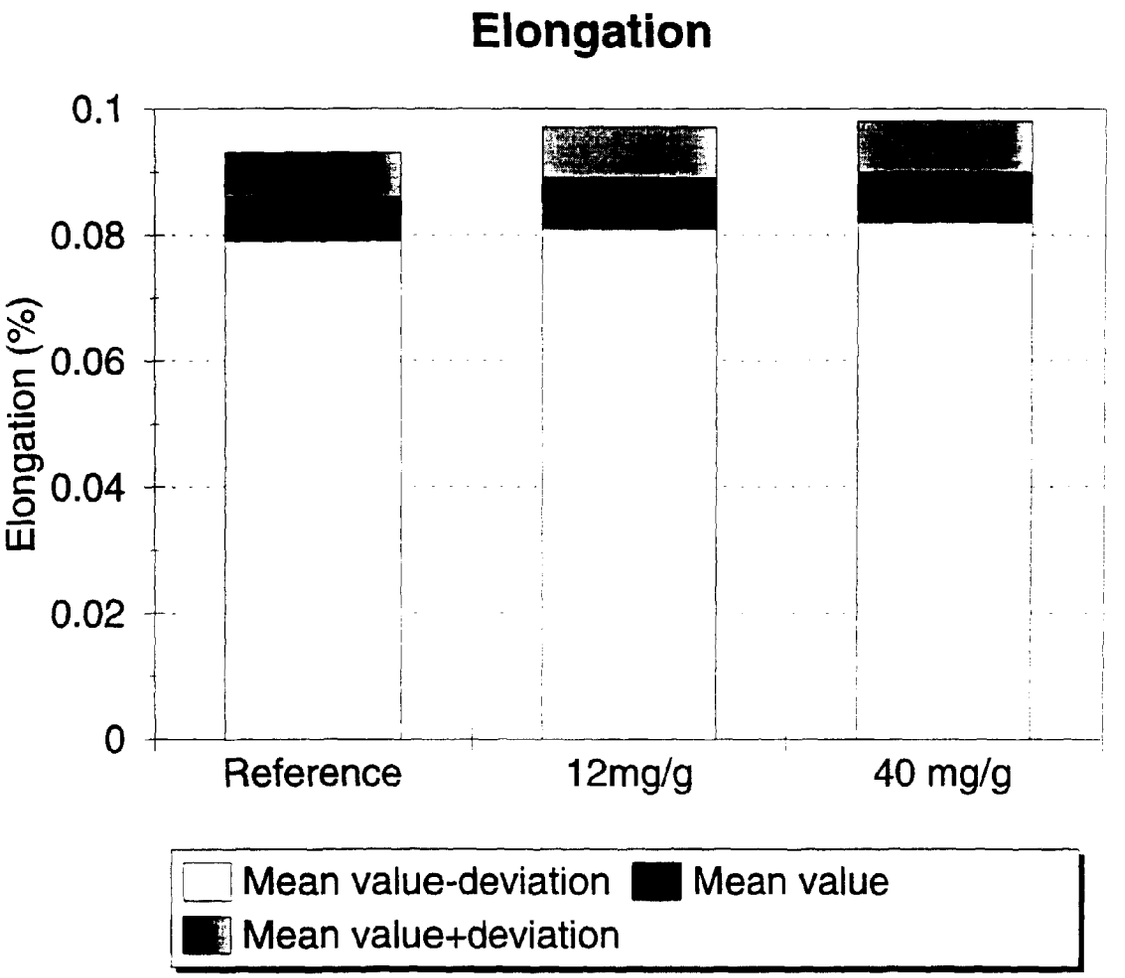
Elongation
0.1
0.08
0.06
0.04
0.02
0
Elongation (%)
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG 10

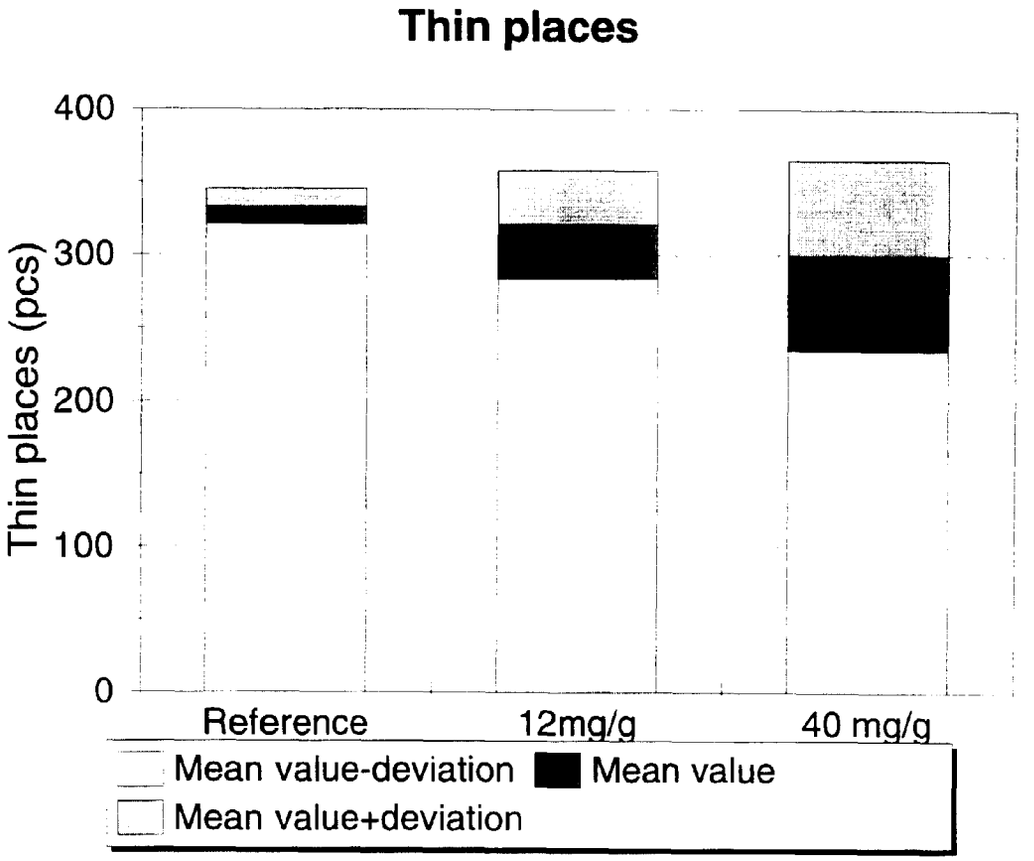
Thin places
400
300
200
100
0
Thin places (pcs)
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG. 11

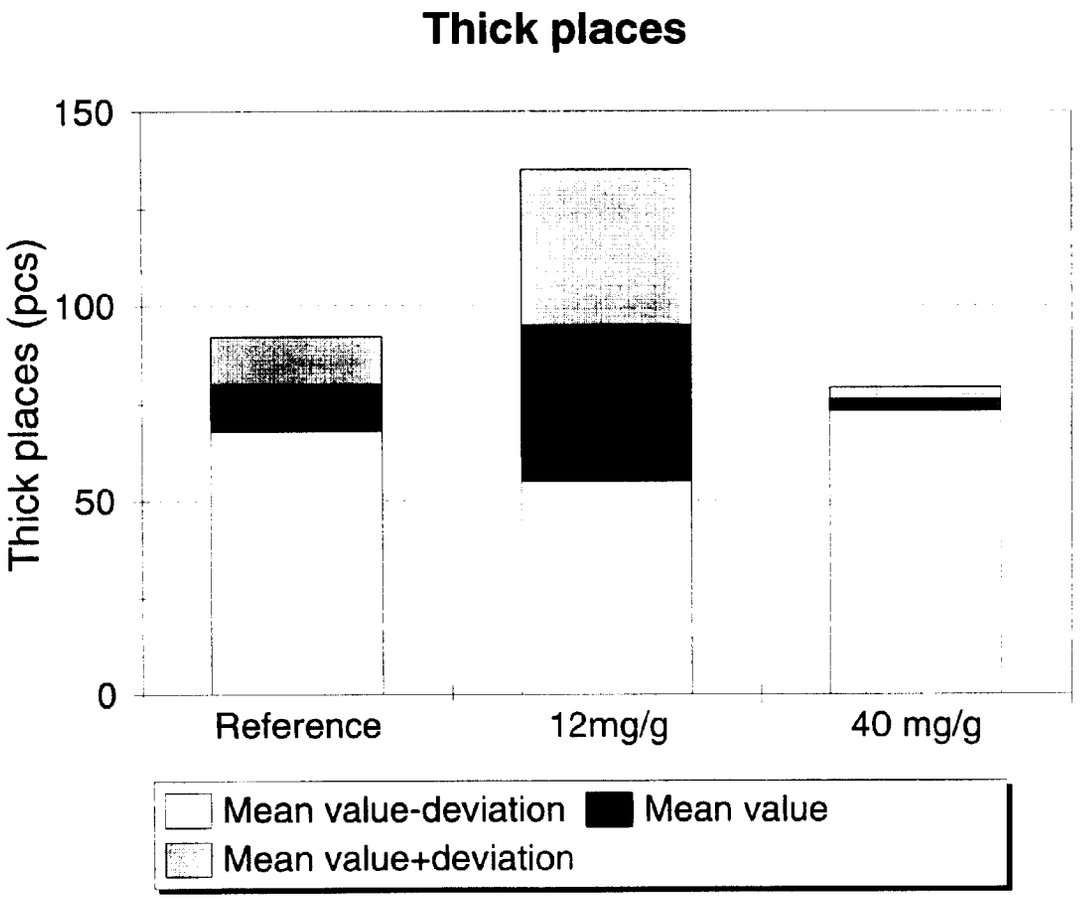
Thick places
150
100
50
0
Thick places (pcs)
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG. 12

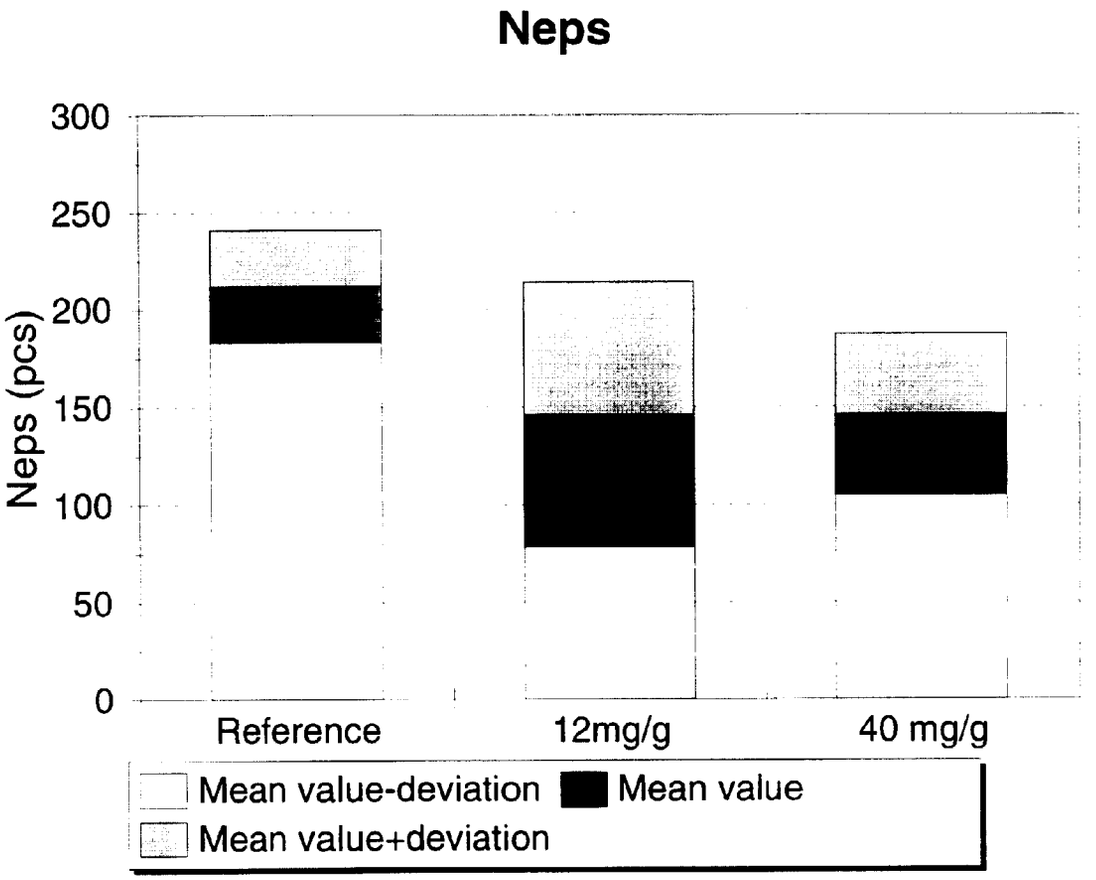
Neps
300
250
200
150
100
50
0
Neps (pcs)
Reference
12mg/g
40 mg/g
Mean value-deviation
Mean value
Mean value+deviation

