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(54) Title: NONWOVENS COMPRISING HYDROPHOBIC CELLULOSIC MAN-MADE FIBRES

(57) Abstract: The invention concerns anonwoven comprising a blend of two fibre types, whereas the first fibre type is a cellulosic fibre and the second fibre type is a biodegradable hydrophobic cellulosic and is characterised in that the absorbency for a liquid is at least 5 % higher compared to a nonwoven with the highest absorbency for this liquid comprising only one of the said fibre types.



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Nonwovens comprising hydrophobic cellulosic man-made fibres

The invention concerns a nonwoven from a blend of hydrophobic cellulosic fibres and cellulosic fibres.

5

Cellulosic man-made fibres like viscose and lyocell fibres are known best for their water absorbency behaviour and used where hydrophilicity is required. In contrast synthetic fibres such as polyester, polyethylene and polypropylene are inherently hydrophobic which means that they do not absorb water into their interior structure.

10

State of the art viscose and lyocell fibres are hydrophilic and have a surface tension of around 85 – 90 mN/m, depending on the type of applied soft finish. Polyester fibres used in nonwoven applications are in the area of 40 – 45 mN/m.

15 Cellulosic fibres of the viscose type and modal type are produced according to the viscose process. Such fibres have been given the generic names viscose and modal by BISFA (The International Bureau for the Standardisation of man-made Fibres).

In recent years the “amine-oxide-process” or “Lyocell process” has been established as an  
20 alternative to the viscose process, wherein cellulose, without forming a derivative, is dissolved in an organic solvent of an amine-oxide, in particular N-Methylmorpholine-N-oxide (NMMO). Cellulosic fibres produced from such solutions are called “solvent-spun” fibres and have been given the generic name lyocell by BISFA (The International Bureau for the Standardisation of man-made Fibres), and are available under the trade mark TENCEL ®  
25 from Lenzing AG.

Other man-made cellulose fibres can be made using chemical processes (e.g. the cuproammonium process) or using other direct solvents such as ionic liquids.

30 Wet wipes for hygiene applications like toilet wipes, facial wipes, cosmetic wipes, baby wipes and sanitary wipes are made from nonwovens soaked with lotions. Lotions are aqueous or aqueous/alcoholic low- to medium viscosity preparations intended for application on skin or aqueous or aqueous/alcoholic containing suspended or emulsified substances. Wipes for household or industrial applications also use a wide range of liquids to improve their efficacy.

35

For nonwoven applications, synthetic fibres such as polyester are widely used as they enhance bulk, opacity and softness in nonwoven and textile applications.

State of the art is a blend between man-made cellulosic fibres and polyester fibres to enhance product performance in wipes, hygiene applications or technical applications. Fabrics made of 100 % cellulosic material have little hydrophobic/non polar effect.

- 5 For ecological reasons there is a demand for nonwovens which are made from renewable raw materials and which are also biodegradable and have good absorption properties for liquids like lotions.

- 10 Hydrophilic cellulosic fibres like standard lyocell fibres are in the range of 8.5 – 9 g water/ g nonwoven while hydrophobic fibres like polyester or hydrophobic lyocell fibres show little to no absorbency (0 – 1.0 g/g).

- Any blend of hydrophilic cellulosic fibres with hydrophobic fibres would be expected to be in an area between these values following a linear function.

15

It has been found that a nonwoven comprising blends of hydrophobic cellulosic fibres with standard cellulosic fibres have higher liquid absorbency than a nonwoven comprising only standard cellulosic fibres or hydrophobic fibres.

- 20 The inventive nonwoven comprises a blend of two fibre types, whereas the first fibre type is a cellulosic fibre and the second fibre type is a biodegradable hydrophobic cellulosic fibre and is characterised in that the absorbency for a liquid is at least 5 % higher compared to a nonwoven with the highest absorbency for this liquid comprising only one of the said fibre types. Depending on the blend the absorbency may increase by up to 15 % or more.

25

Liquids are, for example, lotions (this usually contains water; glycols like propylene glycol; organic acids like benzoic acid, dehydroacetic acid, citric acid; salts of organic acids like sodium citrate, emulsifiers like polysorbate 20, and other ingredients), other liquids are oils.

