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Declarations under Rule 4.17:

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(54) Title: METHOD OF DYE CLEARING TEXTILES

(57) Abstract: A process to remove excess dye from dyed polyester fabric comprising adding a solution of a weak organic acid to the fabric in a dyeing vessel, raising the temperature in the vessel to at least 80 °C, allowing the acid to react with the fabric for at least 6 minutes and removing all liquid.



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PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 150205WO	FOR FURTHER ACTION		See Form PCT/IPEA/416																
International application No. PCT/EP2017/050163	International filing date (<i>day/month/year</i>) 04.01.2017	Priority date (<i>day/month/year</i>) 04.01.2016																	
International Patent Classification (IPC) or national classification and IPC INV. D06P3/54																			
Applicant Nikwax Limited																			
1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36. 2. This REPORT consists of a total of <u>7</u> sheets, including this cover sheet. 3. This report is also accompanied by ANNEXES, comprising: a. ☒ (<i>sent to the applicant and to the International Bureau</i>) a total of <u>4</u> sheets, as follows: ☒ sheets of the description, claims and/or drawings which have been amended and/or sheets containing rectifications authorized by this Authority, unless those sheets were superseded or cancelled, and any accompanying letters (see Rules 46.5, 66.8, 70.16, 91.2, and Section 607 of the Administrative Instructions). ☐ sheets containing rectifications, where the decision was made by this Authority not to take them into account because they were not authorized by or notified to this Authority at the time when this Authority began to draw up this report, and any accompanying letters (Rules 66.4bis, 70.2(e), 70.16 and 91.2). ☐ superseded sheets and any accompanying letters, where this Authority either considers that the superseding sheets contain an amendment that goes beyond the disclosure in the international application as filed, or the superseding sheets were not accompanied by a letter indicating the basis for the amendments in the application as filed, as indicated in item 4 of Box No. I and the Supplemental Box (see Rule 70.16(b)). b. ☐ (<i>sent to the International Bureau only</i>) a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing, in the form of an Annex C/ST.25 text file, as indicated in the Supplemental Box Relating to Sequence Listing (see paragraph 3ter of Annex C of the Administrative Instructions). 4. This report contains indications relating to the following items: <table style="width: 100%; border: none;"> <tr> <td>☒ Box No. I</td> <td>Basis of the report</td> </tr> <tr> <td>☐ Box No. II</td> <td>Priority</td> </tr> <tr> <td>☐ Box No. III</td> <td>Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</td> </tr> <tr> <td>☐ Box No. IV</td> <td>Lack of unity of invention</td> </tr> <tr> <td>☒ Box No. V</td> <td>Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</td> </tr> <tr> <td>☐ Box No. VI</td> <td>Certain documents cited</td> </tr> <tr> <td>☒ Box No. VII</td> <td>Certain defects in the international application</td> </tr> <tr> <td>☐ Box No. VIII</td> <td>Certain observations on the international application</td> </tr> </table>				☒ Box No. I	Basis of the report	☐ Box No. II	Priority	☐ Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability	☐ Box No. IV	Lack of unity of invention	☒ Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement	☐ Box No. VI	Certain documents cited	☒ Box No. VII	Certain defects in the international application	☐ Box No. VIII	Certain observations on the international application
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Name and mailing address of the international preliminary examining authority: European Patent Office P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Fax: +31 70 340 - 3016		Authorized officer Blas, Valérie Telephone No. +31 70 340-3508																	



Box No. I Basis of the report

1. With regard to the **language**, this report is based on
- ☒ the international application in the language in which it was filed
 - ☐ a translation of the international application into , which is the language of a translation furnished for the purposes of:
 - ☐ international search (under Rules 12.3(a) and 23.1(b))
 - ☐ publication of the international application (under Rule 12.4(a))
 - ☐ international preliminary examination (under Rules 55.2(a) and/or 55.3(a) and (b))
2. With regard to the **elements*** of the international application, this report is based on *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report)*:

Description, Pages

1-7 as originally filed

Claims, Numbers

15 as originally filed

1-12 filed with telefax on 01-11-2017

☐ a sequence listing - see Supplemental Box Relating to Sequence Listing.