FIG. 13

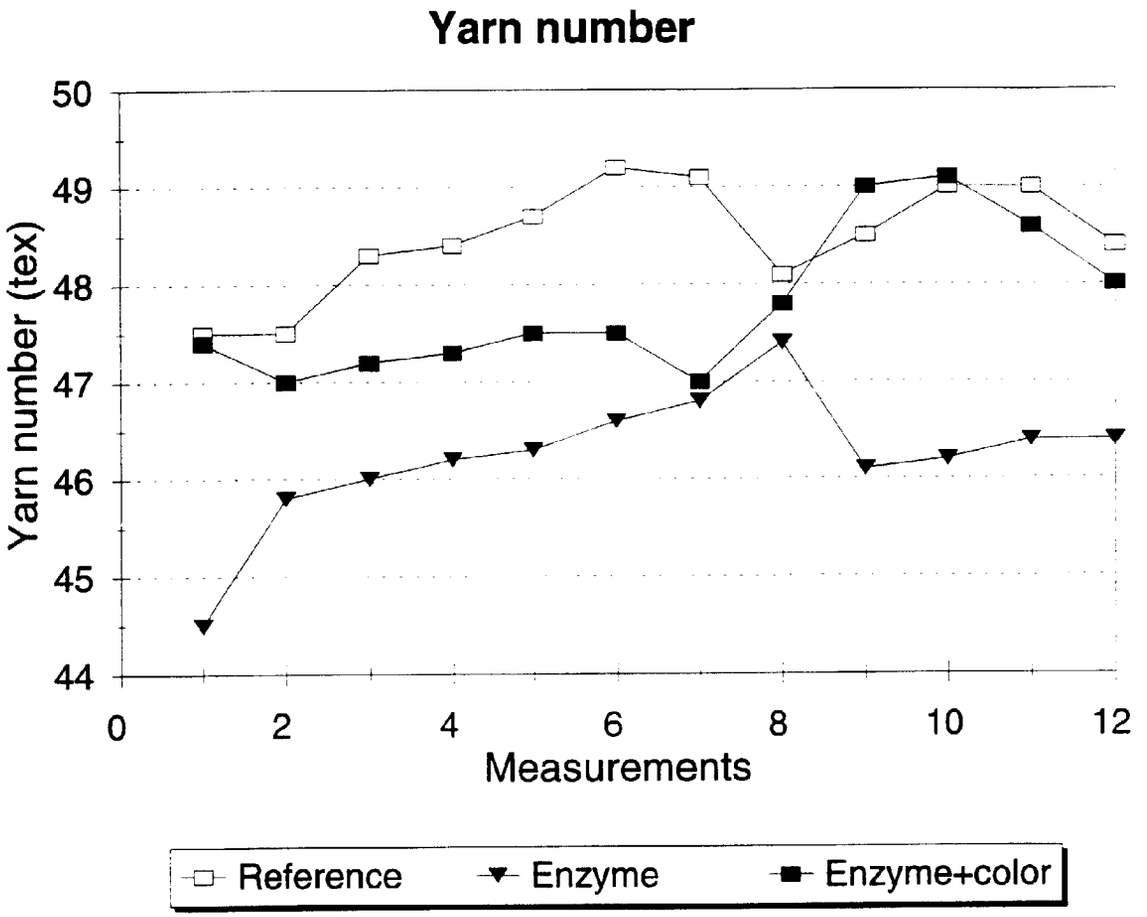
Yarn number
Yarn number (tex)
50
49
48
47
46
45
44
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 14

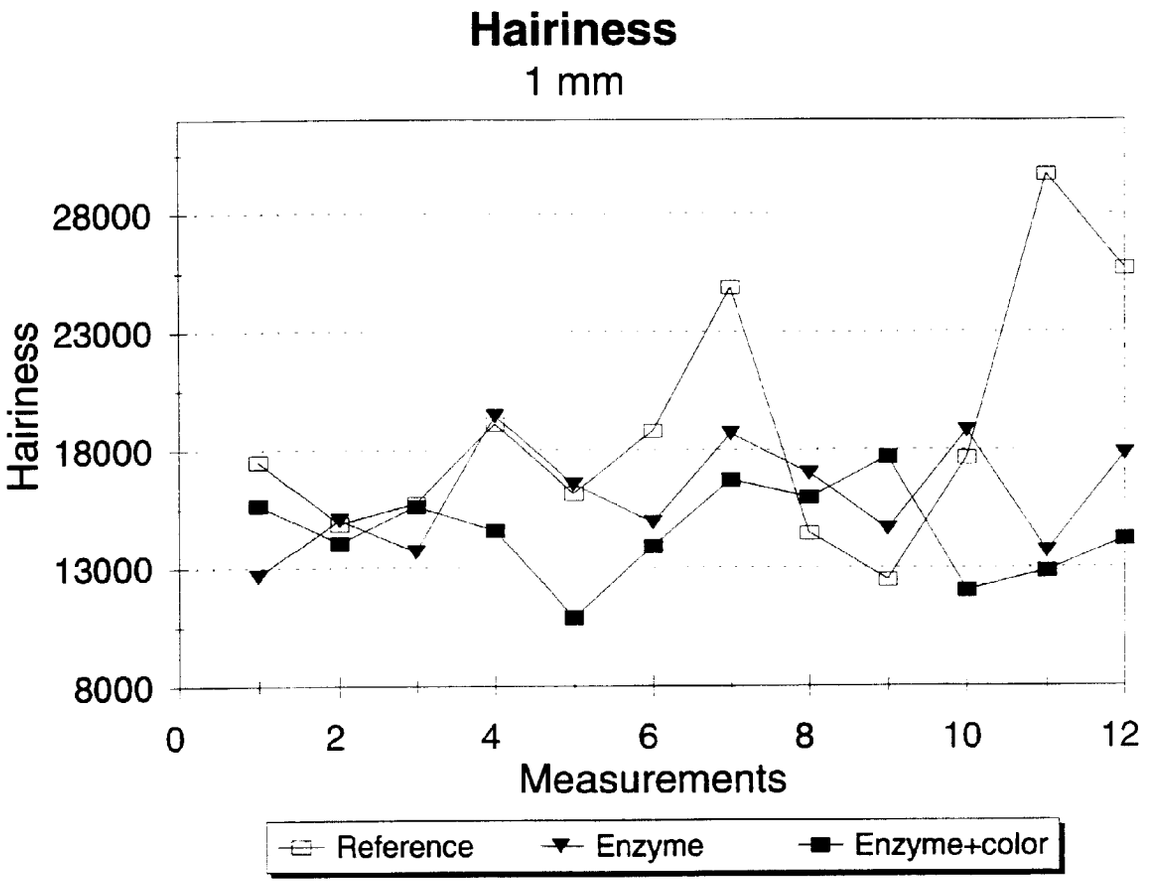
Hairiness
1 mm
28000
23000
18000
13000
8000
Hairiness
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 15

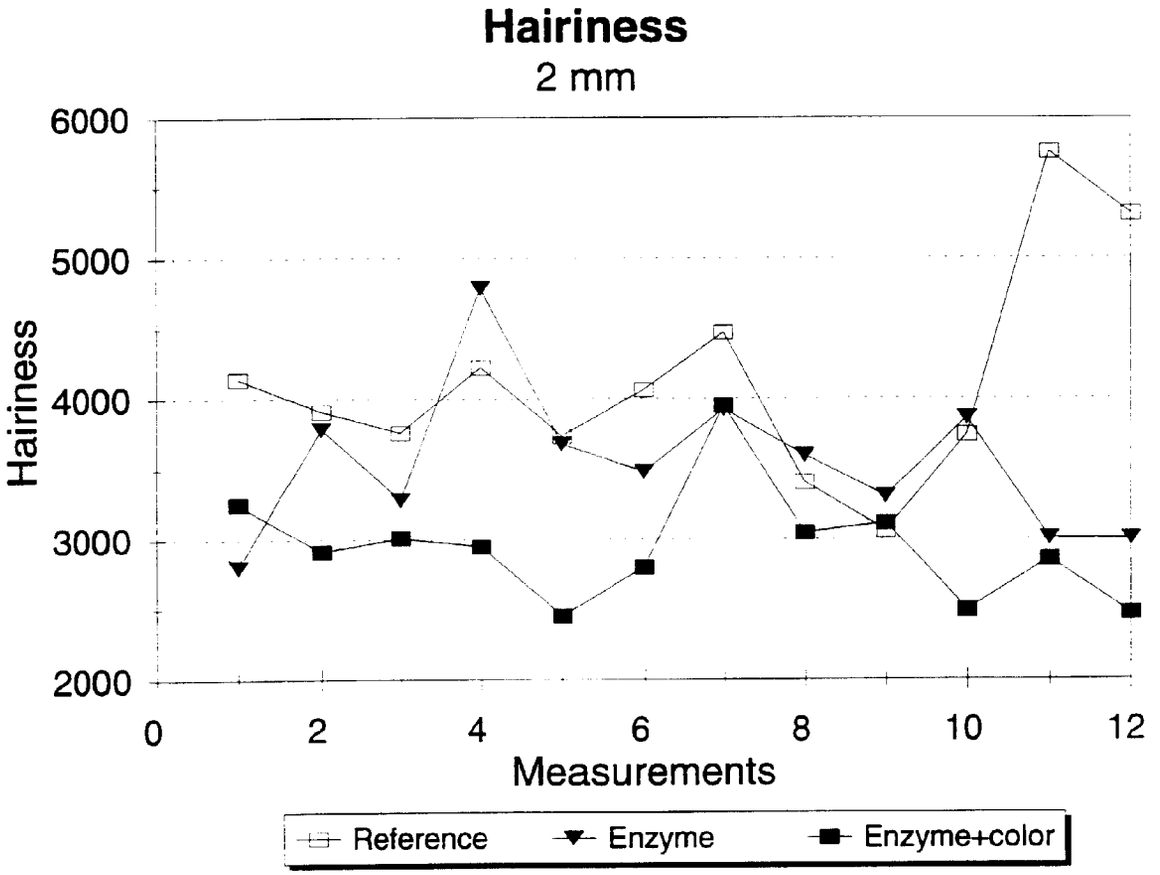
Hairiness
2 mm
6000
5000
4000
3000
2000
Hairiness
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 16

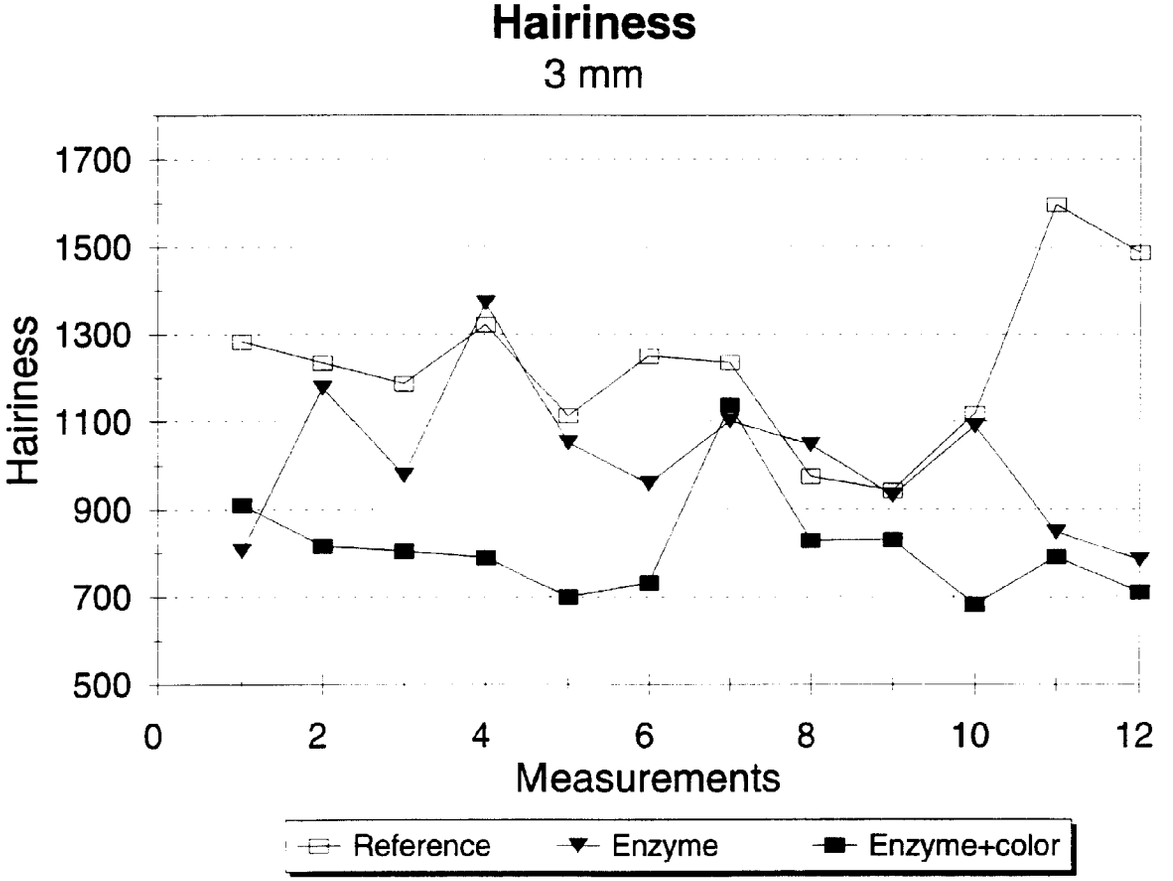
Hairiness
3 mm
1700
1500
1300
1100
900
700
500
Hairiness
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 17