- 30 It has also been found that the increase in the absorbency also depends on the surface tension of the liquid.

- For liquids with a surface tension of above 50 mN/m, like water, the blend comprises 5 to 40 % per weight fibres of the second fibre type (hydrophobic fibres) and 95 to 60 % per weight of the first fibre type (hydrophilic fibres), preferably 5 to 30 % per weight of the second fibre type and 95 to 70 % per weight of the first fibre type, totalling 100 %.
- 35

For liquids with a surface tension of below 50 mN/m, like alcohols, the blend comprises 40 to 95 % per weight fibres of the second fibre type (hydrophobic fibres) and 60 to 5 % per weight of the first fibre type (hydrophilic fibres), preferably 30 to 95 % per weight of the second fibre type and 70 to 5 % weight of the first fibre type, totalling 100 %.

5

The first and second fibre type is a man-made cellulosic fibre, typically selected from the group consisting of viscose fibres, lyocell fibres or modal fibres. The first and second fibre type may also be a natural cellulosic fibre, like cotton.

- 10 The production of hydrophobic cellulosic fibres is described in the yet unpublished application AT-A 1658/2011 of Lenzing AG.

The hydrophobic agents, such as Alkyl Ketene Dimere formulations (AKD formulations) are commercially available (for example Hydrores® compounds sold by Kemira). The most  
15 common are formulations with around 5 – 25 % of active compound. In the case of this example the formulation is an acidic emulsion with an active compound of around 20 – 22 %.

The hydrophobic fibres contains the hydrophobising agent in an amount of 0.01 to 1 % by weight, preferably of 0.05 to 0.2% per weight based on cellulose. The preferred hydrophobic  
20 agent is an Alkyl Ketene Dimere.

Production of hydrophobic cellulose fibres used in the examples:

1000 g never dried lyocell fibres (1.7 dtex /38 mm) were taken wet from the lyocell production  
25 line before the addition of soft finish and were pressed to a moisture content of around 50 %. These fibres were treated in 15 l of an aqueous solution containing 41 g Alkyl Ketene Dimere (13 g/l with 21 % active substance) solution (e.g. Hydrores ® obtained commercially from Kemira OYI) at ca. 60 °C for 5 minutes and dried to a moisture content of ca. 10 %.

- 30 These fibres have a water retention value of around 55 – 75 % most typically in the area of 60 – 65 %, which is around 20% lower than Standard Tencel 1.7/38 Fibers (85-90 %).

These fibres were used to make around 60 g/m<sup>2</sup> spun laced samples blended with commercially available Tencel 1.7/38 mm bright Nonwoven fibres and also commercially  
35 available Lenzing NW 1.7 dtex/40mm bright Viscose fibres.

Nonwoven production

The samples were made by the following common steps

Fibre Opening and Blending,

Carding,

Hydro-Entanglement,

5 Dewatering,

Through Air Drying and

Winding

10 The line speed was in a range of 55 -100 m/min and on the hydroentanglement unit cylinders were used with a total of 4 water injectors. The adjusted water pressures were in the range of 30-80 bar. The jet strips in the injectors were common jet strips which can be used for any standard hydro-entangled product in this weight range.

15 The nonwoven may also be formed by other nonwoven processes including drylaying, wetlaying or airlaying.

The nonwoven may be bonded using any conventional nonwoven bonding process including hydroentangling, needlepunching or chemical bonding.

20 The nonwoven may have a basis weight from 10 to 2000g/m<sup>2</sup>. For wipes a nonwoven with a basis weight of 25 to 200 g/m<sup>2</sup> is preferred. For technical applications such as oil uptake mats, basis weights of up to 2000 g/m<sup>2</sup> are appropriate

25 The Gravimetric Absorbency Time (GAT) is measured according to WSP 242.3 [or ERT 442.2 similar to IST 242(02)]. This shows how much water is absorbed in a Nonwoven in given time.