3. ☐ The amendments have resulted in the cancellation of:
- ☐ the description, pages
 - ☐ the claims, Nos.
 - ☐ the drawings, sheets/figs
 - ☐ the sequence listing (*specify*):
4. ☒ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since either they are considered to go beyond the disclosure as filed, or they were not accompanied by a letter indicating the basis for the amendments in the application as filed, as indicated in the Supplemental Box (Rules 70.2(c) and (c-bis)):
- ☐ the description, pages
 - ☒ the claims, Nos. 13
 - ☐ the drawings, sheets/figs
 - ☐ the sequence listing (*specify*):
5. ☐ This report has been established:
- ☐ taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rules 66.1(d-bis) and 70.2(e)).
 - ☐ without taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91(Rules 66.4bis and 70.2(e)).

**INTERNATIONAL PRELIMINARY REPORT
ON PATENTABILITY**

International application No.
PCT/EP2017/050163

6. ☒ With regard to top-up searches (Rules 66.1 *ter* and 70.2(f)):
- ☒ A top-up search was carried out by this Authority on 05.03.2018 (all discovered documents are listed in the Supplemental Box Relating to Top-up Search).
 - ☐ Additional relevant documents have been discovered during the top-up search.
 - ☐ No top-up search was carried out by this Authority because it would serve no useful purpose.
7. ☐ Supplementary international search report(s) from Authority(ies) has/have been received and taken into account in establishing this report (Rule 45bis.8(b) and (c)).

* If item 4 applies, some or all of those sheets may be marked "superseded".

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	<u>1-12</u>
	No: Claims	<u>15</u>
Inventive step (IS)	Yes: Claims	<u>1-12</u>
	No: Claims	<u>15</u>
Industrial applicability (IA)	Yes: Claims	<u>1-12</u>
	No: Claims	

2. Citations and explanations (Rule 70.7):

see separate sheet

Box No. VII Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

see separate sheet

Re Item I

Basis of the report

The amendments filed with the International Bureau under Article 19(1) introduce subject-matter which extends beyond the content of the application as filed, contrary to Article 19(2) PCT.

The amendments concerned are the following:

Claim 13 is defining the sequential use of a reduction clearing agent which is a weak organic acid, and an alkaline hydroxide, to remove dye from dyed polyester.

This claim is based on original claim 15 which has been amended to be conform with amended claim 1.

Such an amendment introduces subject-matter which extends beyond the content of the application as filed because the term "sequential use" does not give the order of the use of the weak organic acid and the alkaline hydroxide. It just means that they are used one after the other but not necessarily the weak acid in a first step followed by the alkaline hydroxide.

The possibility of the use of alkaline hydroxide in a first step followed by the weak organic acid is also included in this term and this order was not foreseen in the application as originally filed.

Therefore the subject-matter of claim 13 extends beyond the content of the application as originally filed and hence present claim 13 has not been taken into account in this present opinion.

Claims 1-12 are considered to meet the requirements of Article 19(2) PCT.

Consequently, the present opinion is given on claims 1-12 filed with telefax on 01.11.2017 and claim 15 of the application as originally filed.

Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Novelty/ Inventive step:

Reference is made to the following documents:

- D1 DATABASE WPI
 Week 201551
 Thomson Scientific, London, GB;
 AN 2015-40530L
 XP002768614,
 & CN 104 562 790 A (QINGDAO SANXIU NEW TECHNOLOGY
 COMPOSITE) 29 April 2015 (2015-04-29)
- D2 JP H02 91285 A (SANYO CHEMICAL IND LTD) 30 March 1990
 (1990-03-30)
- D3 GB 2 059 975 A (KAO CORP) 29 April 1981 (1981-04-29)

1. Document D1 discloses a process to wash dyed polyester fabric and thus to remove excess dye from the dyed polyester fabric comprising a reduction cleaning where a solution of citric acid which is defined in the present application as a weak acid is added to the fabric in the dyeing vessel and is also known as a reducing agent , the temperature in the dyeing vessel is raised and the acid is remaining in contact with the fabric for a period of time and dried to remove all the liquid (see abstract).