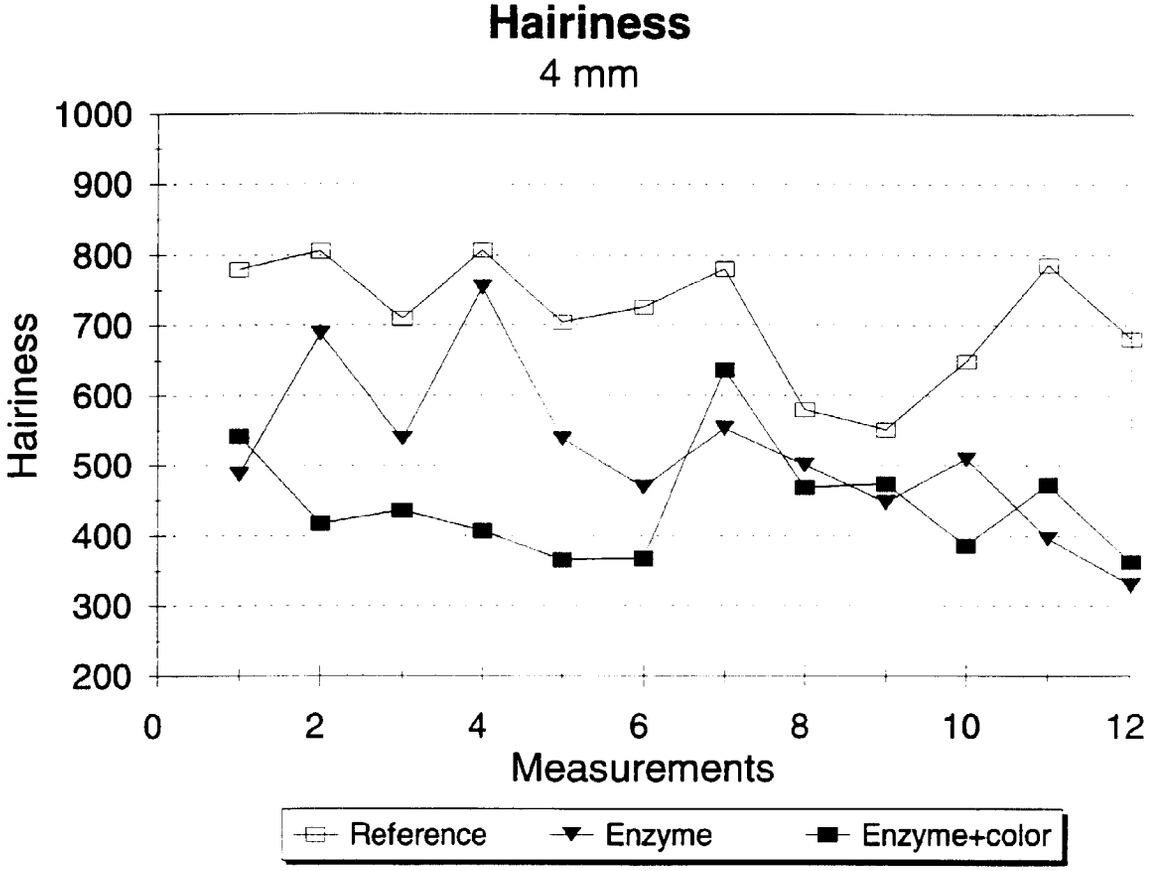
Hairiness
4 mm
1000
900
800
700
600
500
400
300
200
Hairiness
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 18

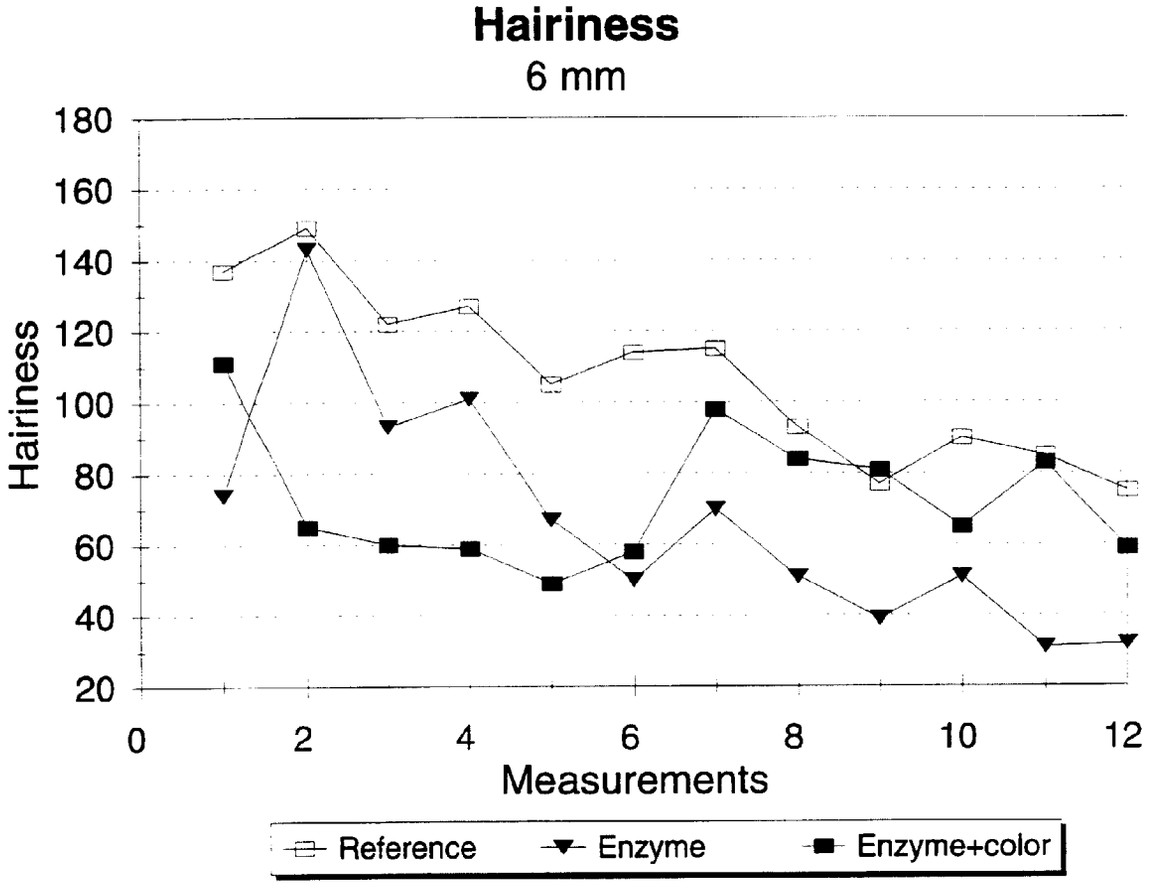
Hairiness
6 mm
180
160
140
120
100
80
60
40
20
Hairiness
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 19

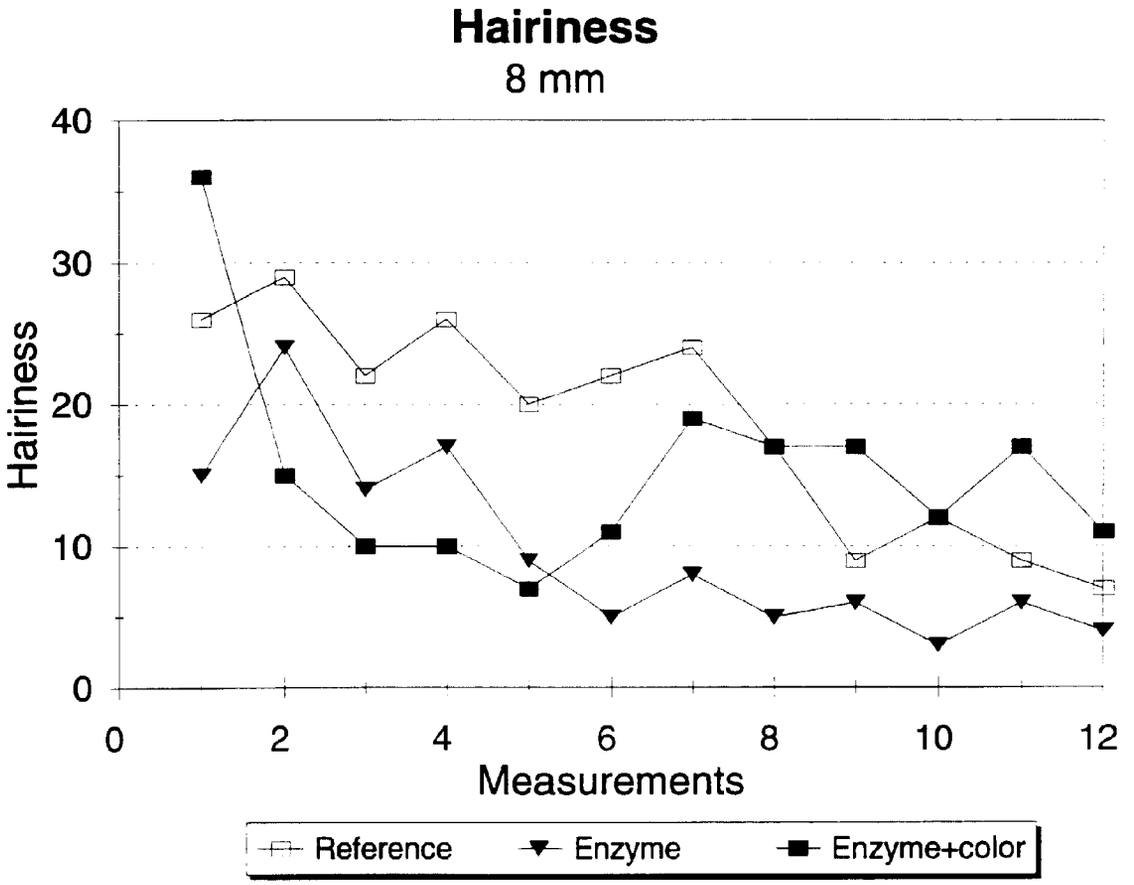
Hairiness
8 mm
40
30
20
10
0
Hairiness
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 20

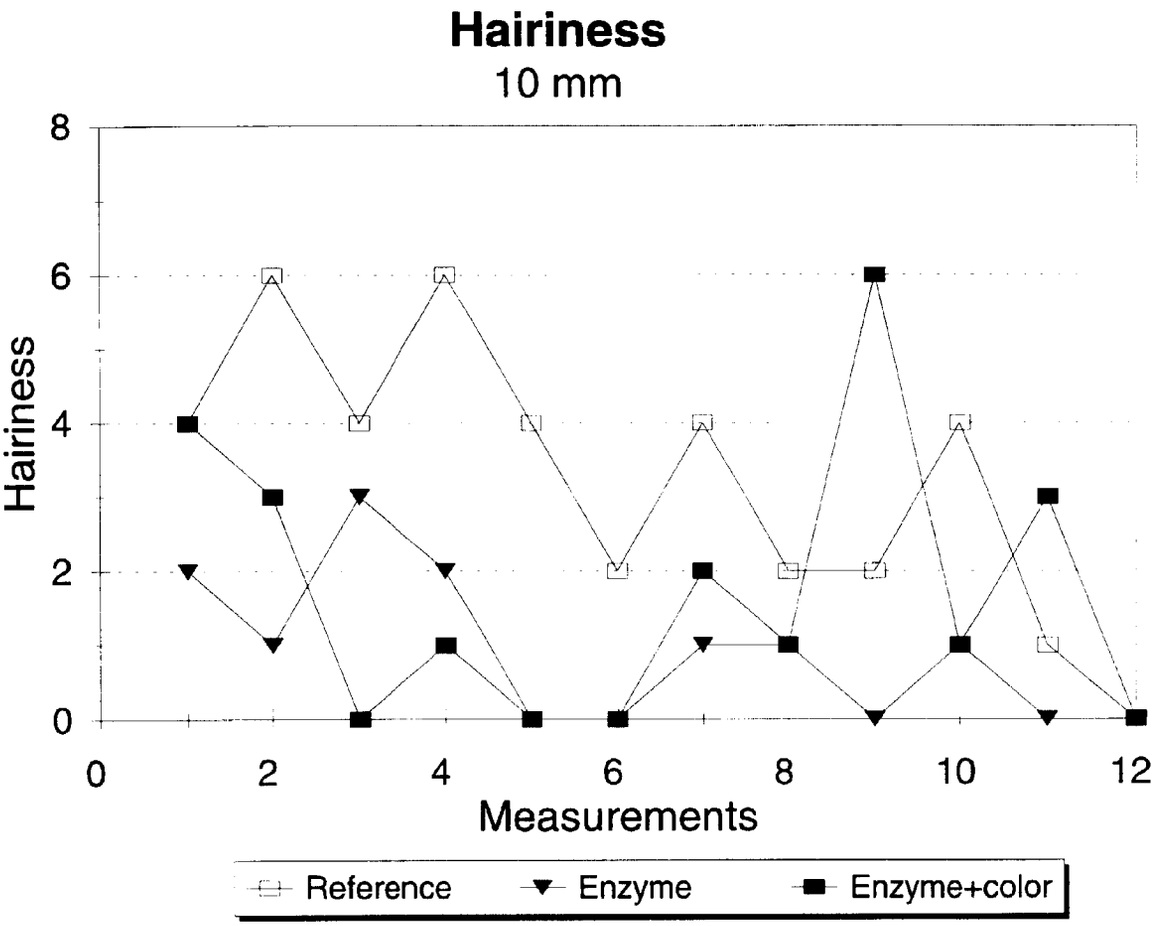
Hairiness
10 mm
Hairiness
8
6
4
2
0
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 21