30 Table 1 shows the water absorbency (GATS values) of nonwovens produced by the described process with different fibre blends of hydrophobic lyocell fibres and standard lyocell fibres. The lyocell fibres are lyocell fibres produced by Lenzing AG under the trade name Tencel.

35 Lower blending amounts (5 – 10 % hydrophobic Tencel) lead to an absorbency increase of 5 %, higher amounts (20 – 30 %) lead to 15 % higher absorbency, while amounts of hydrophobic fibers higher than 40 % lead to water repellence and low absorbency.

Table 2 shows similar results for Hydrophobic Tencel / Viscose blends.

Table 1

| Sample | Blend                                            | GATS                                     |
|--------|--------------------------------------------------|------------------------------------------|
|        | <b>Hydrophobic Tencel / Tencel blends</b>        | <b>[g H<sub>2</sub>O/g spunlaced NW]</b> |
| 1      | 100% Tencel 1,7/38                               | 8,6                                      |
| 2      | 5% Hydrophobic Tencel 1,7/38 - 95% Tencel 1,7/38 | 8,9                                      |
| 3      | 30%Hydrophobic Tencel 1,7/38 - 70% Tencel 1,7/38 | 9,8                                      |
| 4      | 50%Hydrophobic Tencel 1,7/38 - 50% Tencel 1,7/38 | 0,8                                      |
| 5      | 70%Hydrophobic Tencel 1,7/38 - 30% Tencel 1,7/38 | 1,2                                      |
| 6      | 100% Hydrophobic Tencel 1,7/38                   | 1,0                                      |

Table 2:

| Sample | Blend                                              | GATS                                     |
|--------|----------------------------------------------------|------------------------------------------|
|        | <b>Hydrophobic lyocell / Viscose blends</b>        | <b>[g H<sub>2</sub>O/g spunlaced NW]</b> |
| 7      | 100% Viscose 1,7/38                                | 7,8                                      |
| 8      | 30%Hydrophobic Tencel 1,7/38 - 70% Viscose 1,7/40  | 8,3                                      |
| 9      | 50% Hydrophobic Tencel 1,7/38 - 50% Viscose 1,7/40 | 2,9                                      |
| 10     | 70% Hydrophobic Tencel 1,7/38 - 30% Viscose 1,7/40 | 0,7                                      |

Another object of the invention is to provide a fully biodegradable nonwoven which has a surface tension optimized to suit different liquids. This can be used to enhance the absorbency or cleanability of a wipe for a particular duty by optimisation for liquid pick up or release.

A method to determine the surface tension of a surface in the laboratory is shown in BS ISO 8296:2003: A set of different liquids (like water, methanol, ethylene glycol mono ethyl ether or formamide) as shown in table 3 are used to determine the surface tension. These tests are used in, for example, plastic foil production to check that the surface is printable. Test inks from ARCOTest GmbH/Germany were also used.

**Table 3:**

| Liquid                                                          | Surface Tension |
|-----------------------------------------------------------------|-----------------|
|                                                                 | [mN/m]          |
| 80ml MeOH + 20ml H <sub>2</sub> O                               | 27,3            |
| 36,5 ml Ethyleneglycolmonoethylether (EGMEE) + 63,5ml Formamide | 40              |
| 100ml Ethyleneglykol                                            | 48              |
| 9,3 ml EGMEE + 90,7ml Formamide                                 | 50              |
| 6,3 ml EGMEE + 93,7 ml Formamide                                | 52              |
| 100ml Formamide                                                 | 58              |
| 50ml Formamide + 50ml Water                                     | 63              |
| 100ml H <sub>2</sub> O (purified for HPLC use)                  | 73              |
| Test Ink from ARCO                                              | 76              |
| Test Ink from ARCO                                              | 84              |
| Test Ink from ARCO                                              | 90              |

- 5 This method was used to determine the surface tension of a range of fabric blends. A trend is seen in that a higher amount of hydrophobic Tencel fibres in the blend lowers the surface tension as demonstrated by the observation that a liquid with a lower surface tension is able to wet the surface of the fabric. Table 4 and Fig. 1 shows the calculated surface tension of different fabric blends.