The subject-matter of claim 1 therefore differs from this known D1 in that the process further comprises a step of raising the pH to between pH 9 and pH 12 before removing all liquid and is novel (Article 33(2) PCT).

The technical effect of this difference is the provision of a process which is advantageous for subsequent treatment such as water repellent treatment (see paragraph [0019] of the present application).

Consequently, the objective problem underlying the present invention is the provision of a process to remove excess dye from dyed polyester fabric which is also advantageous for subsequent treatment of the fabric.

The solution proposed in claim 1 is considered to involve an inventive step (Article 33(3) PCT) because there are neither hints or suggestions in the prior art for the skilled person to add a step of raising the pH between 9 and 12 to carry out subsequent treatment of the fabric (In document D1, pH is adjusted between 5 and 6 and in document D2 the pH is preferably neutral and certainly not exceeding 8).

2. A similar reasoning starting from D2 would also lead to the same conclusion as it there is no indication to raise the pH after reducing cleaning.

Therefore the subject-matter of claim 1 is also considered to involve an inventive step regarding to document D2 (Article 33(3) PCT).

3. Claims 2-12 are dependent on claim 1 and as such also meet the requirements of the PCT with respect to novelty and inventive step.

4. Document D1 discloses the use of citric acid which is defined in the present application as a weak acid to remove excess dye from the dyed polyester fabric (see abstract).

Consequently, the subject-matter of independent claim 15 is not novel regarding to document D1 (Article 33(2) PCT).

Document D2 discloses the use of ascorbic acid which is a weak acid to remove dye from the dyed polyester fabric (see abstract).

Consequently, the subject-matter of independent claim 15 is also not novel regarding to document D2 (Article 33(2) PCT).

Industrial applicability:

The subject-matter of claims 1-12 and 15 is considered to meet the requirements of industrial applicability of Article 33(4) PCT.

Re Item VII

Certain defects in the international application

Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in documents D1-D3 is not mentioned in the description, nor are these documents identified therein.

International Searching Authority
European Patent Office
Postbus 5818
2280 HV Rijswijk
The Netherlands

CONFIRMATION

Via facsimile with confirmation by mail

EPO - DG 2

Our Ref: EH/150205WO

06. 11. 2017

1 November 2017

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Dear Sirs

International Patent Application No. PCT/EP2017/050163
NIKWAX LIMITED
IMPROVED METHOD OF DYE CLEARING TEXTILES

Demand

The Applicant hereby files a demand for International Preliminary Examination. We authorise debit of the fees from our deposit account 28050035.

Amendments

A set of amended claims is enclosed. The Applicant reserves the right to pursue any deleted subject matter in a divisional application during the national phase.

Claim 1 has been amended by:

- The wording of step (a) has been amended to '*a reduction clearing agent which is a weak organic acid*'. This has basis in paragraph [0018] on page 4 of the application as filed.
- An additional step has been added, step (c1), directed to '*raising the pH to between pH 9 and pH 12*'. Basis can be found in claim 11, and paragraph [0019] on page 4 of the application as filed.

Claim 6 has been deleted and Claim 7 has been limited to ascorbic acid or salts thereof.

Claim 11 has been deleted.

Claim 15 (renumbered as Claim 13) has been amended to conform with amended Claim 1 and now reads '*the sequential use of a reduction clearing agent which is a weak organic acid, and an alkaline hydroxide*'. Clear basis for this may be found in original Claim 11.

Novelty

We are pleased to note that the International Search Authority (ISA) has acknowledged novelty for original claim 11. Since the limitations of this claim have been incorporated into Claim 1, novelty of present Claim 1 should also be recognised. Claim 1 has also been limited by defining the weak

organic acid as an acid which is also reducing, such as ascorbic acid. Citric acid, for example, while a weak organic acid, does not have the same free radical activity as ascorbic acid and therefore is not an adequate reducing agent.