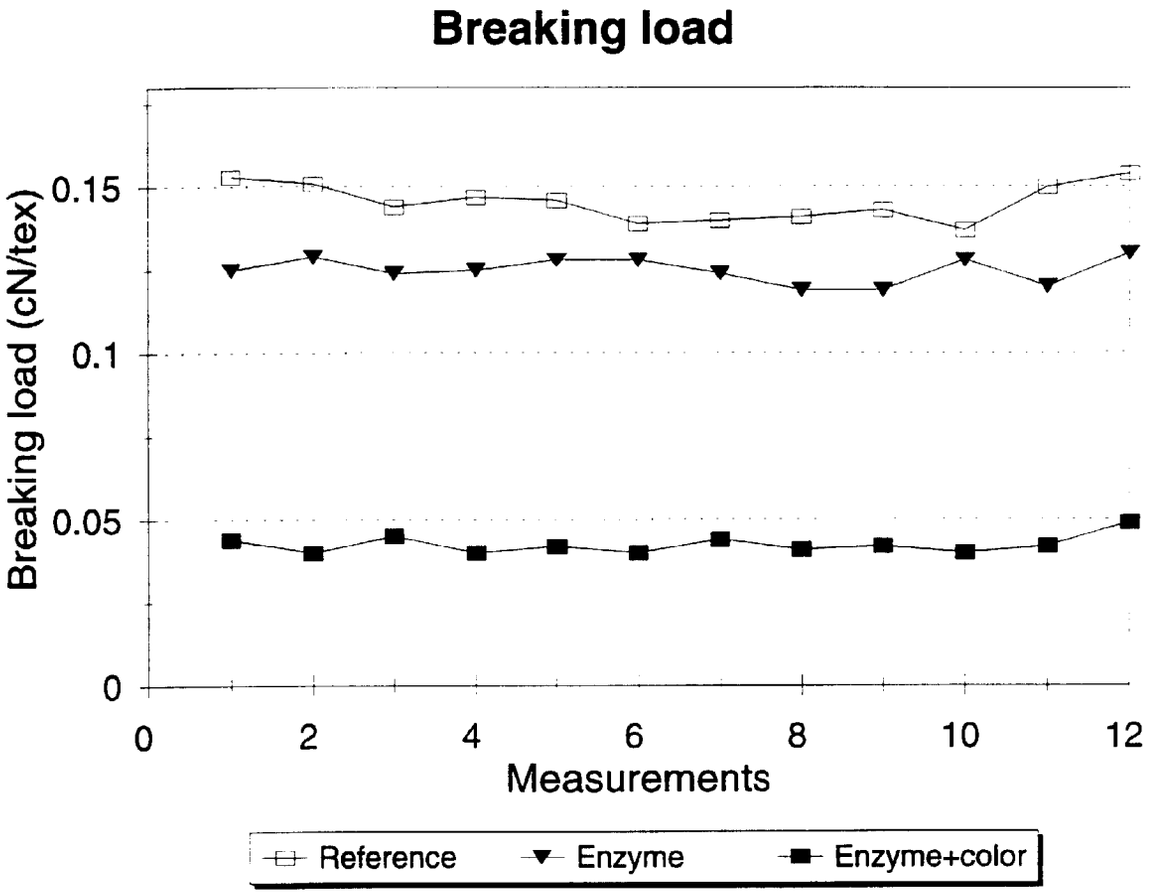
Breaking load
Breaking load (cN/tex)
0.15
0.1
0.05
0
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 22

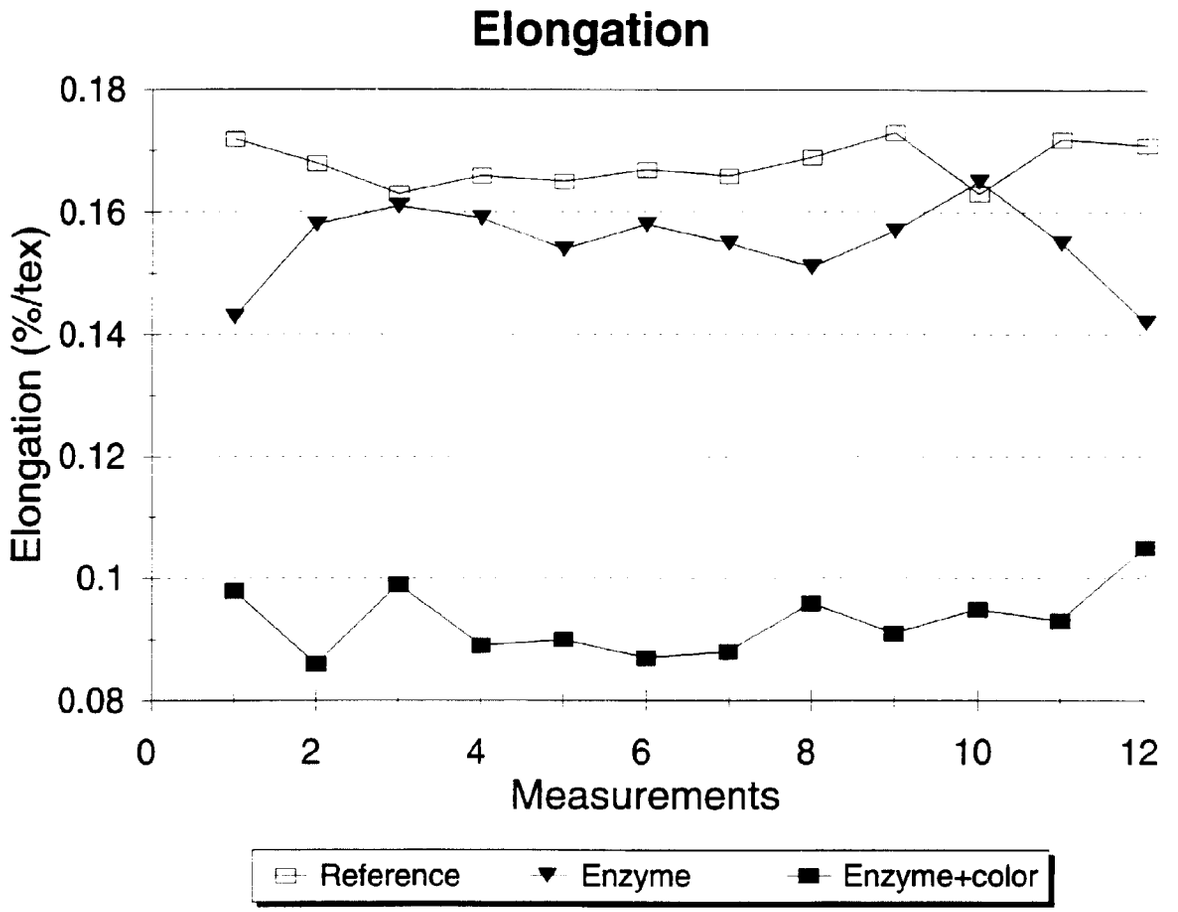
Elongation
Elongation (%/tex)
0.18
0.16
0.14
0.12
0.1
0.08
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 23

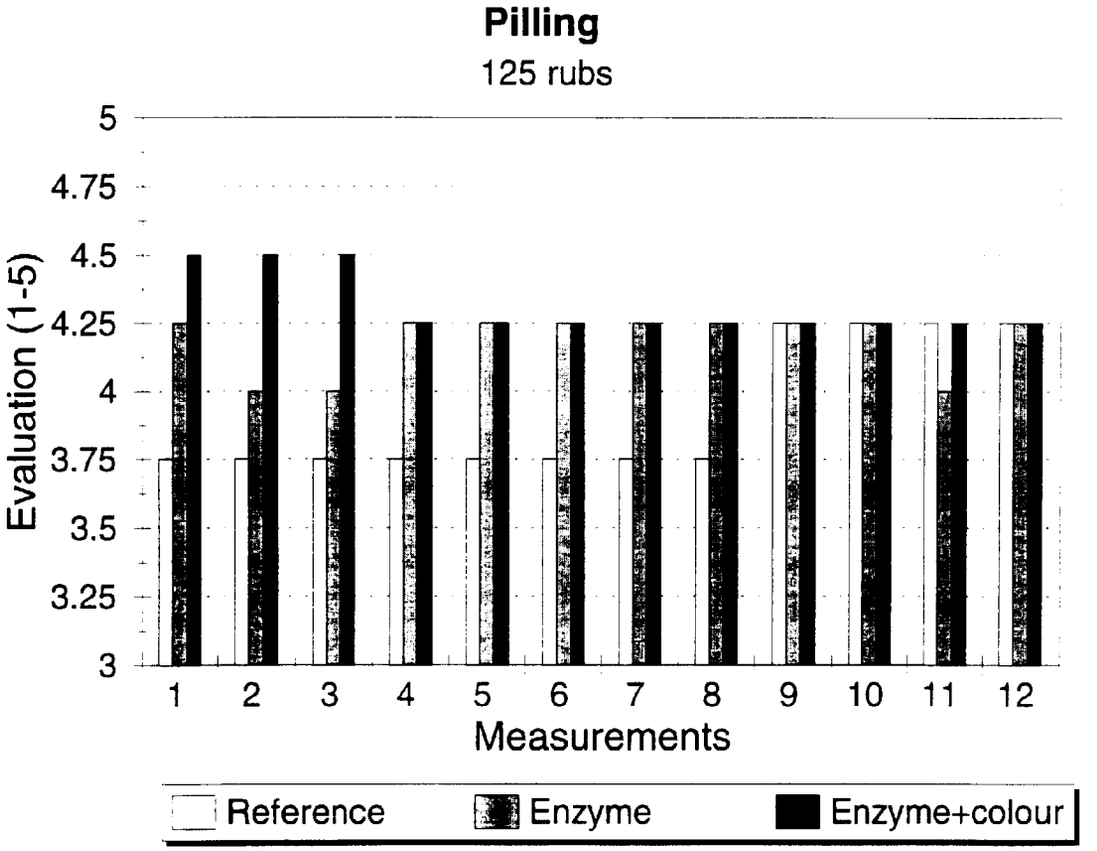
Pilling
125 rubs
Evaluation (1-5)
5
4.75
4.5
4.25
4
3.75
3.5
3.25
3
1
2
3
4
5
6
7
8
9
10
11
12
Measurements
Reference
Enzyme
Enzyme+colour

FIG. 24

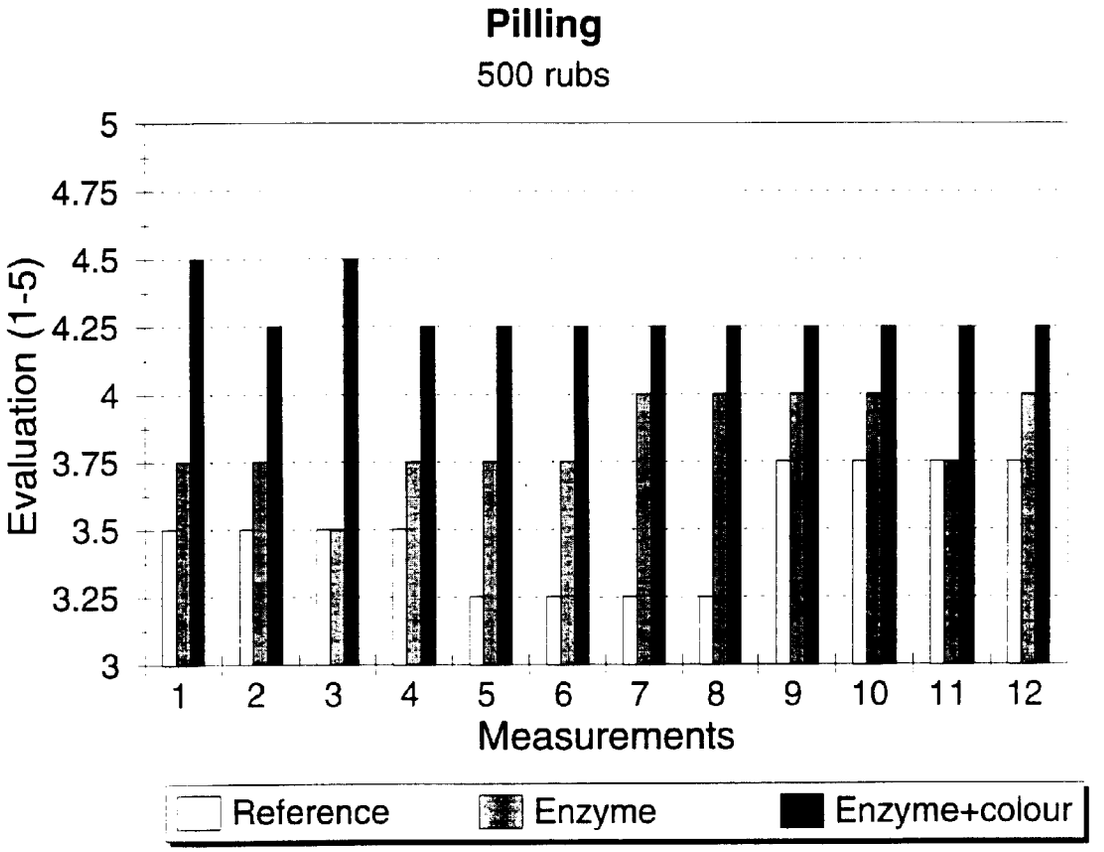
Pilling
500 rubs
Evaluation (1-5)
5
4.75
4.5
4.25
4
3.75
3.5
3.25
3
1
2
3
4
5
6
7
8
9
10
11
12
Measurements
Reference
Enzyme
Enzyme+colour