10

**Table 4::**

| Sample No. | Blend                                     | Surface Tension<br>[mN/m] |
|------------|-------------------------------------------|---------------------------|
| 6          | 100% Hydrophobic Tencel                   | 27,3                      |
| 5          | 70% Hydrophobic Tencel, 30% Tencel 1,7/38 | 40                        |
| 4          | 50% Hydrophobic Tencel, 50% Tencel 1,7/38 | 48                        |
| 3          | 30% Hydrophobic Tencel, 70% Tencel 1,7/38 | 58                        |
| 11         | 20% Hydrophobic Tencel, 80% Tencel 1,7/38 | 63                        |
| 12         | 10% Hydrophobic Tencel, 90% Tencel 1,7/38 | 73                        |
| 2          | 5% Hydrophobic Tencel, 95% Tencel 1,7/38  | 84                        |
| 1          | 100% Tencel                               | 90                        |

The inventive nonwoven product comprises a nonwoven of blend of hydrophilic and hydrophobic man-made cellulosic fibres and a liquid and is characterised in that the surface tension of the nonwoven is in the range of the surface tension of the liquid with a deviation of less than +/- 5 %.

In a blend of hydrophilic and hydrophobic man-made cellulosic fibres the surface tension in a fabric can be tuned to fit to the liquid used to wet the fabric. That means that the blend used in wipes influences the surface tension so that it is possible to tailor a wipe which fits to a range where a liquid is able to wet the surface quickly or where the wipe may be used to release the liquid efficiently. A category of liquids are lotions (this usually contains water; glycols like propylene glycol; organic acids like benzoic acid, dehydroacetic acid, citric acid; salts of organic acids like sodium citrate, emulsifiers like polysorbate 20, and other ingredients).

It has also been found that additionally, a better distribution of a lotion in a wipes pack can be achieved by selecting the optimal blend of hydrophilic and hydrophobic man made cellulosic fiber.

Wet wipes are normally packed in packages with stacks of typically 10 to over 100 single wipes. During storage, the lotion tends to seep from top to bottom of the pack due to gravity, so the topmost wipes of the stack become drier, while the bottom wipes are saturated and liquid may pool on the bottom of the pack. This leads to an inconvenient, non-uniform distribution of the lotion which affects the effectiveness of a single wipe from the pack.

#### **Test Procedure Liquid Retention & Release:**

80 pieces of spunlaced nonwoven (10x10cm) were cut and conditioned and the weight was measured for each piece. The pieces were stacked and soaked with a typical wet wipe lotion (containing water, propylene glycol, benzoic acid, dehydroacetic acid, citric acid, sodium citrate, polysorbate 20) in the ratio of 1: 2.5 (1g wipe, 2.5g lotion) after which the stack was put into a plastic film and vacuum sealed. This was kept in a conditioned room in darkness for 4 weeks. After that every single layer was weighed again and the results are shown in the following diagrams:

Fig. 2 shows the **Liquid Retention & Release behaviour** of a blend of 30 % hydrophobic Tencel 1.7 dtex /38 mm dull with 70 % Viscose 1.7 dtex/40 mm dull

Fig. 3 shows the **Liquid Retention & Release behaviour** of a blend of 70% Trevira PES 1.7/ 38 dull with 30% Tencel standard 1.7/38 dull.

- 5 It can be seen that the blend of hydrophobic Tencel with viscose (Fig. 2) shows much lower variation in the lotion content of the wipes between the bottom and top of the stack , by about 40% compared to the blend of polyester with Tencel (Fig. 3).

- 10 The inventive nonwoven product comprising a nonwoven of a blend of hydrophobic cellulosic fibres with cellulosic fibres which is characterised in that deviation of the uniformity of the distribution of the aqueous solution is better than  $\pm 15\%$  when tested according to the described **Liquid Retention & Release** test.

- 15 Additionally all fabrics made from hydrophobic lyocell and blends with cellulosic fibres are totally biodegradable.

- 20 The inventive nonwoven is used advantageously in hygiene applications like toilet wipes, facial wipes, cosmetic wipes, baby wipes, and sanitary wipes, household wipes, industrial wipes or in food wrappings.