Inventive Step

The ISA points to D1 and alleges that the '*subject matter of [original] claims 8-14 is an obvious alternative or modification of the prior art: The skilled person would monitor the contact time by simple routine, rinsing and neutralisation are also obvious for the skilled person after treatment with acid.*' We disagree. As a first point, the present claims require that the pH is raised to between 9 and 12, significantly above a neutral pH. There is nothing in either D1 or D2 to suggest to the skilled person that the pH should be increased to such a level. In contrast, D1 requires that the pH is adjusted to between 5 and 6 using citric acid (which as explained above is not an adequate reducing agent) and the fabric is then simply dried at a temperature of 130-135°C. D2 requires that the fabric is '*soaped with a neutral bath containing the [ascorbic acid and surfactant]*' (emphasis added). Both of these references therefore teach that the fabric is treated at a neutral or slightly acidic pH and then simply dried. There is absolutely no hint or suggestion in D1 or D2 that following treatment with reduction clearing agent which is a weak organic acid, the pH should be raised to between 9 and 12. Raising the pH to between 9 and 12 is advantageous for subsequent water repellent treatment of the dyed polyester fabric, as explained in paragraph [0019] of the application as filed. Neutralisation, which is alleged to be obvious, would not achieve the same advantages.

There is nothing in D1 and D2, taken alone or in combination, that would motivate the person skilled in the art to modify the process of D2 by raising the pH with any expectation of the advantages shown by the present invention. Hence, the present invention involves an inventive step.

Concluding Remarks

In summary, we submit that present claims 1 to 14 meet all the requirements of the PCT and we look forward to receiving a favourable International Preliminary Examination Report.

Yours faithfully,



Emma Hodge
Association No. 14
Brookes IP

Enc. Amended claims (marked up and clean versions)

Claims

1. A process to remove excess dye from dyed polyester fabric comprising (a) adding a reduction clearing agent which is a weak organic acid or salt thereof to the fabric in a dyeing vessel, (b) raising the temperature in the vessel, (c) allowing the acid or salt thereof to remain in contact with the fabric for a period of time, (c1) raising the pH to between pH 9 and pH 12, and (d) removing all liquid.
2. The process according to Claim 1 which comprises the additional step of removing dyeing liquor from the dyeing vessel prior to (a).
3. The process according to Claim 1 or Claim 2, where in step (c) the weak organic acid or salt thereof remains in contact with the fabric for at least 6 minutes, and/or for up to 60 minutes.
4. The process according to any one of Claims 1 to 3, where in step (b) the temperature in the vessel is raised to a value in the range of from 60 to 100°C.
5. The process according to any one of Claims 1 to 4, in which the weak organic acid is selected from those having at least 4 carbon atoms and a pK_a or pK_{a1} value of at least 1.
6. The process according to Claim 5 wherein the weak organic acid is ascorbic acid or salt thereof.
7. The process according to Claim 6 wherein the weak organic acid is ascorbic acid.
8. The process according to any one of Claims 1 to 6 wherein the salt of the weak organic acid is selected from an alkali metal salt, and is preferably selected from a sodium or potassium salt.
9. The process according to any one of Claims 1 to 8 comprising the additional step of rinsing the fabric with water at ambient temperature after the removal of all liquid.
10. The process according to any one of Claims 1 to 9 wherein an alkaline hydroxide is used to raise the pH, which is preferably selected from sodium hydroxide, potassium hydroxide or ammonium hydroxide.
11. The process according to any one of Claims 1 to 10 wherein the weak organic acid or salt thereof is added at a rate of from 80g to 120g per litre.

12. The process according to Claim 2 wherein the weak organic acid or salt thereof is added at a rate of from 2g to 50g per litre, preferably 2g to 10g per litre, most preferably 5g per litre.

13. The sequential use of a reduction clearing agent which is a weak organic acid, and an alkaline hydroxide, to remove dye from dyed polyester.