FIG. 25

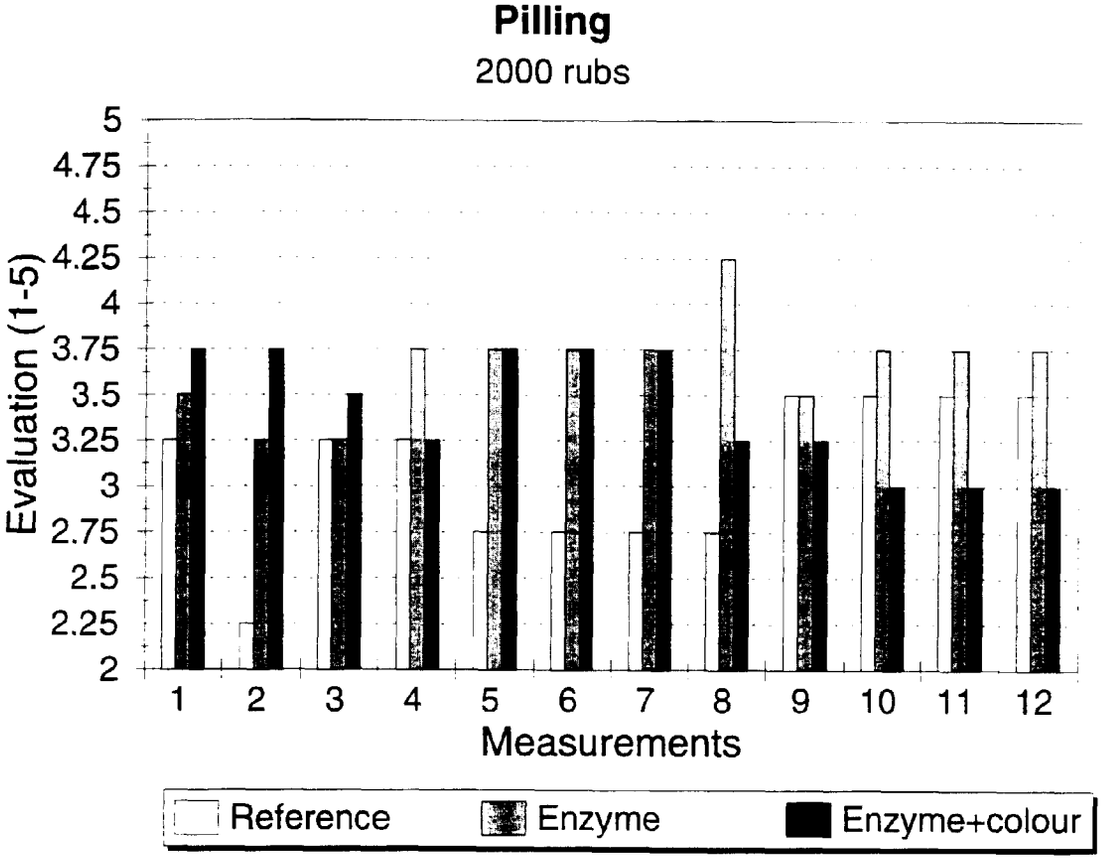
Pilling
2000 rubs
Evaluation (1-5)
5
4.75
4.5
4.25
4
3.75
3.5
3.25
3
2.75
2.5
2.25
2
1
2
3
4
5
6
7
8
9
10
11
12
Measurements
Reference
Enzyme
Enzyme+colour

FIG. 26

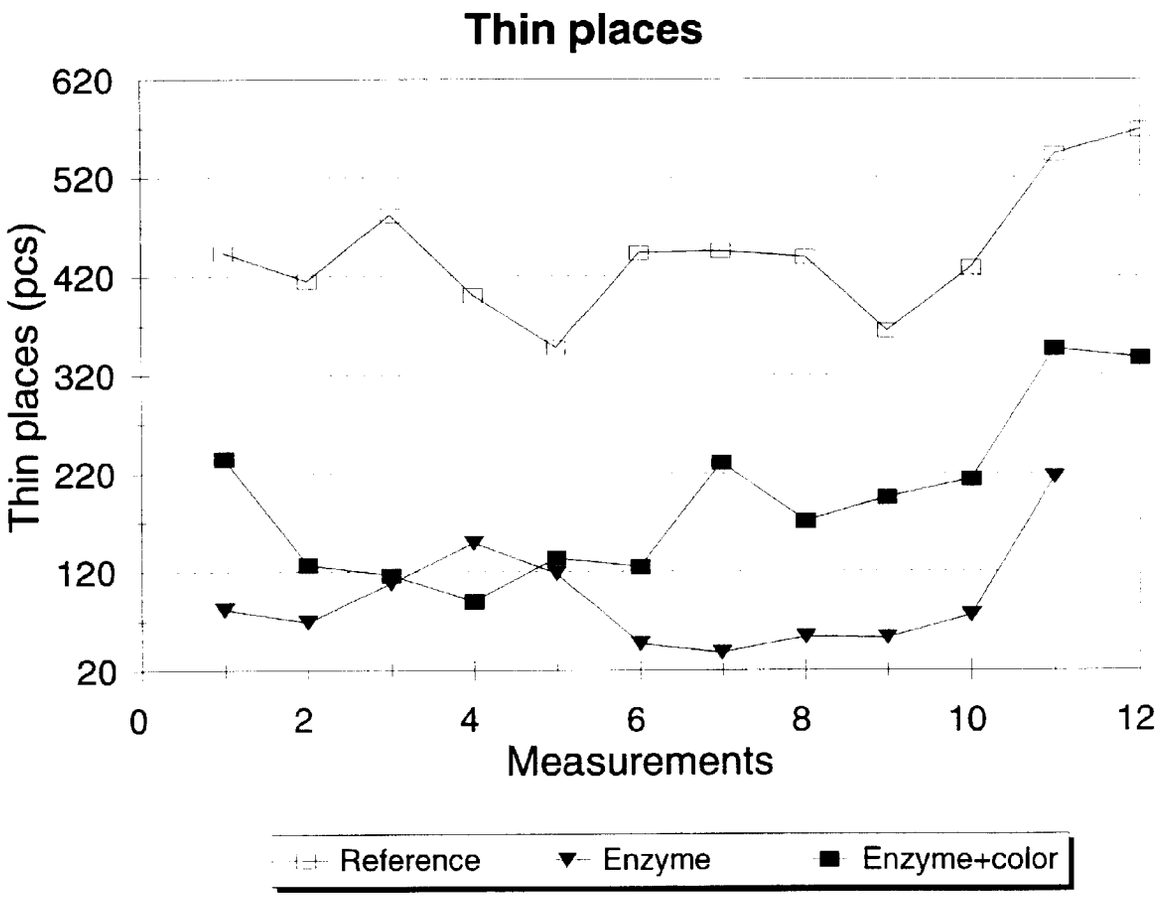
Thin places
620
520
420
320
220
120
20
Thin places (pcs)
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 27

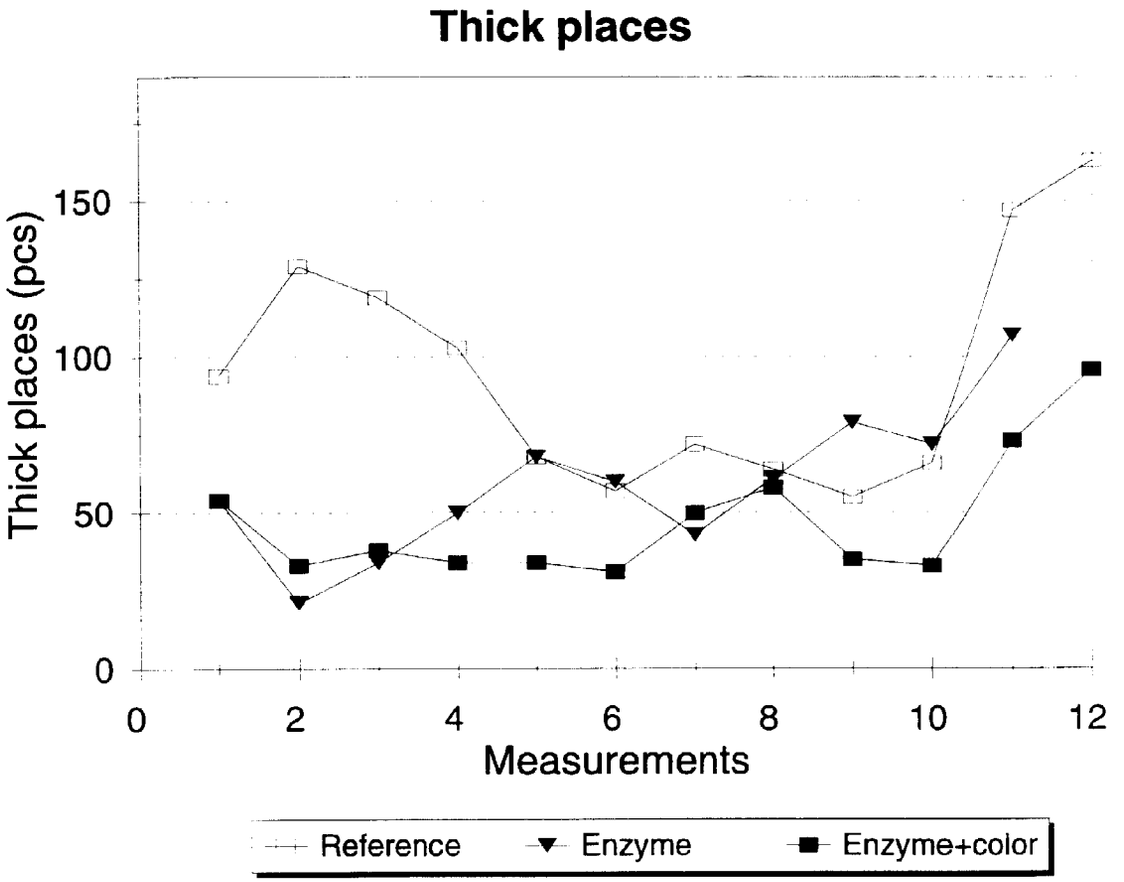
Thick places
Thick places (pcs)
150
100
50
0
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 28

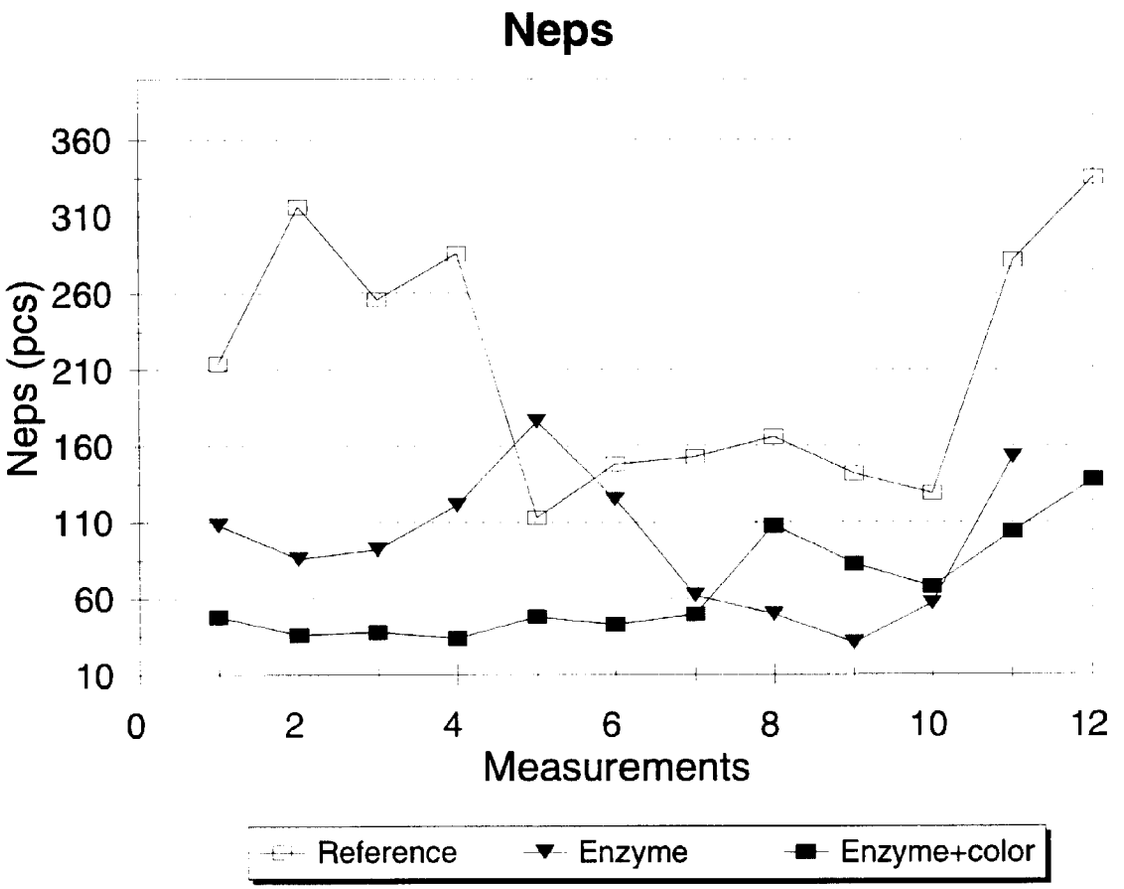
Neps
Neps (pcs)
360
310
260
210
160
110
60
10
0
2
4
6
8
10
12
Measurements
Reference
Enzyme
Enzyme+color