Another use is in technical applications for example for oil uptake materials, like mats.

Claims:

- 1) Nonwoven comprising a blend of two fibre types, whereas the first fibre type is a  
cellulosic fibre and the second fibre type is a biodegradable hydrophobic cellulosic fibre,  
5 characterised in that the absorbency for a liquid is at least 5 % higher compared to a  
nonwoven with the highest absorbency for this liquid comprising only one of the said fibre  
types.
- 2) Nonwoven according to claim 1, characterised in that the blend comprises 5 to 40 % by  
10 weight of fibres of the second fibre type and 95 to 60 % by weight of fibres of the first  
fibre type, preferably 5 to 30 % by weight of fibres of the second fibre type and 95 to 70  
% by weight of fibres of the first fibre type, totalling 100 % and the liquid has a surface  
tension of greater or equal to 50 mN/m.
- 3) Nonwoven according to claim 1, characterised in that the blend comprises 40 to 95 % by  
15 weight of fibres of the second fibre type and 60 to 5 % by weight of fibres of the first fibre  
type, preferably 30 to 95 % by weight of fibres of the second fibre type and 70 to 5 % by  
weight of fibres of the first fibre type, totalling 100 % and the liquid has a surface tension  
of less than 50 mN/m.
- 20 4) Nonwoven according to claim 1 to 3 characterised in that the first and second fibre type is  
a man-made cellulosic fibre, selected from the group consisting of viscose fibres, lyocell  
fibres or modal fibres.
- 25 5) Nonwoven according to claim 1 to 4 characterised in that the first and second fibre type is  
a natural cellulosic fibre, like cotton.
- 6) Nonwoven according to claim 1 to 5 characterised in that the hydrophobic fibre contains a  
hydrophobising agent in an amount of 0.01 to 1 % per weight, preferably of 0.05 to 0.2 %  
30 per weight based on cellulose.
- 7) Nonwoven according to the claim 6 characterised in that the hydrophobising agent is an  
Alkyl-Ketene-Dimere.
- 35 8) Nonwoven according to one of the previous claims characterised in that the nonwoven is  
formed by a typical nonwoven process including drylaying, wetlaying or airlaying .

- 9) Nonwoven according to one of the previous claims characterised in that the nonwoven is bonded by a typical nonwoven process including hydroentangling, needlepunching or by chemical bonding.
- 5 10) Nonwoven according to one of the previous claims, characterised in that the nonwoven has a basis weight of 10 to 2000 g/cm<sup>2</sup>.
- 10 11) Nonwoven product comprising a nonwoven according to claim 1 to claim 10 and a liquid characterised in that the surface tension of the nonwoven is in the range of the surface tension of the liquid with a deviation of less than +/- 5 %.
- 12) Nonwoven product according to claim 11 characterised in that the liquid is a lotion.
- 15 13) Nonwoven product comprising a nonwoven according to claim 11 to claim 12, characterised in that *deviation* of the uniformity of the distribution of the aqueous solution is better than +/- 15 % when tested according to the described "Liquid Retention & Release test"
- 20 14) Use of nonwoven according to claim 1 to claim 13 in hygiene applications like toilet wipes, facial wipes, cosmetic wipes, baby wipes, sanitary wipes, household wipes, industrial wipes and in food wrappings.
- 15) Use of nonwoven according to claim 1 to claim 13 in technical applications like oil uptake materials.