FIG. 29

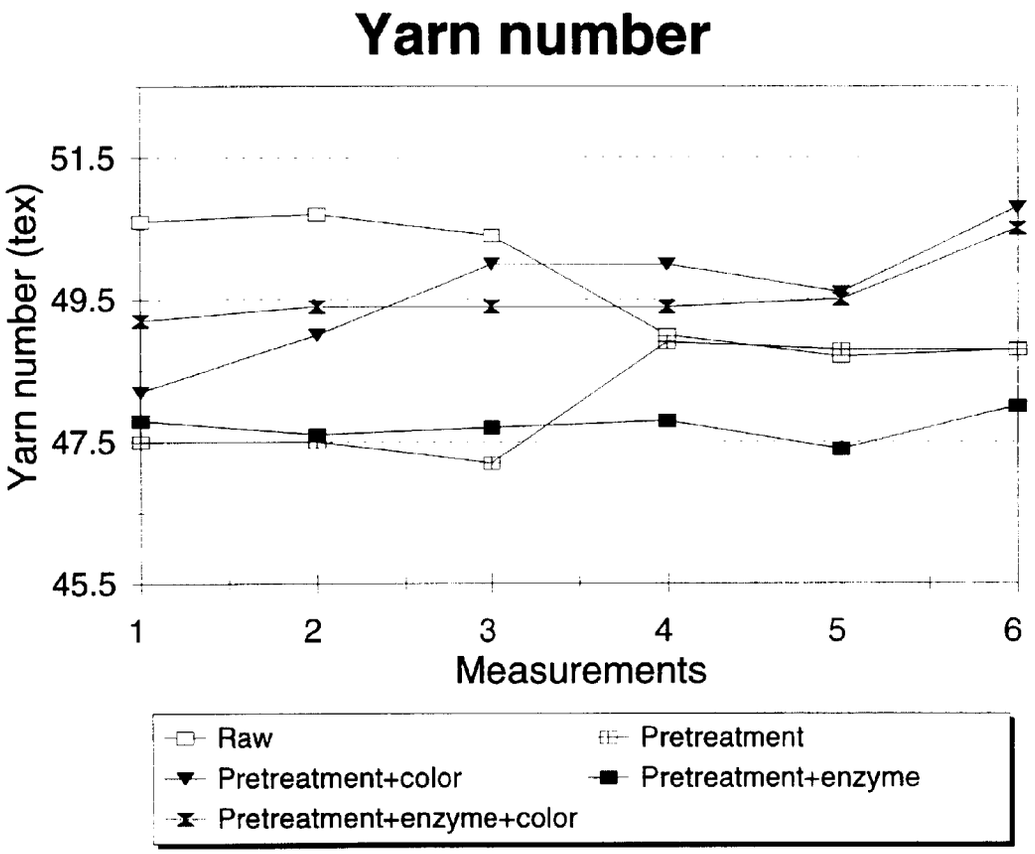
Yarn number
Yarn number (tex)
51.5
49.5
47.5
45.5
1
2
3
4
5
6
Measurements
Raw
Pretreatment
Pretreatment+color
Pretreatment+enzyme
Pretreatment+enzyme+color

FIG. 30

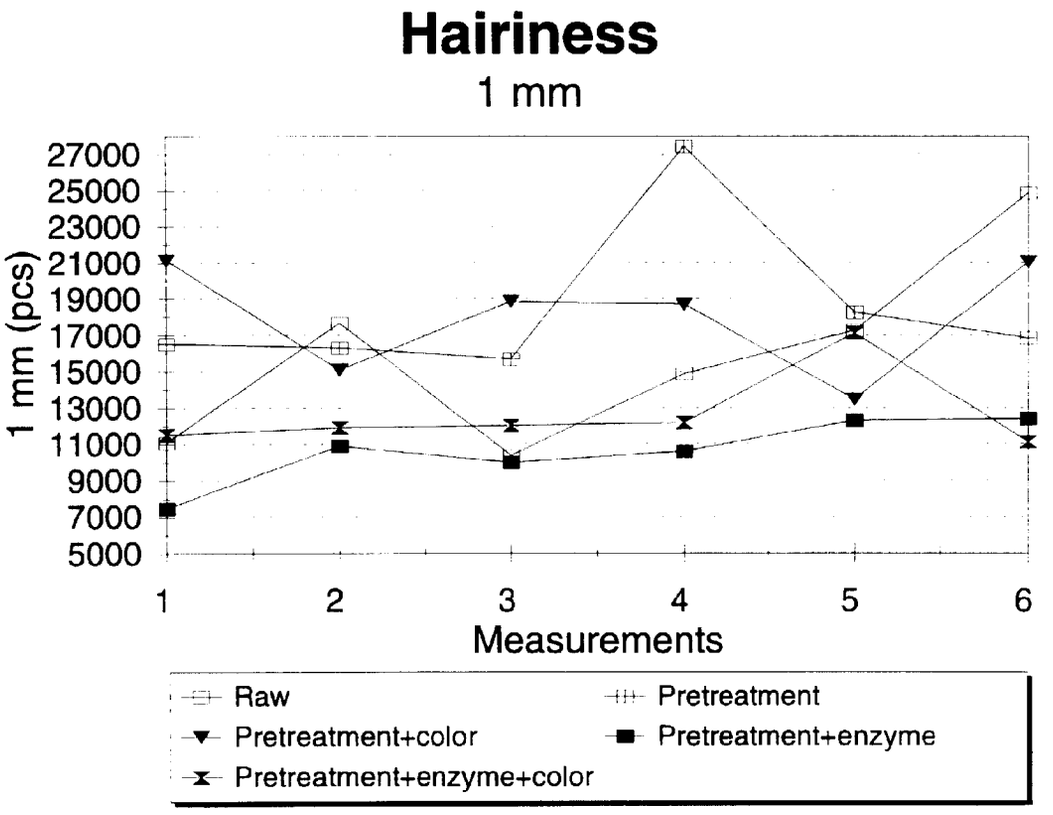
Hairiness
1 mm
1 mm (pcs)
27000
25000
23000
21000
19000
17000
15000
13000
11000
9000
7000
5000
1
2
3
4
5
6
Measurements
Raw
Pretreatment
Pretreatment+color
Pretreatment+enzyme
Pretreatment+enzyme+color

FIG. 31

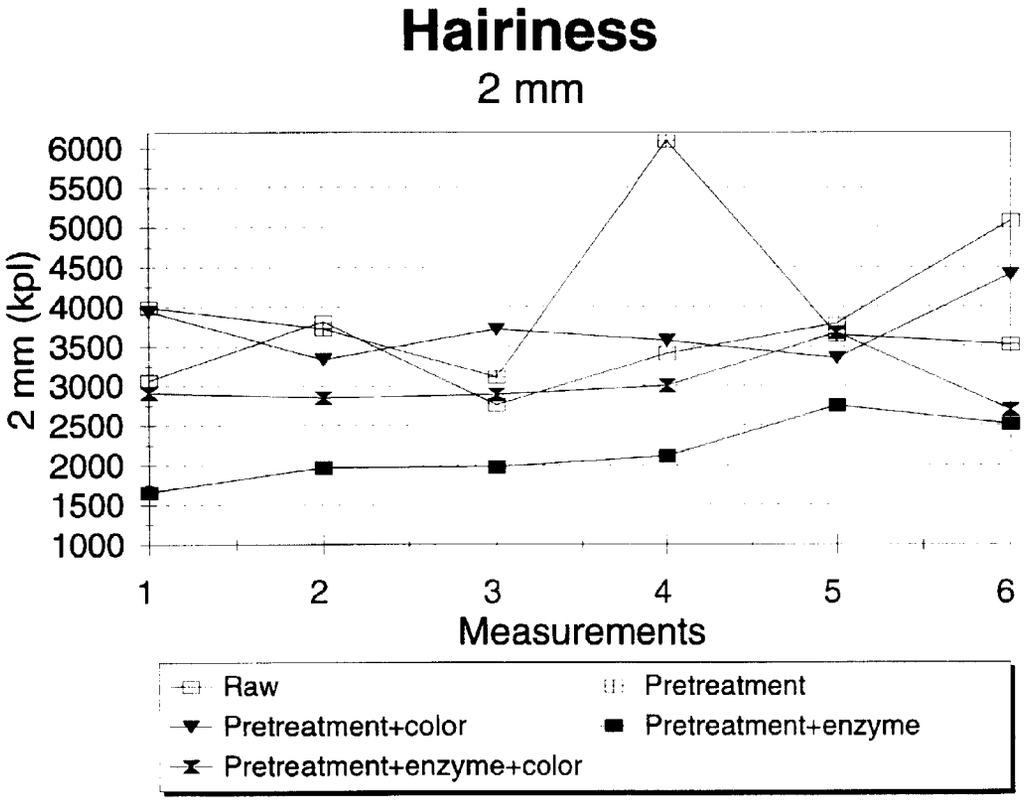
Hairiness
2 mm
2 mm (kpl)
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
1
2
3
4
5
6
Measurements
Raw
Pretreatment
Pretreatment+color
Pretreatment+enzyme
Pretreatment+enzyme+color

FIG. 32

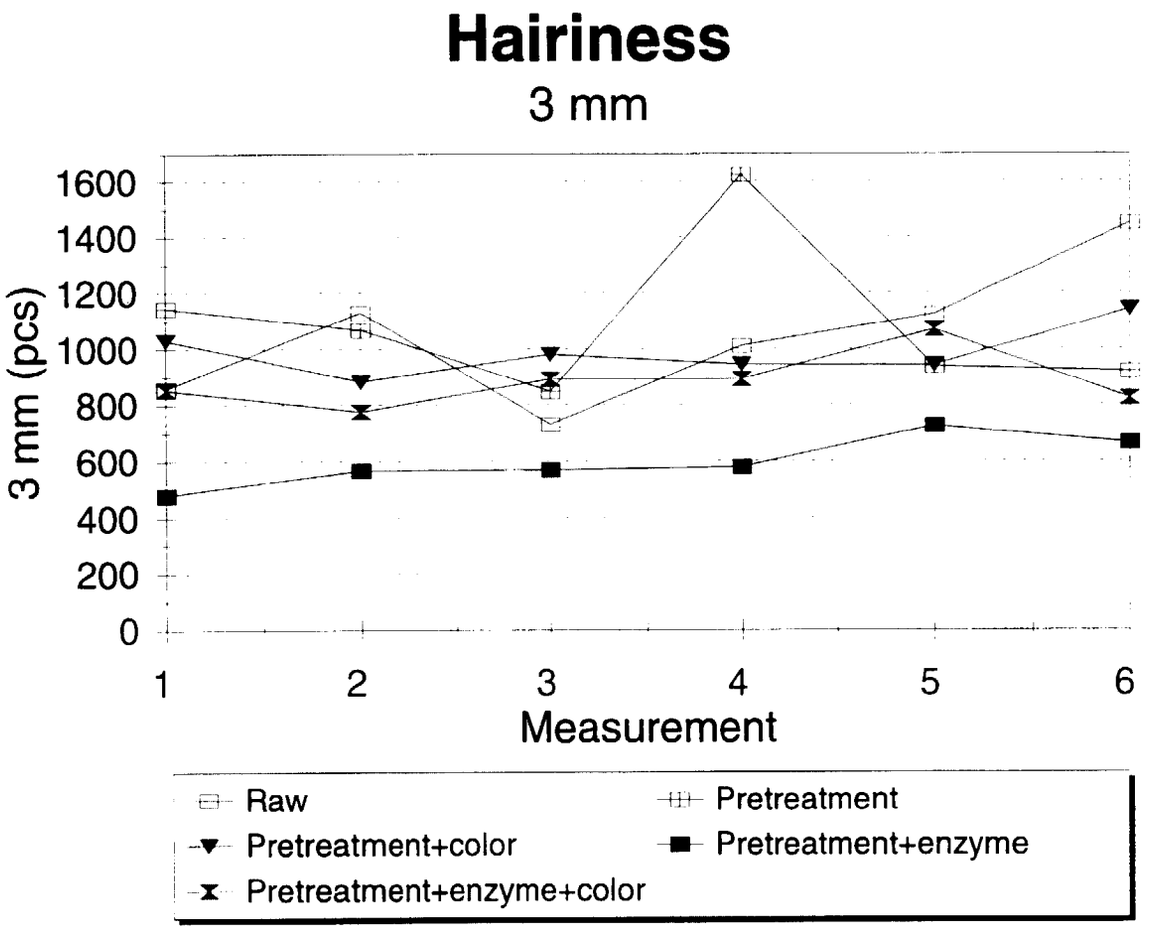
Hairiness
3 mm
3 mm (pcs)
1600
1400
1200
1000
800
600
400
200
0
1
2
3
4
5
6
Measurement
Raw
Pretreatment
Pretreatment+color
Pretreatment+enzyme
Pretreatment+enzyme+color

FIG. 33

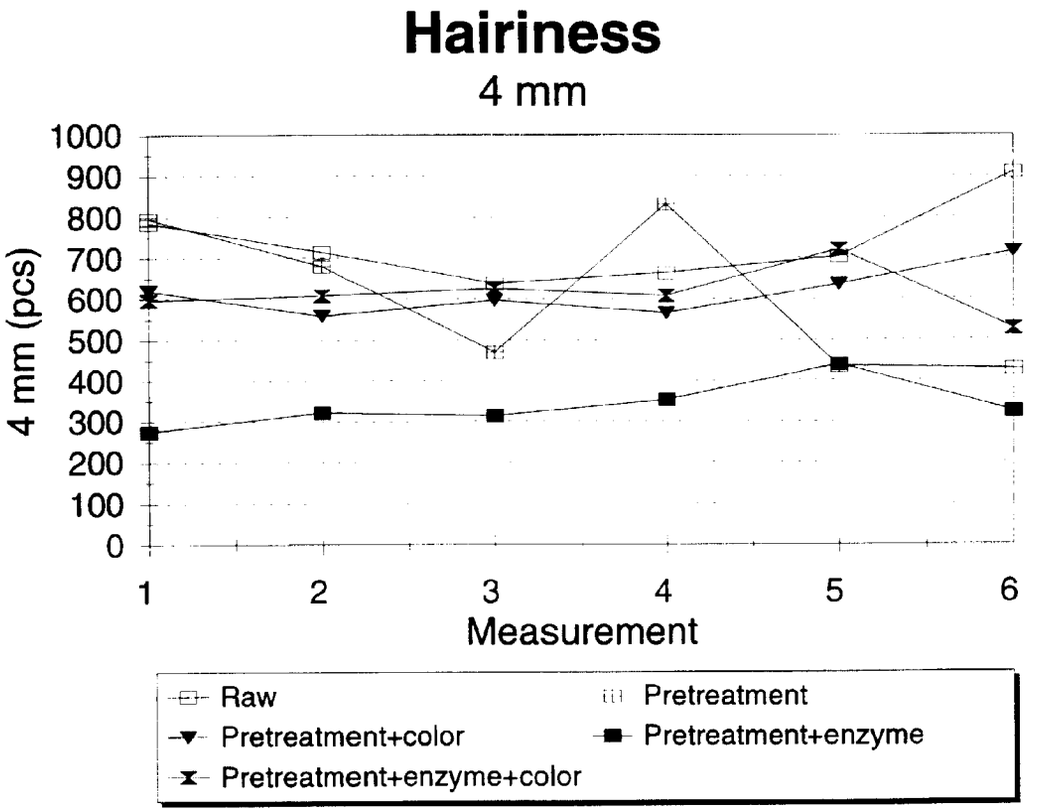
Hairiness
4 mm
4 mm (pcs)
1000
900
800
700
600
500
400
300
200
100
0
1
2
3
4
5
6
Measurement
Raw
Pretreatment
Pretreatment+color
Pretreatment+enzyme
Pretreatment+enzyme+color

FIG. 34

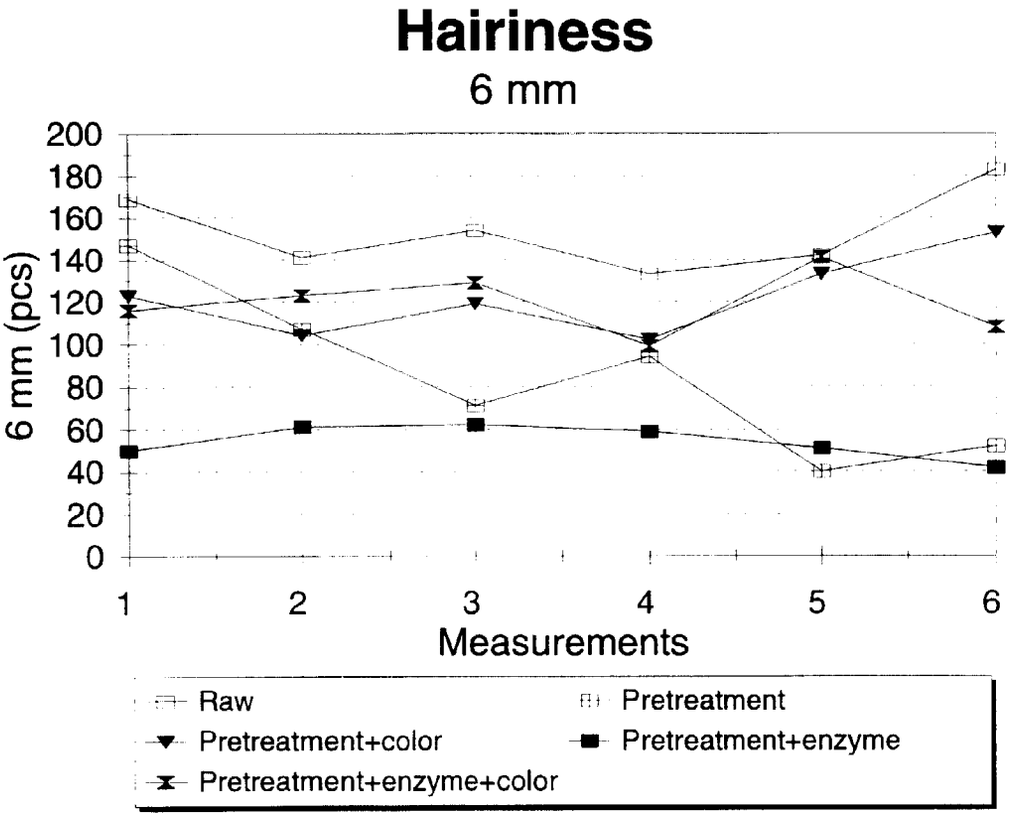
Hairiness
6 mm
6 mm (pcs)
200
180
160
140
120
100
80
60
40
20
0
1
2
3
4
5
6
Measurements
Raw
Pretreatment
Pretreatment+color
Pretreatment+enzyme
Pretreatment+enzyme+color

FIG. 35

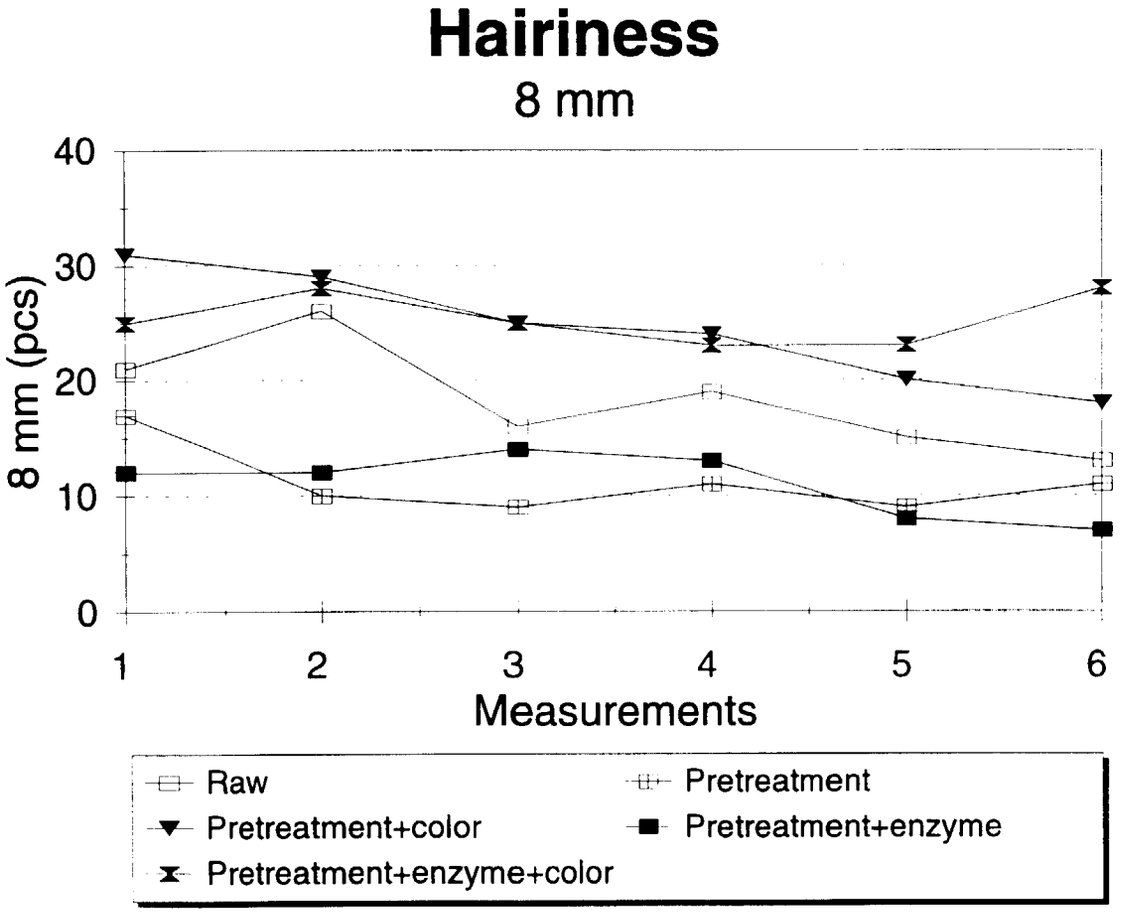
Hairiness
8 mm
40
30
20
10
0
8 mm (pcs)
1
2
3
4
5
6
Measurements
Raw
Pretreatment
Pretreatment+color
Pretreatment+enzyme
Pretreatment+enzyme+color

FIG. 36

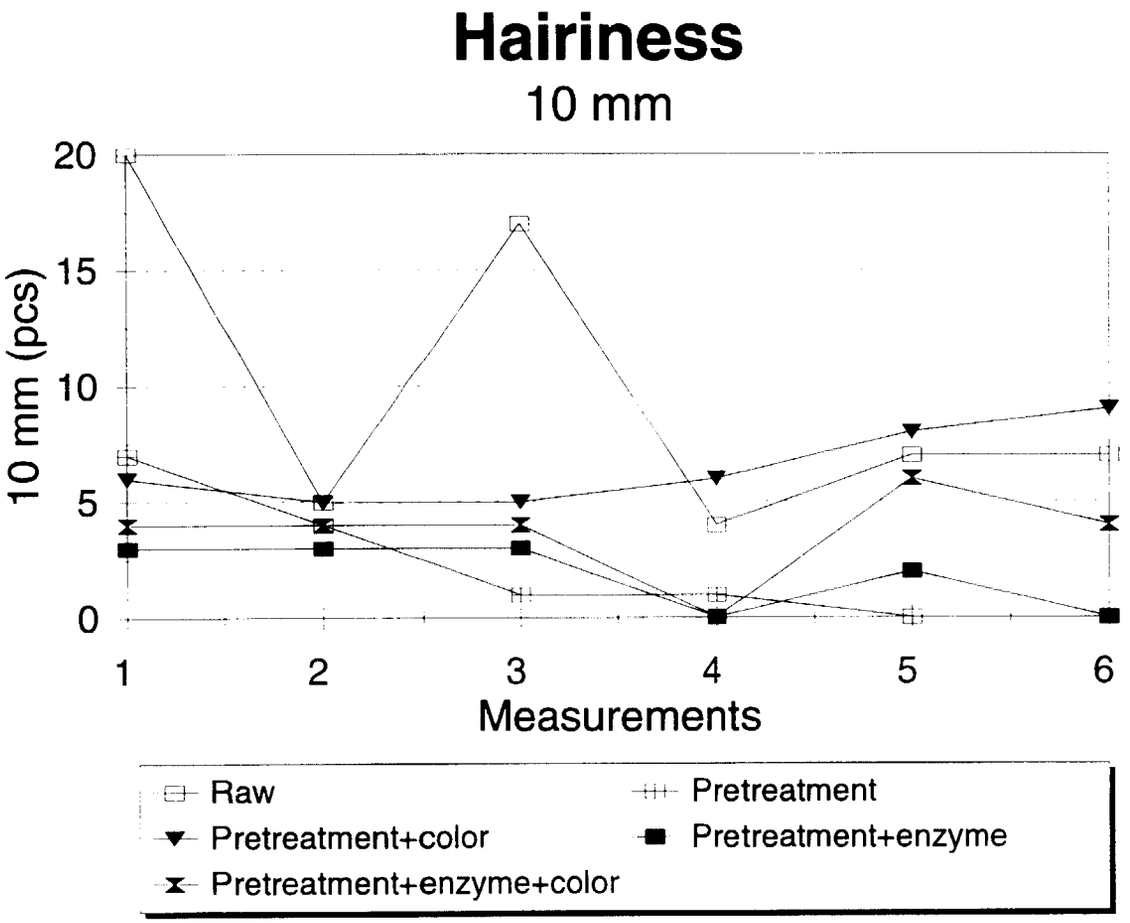
Hairiness
10 mm
20
15
10
5
0
10 mm (pcs)
1
2
3
4
5
6
Measurements
Raw
Pretreatment
Pretreatment+color
Pretreatment+enzyme
Pretreatment+enzyme+color