Fig. 1

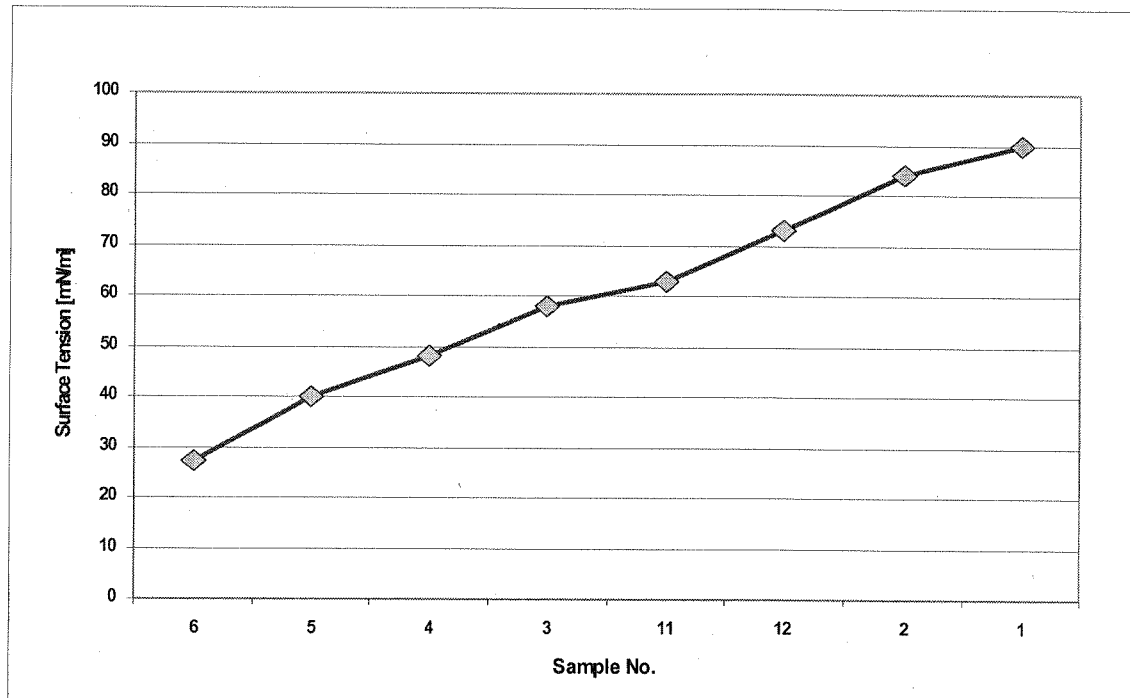


Fig. 2

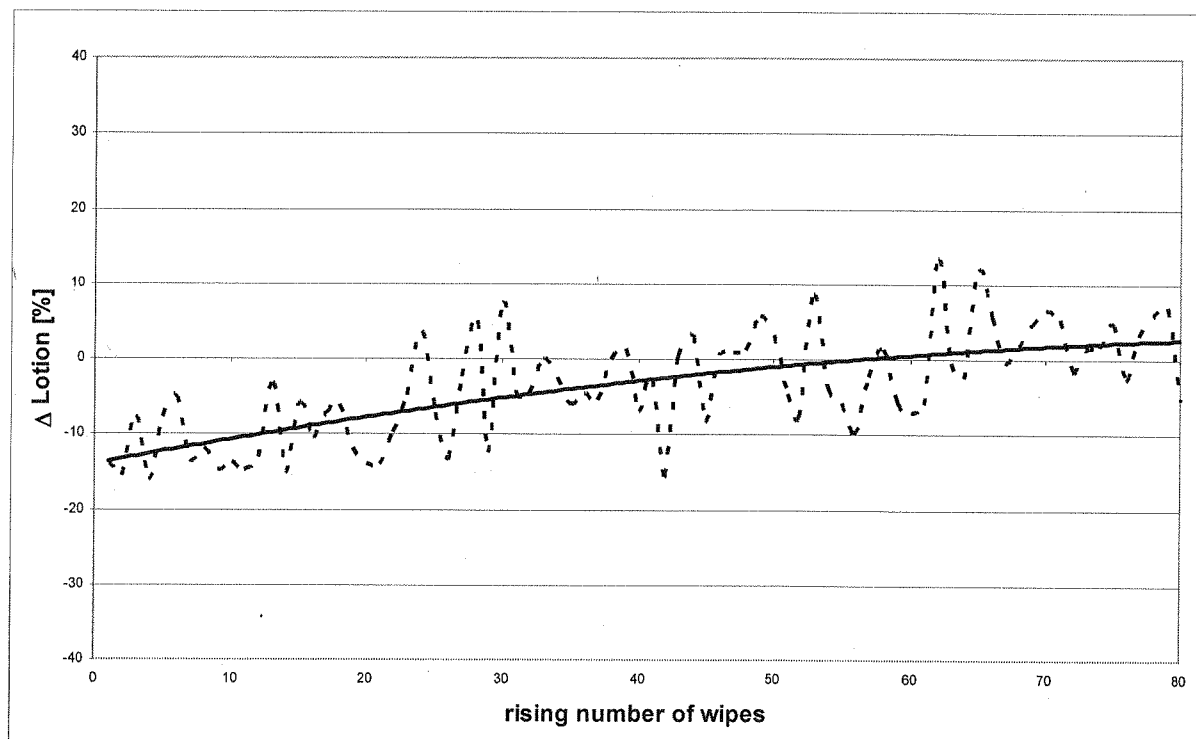
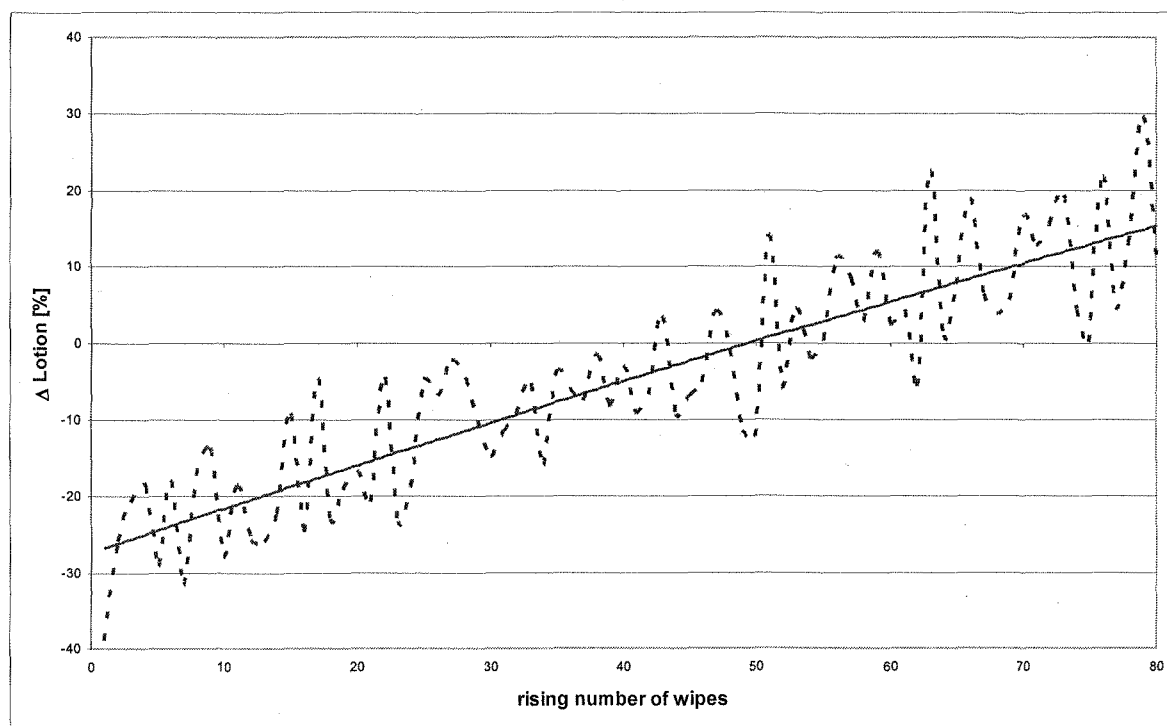


Fig. 3



## INTERNATIONAL SEARCH REPORT

International application No  
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A. CLASSIFICATION OF SUBJECT MATTER  
INV. D04H1/28 D04H1/4258 D04H1/492 D04H1/74 D06M13/13  
ADD.

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)  
D04H D06M

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

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, PAJ, WPI Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                                             | Relevant to claim No.    |
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Further documents are listed in the continuation of Box C.



See patent family annex.

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Date of the actual completion of the international search

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## INTERNATIONAL SEARCH REPORT

International application No  
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| C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT |                                                                                                                                                                 |                       |
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Information on patent family members

International application No

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