FIG. 37

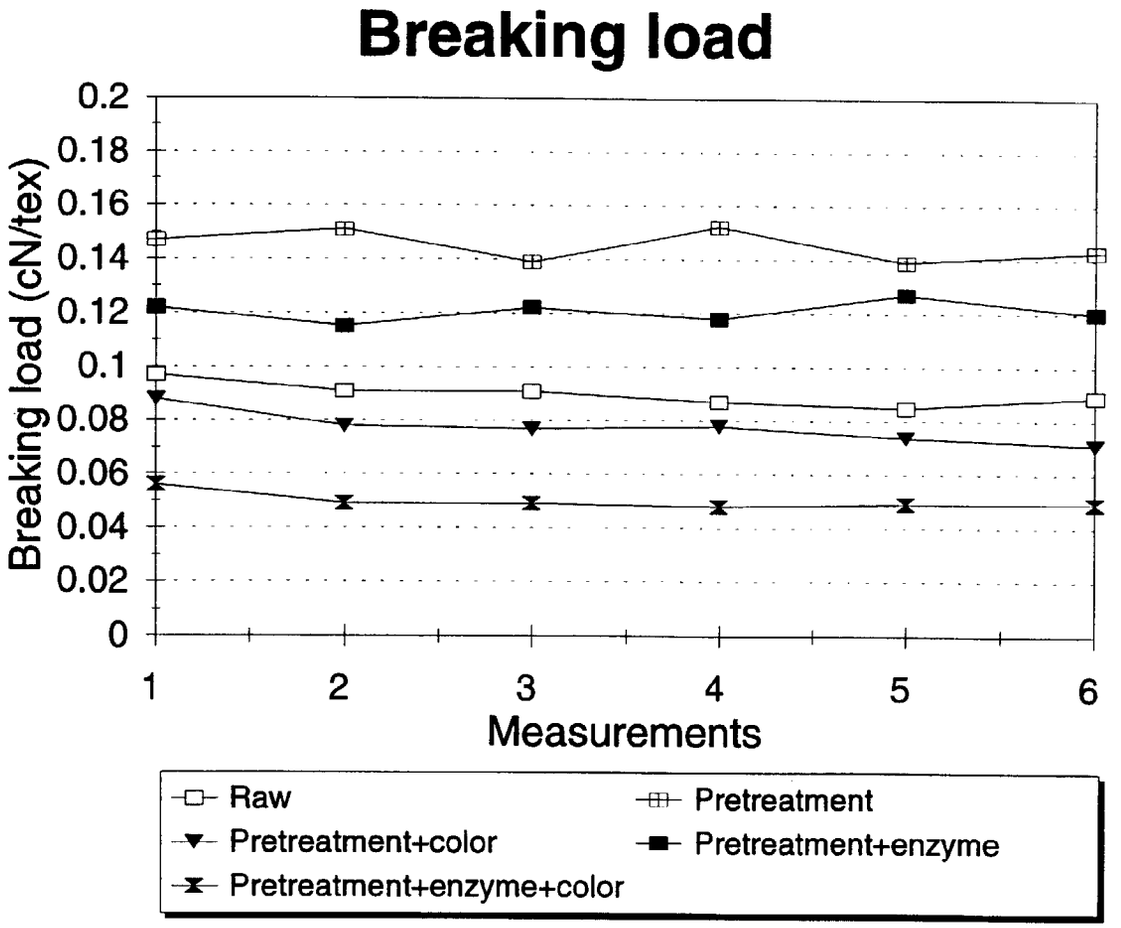
Breaking load
Breaking load (cN/tex)
0.2
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
1
2
3
4
5
6
Measurements
Raw
Pretreatment
Pretreatment+color
Pretreatment+enzyme
Pretreatment+enzyme+color

FIG. 38

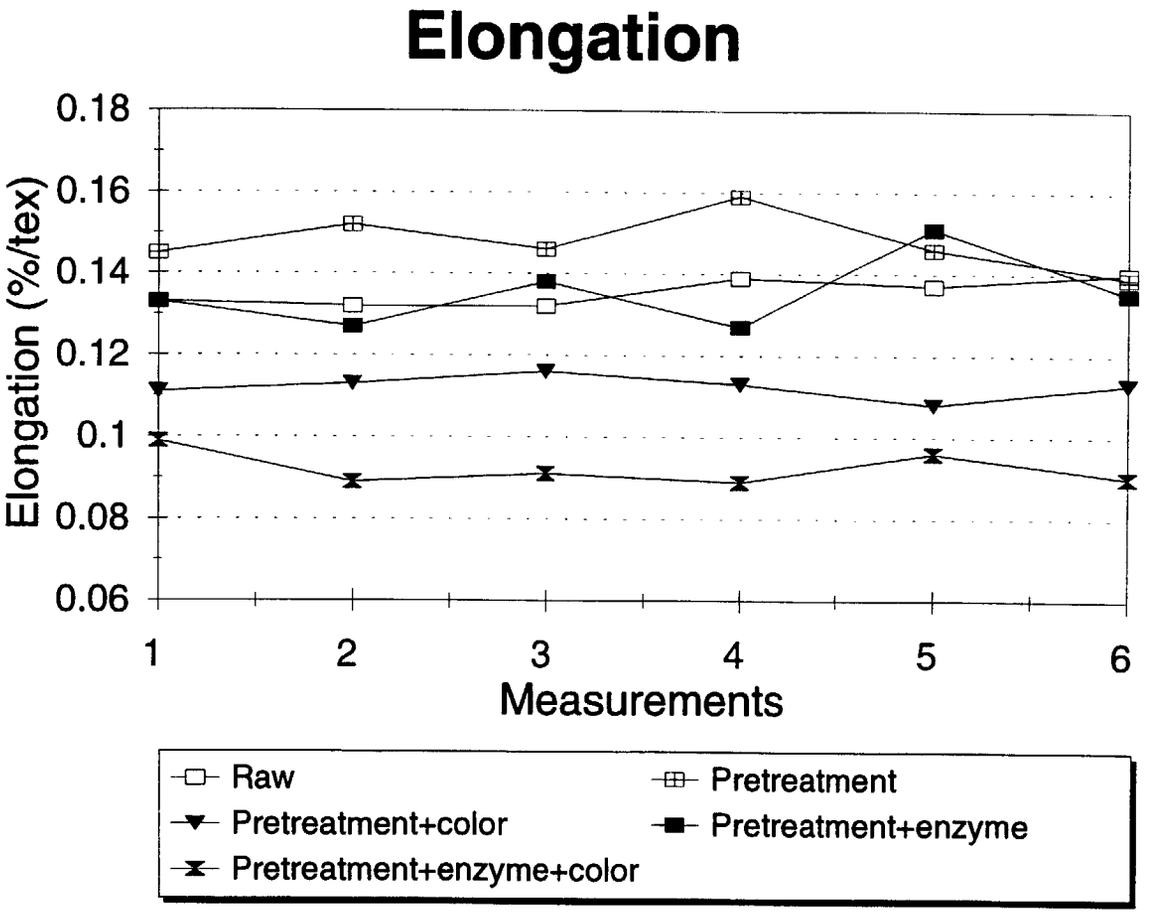
Elongation
Elongation (%/tex)
0.18
0.16
0.14
0.12
0.1
0.08
0.06
1
2
3
4
5
6
Measurements
Raw
Pretreatment
Pretreatment+color
Pretreatment+enzyme
Pretreatment+enzyme+color

FIG. 39

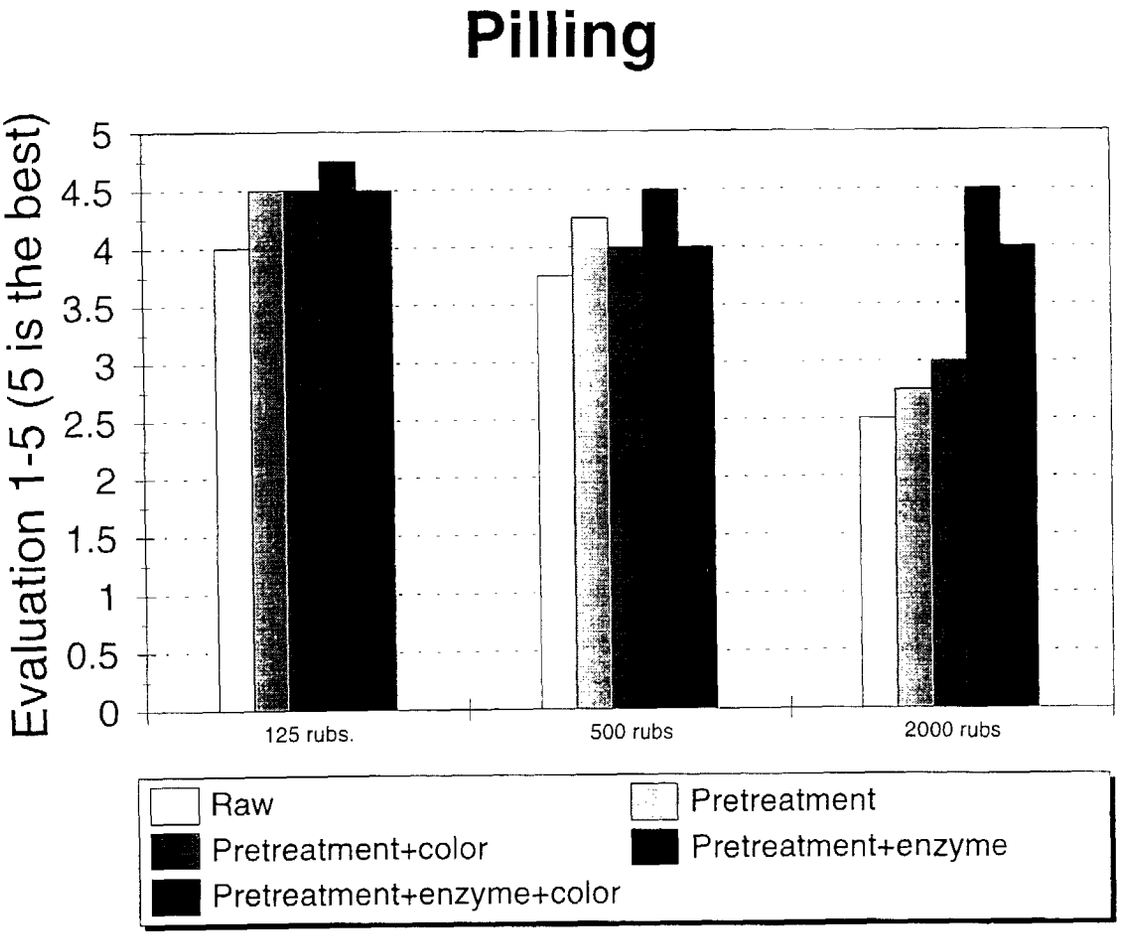
Pilling
Evaluation 1-5 (5 is the best)
5
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
125 rubs.
500 rubs
2000 rubs
Raw
Pretreatment
Pretreatment+color
Pretreatment+enzyme
Pretreatment+enzyme+color

FIG. 40

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 98 66 0109

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
A	BUSCHLE-DILLER G ET AL: "ENZYMATIC AND ACID HYDROLYSIS OF COTTON CELLULOSE AFTER SLACK AND TENSION MERCERIZATION" TEXTILE CHEMIST AND COLORIST, vol. 26, no. 4, 1 April 1994, pages 17-24, XP000442817	1,8,9, 31,35,36	D06M16/00
X	* page 18, column 1, paragraph 4 - column 2, paragraph 1; figure 2 *	14,20, 22,29,30	
A	E. BACH, E. SCHOLLMEYER: "UNTERSUCHUNGEN ZUR QUELLUNGSKINETIK DURCH CELLULASEN IN DER VORBEHANDLUNG VON BAUMWOLLE" TEXTILVEREDLUNG, vol. 29, no. 10, 1 October 1994, pages 284-288, XP000469124	1,9,31, 32	
X	* page 284, column 3, paragraph 2.2 - page 285, column 1, paragraph 1 *	14,16, 17,22, 28,30	
A	WO 93 20278 A (NOVONORDISK AS) 14 October 1993 * the whole document *	1-6, 9-19, 22-26, 28,30, 35,36	TECHNICAL FIELDS SEARCHED (Int.Cl.6) D06M D06B D06C
A	KUMAR A ET AL: "OPTIMIZING THE USE OF CELLULASE ENZYME IN FINISHING CELLULOSIC FABRICS" TEXTILE CHEMIST AND COLORIST, vol. 29, no. 4, April 1997, pages 37-42, XP000693773 * page 37, column 1, paragraph 2 - column 2, paragraph 1 *	1-6, 9-13,35, 36	

-/--

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	29 January 1999	Blas, V

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

EPO FORM 1503 03.82 (P04C01)

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 98 66 0109

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
D,A	WO 94 12578 A (NOVONORDISK AS ;SCREWS GARRETT A (US); PEDERSEN GITTE (DK)) 9 June 1994 * the whole document * ---	1	
A	WO 92 07134 A (GENENCOR INT ;COX THOMAS C (US); HAWKS PATRICK E (US); KLAHORST SU) 30 April 1992 * the whole document * -----	31	
			TECHNICAL FIELDS SEARCHED (Int.Cl.6)

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	29 January 1999	Blas, V

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

EPO FORM 1503 03.82 (P04C01)

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 98 66 0109

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

29-01-1999

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 9320278 A	14-10-1993	AT 142291 T	15-09-1996
		BR 9306160 A	13-01-1998
		CA 2132300 A	14-10-1993
		DE 69304520 D	10-10-1996
		EP 0635078 A	25-01-1995
		JP 7505451 T	15-06-1995
WO 9412578 A	09-06-1994	EP 0670866 A	13-09-1995
		JP 2749203 B	13-05-1998
		JP 8503752 T	23-04-1996
		US 5707858 A	13-01-1998
WO 9207134 A	30-04-1992	US 5232851 A	03-08-1993
		CA 2092899 A	17-04-1992
		EP 0553270 A	04-08-1993
		FI 931709 A	15-04-1993
		JP 6502458 T	17-03-1994

EPO FORM P0459

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82