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(54) **NEW PROCESS FOR PRE-TREATING CELLULOSIC FIBERS AND CELLULOSIC FIBER BLENDS**

NEUE VERFAHREN ZUR VORBEHANDLUNG VON ZELLULOSEFASER UND DEREN  
MISCHUNGEN

NOUVEAU PROCÉDE POUR PRETRAITER LES FIBRES DE CELLULOSE ET LES MELANGES  
DE FIBRES DE CELLULOSE

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**EP-A- 0 369 711 EP-A- 0 449 797**  
**EP-A- 0 585 038 EP-A- 1 038 947**  
**US-A- 5 562 740 US-A- 5 698 507**

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- **DATABASE WPI Section Ch, Week 198023 Derwent Publications Ltd., London, GB; Class F06, AN 1980-40990C XP002177014 & SU 691 511 A (GUSAKOV M B), 15 October 1979 (1979-10-15)**

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**Description**

**FIELD OF THE INVENTION**

5 **[0001]** The present invention is directed to a new process for pre-treating cellulosic fibers and cellulosic fiber blends with synthetic fibers, eliminating the need for rinses or significantly reducing the amount of rinsing necessary.

**[0002]** Various processes for pre-treating cellulosic fibres are known. US 5,562,740 discloses a process for treating cellulosic fibres comprising reacting said fibres with a polycarboxylic acid crosslinking agent such as citric acid, raising the pH to 5.5 to 6.5 with sodium hydroxide and contacting the fibres with an oxidising bleaching agent such as hydrogen peroxide. In an article titled "Treating Fabric Through Alkaline Oxidation For A Silk-Like Effect" of Ming et. al. in "American Dyestuff Reporter", July 1996, pages 20-26, a process is disclosed wherein fabric is treated comprising padding the fabric with a copper sulfate solution and adding sodium hydroxide and hydrogen peroxide.

**BACKGROUND OF THE INVENTION**

15 **[0003]** A typical example of the preparation for dyeing of 100% cotton materials includes:

**Exhaust Procedure:**

20 a) bath composition

**[0004]**

<p>25 0.5-2.0 g/l Wetting Agent/Detergent: 0.3-0.6 g/l Peroxide Stabilizer:</p> <p>30 1.5-3.0 g/l Caustic Soda (100%) 1.5-3.0 g/l Hydrogen Peroxide (100%)</p>	<p>nonionic and/or anionic surfactants organo-phosphate based (e.g., diethylenetriamine pentamethylene phosphonic acid (DTPMP)) and/or amino-organic acid based (e.g., diethylenetriamine pentaacetic acid (DTPA)) and/or polyacrylic acid based and/or organic acid based (e.g., sodium salt of gluconic Acid) and/or silicate based and/or earth alkaline salts (e.g., MgCl<sub>2</sub>)</p>
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b) typical application:

35 **[0005]**

Cellulosic material is loaded into an exhaust dyeing machine or apparatus (e.g., Jet Dyeing machine, winch, package machine, beam etc.). The machine is filled with water and possibly with a wetting agent to produce a bath before a material load is introduced to the machine. The water amount is typically calculated based on the weight of the material load and expressed in a liquor ratio. A typical liquor ratio is 1:10, or for 1kg fabric, 10l liquid are used.

Subsequent to loading the machine, the remaining chemicals are added and the resulting bath is heated to a suitable temperature, typically 98°C-110°C. Depending on the construction of the machine/apparatus, material and/or liquor is moved to ensure homogeneous and efficient pretreatment.

The bath is then cooled and dropped, or drained, after a treatment time of 15-30 min. Multiple rinses and/or overflow washes of the cellulosic material are necessary to remove impurities and especially residual alkalinity in the material that otherwise would harm or interfere with the effectiveness of subsequent processes.

Alkalinity, typically provided by caustic soda, is considered necessary to activate the oxidizing component, hydrogen peroxide, and to saponify waxes and other fatty based cotton byproducts allowing easier removal of these impurities. This process of pre-treating cellulosic material is commonly referred to as a bleaching cycle that occurs prior to the dyeing of the material.

**SUMMARY OF THE INVENTION**

55 **[0006]** The present invention is a process for pre-treating a cellulosic, or cellosic blends with synthetic fiber, substrate.

In a most basic form, the invented pre-treating process of cellulosic, or cellulosic blends, substrate is a bleaching cycle comprising the steps of: providing a vessel; providing the cellulosic, or their blends with synthetic fiber, substrate; providing a water bath; adding an active amount of an activating compound selected from the group of: salts of organic acids, organic amine derivatives, transitional metals, transitional metal salts and transitional metal complexes, pigments and combinations thereof; adding an active amount of caustic soda; adding an active amount of hydrogen peroxide during the bleaching cycle; achieving a pH from about 6.0 to about 9.0 at the end of the bleaching cycle pretreatment process; heating the water bath to a temperature in excess of 50 degrees centigrade for a pre-determined period of time; and, dropping the bath.

## DESCRIPTION OF THE INVENTION

**[0007]** The present invention is an innovative and novel process and composition for pre-treating a cellulosic, or cellulosic blends with synthetic fiber, substrate that eliminates or greatly reduces the need for rinses. The invented process is ideally used as a pre-treatment process of cellulosic, or cellulosic blended fibers or materials, prior to dyeing the same. Using the invented process, significant amounts of water, waste-water, energy, and process time are saved. Furthermore, the invented process affords additional machine capacity.

**[0008]** In the invented process and composition, significant amounts of alkali that are normally used in conventional processes are replaced by alternative chemicals and chemical systems leading to a well prepared cellulosic, or cellulosic blends with synthetic fiber, substrate (e.g., cotton) that does not require rinsing after a bleach application. This can be achieved due to a resulting neutral or nearly neutral final pH and sufficient cleanliness of the prepared goods. The cleanliness is indicated by a degree of water absorption and whiteness as well as by visual aspect (removal of seeds) of the bleach goods.

**[0009]** Alternatives to a commonly used alkali caustic soda include but are not limited to: alkali salts of organic acids, preferably Trisodiumcitrate; transition metal salts and complexes, preferably Copper salts and complexes; organic activators, preferably Urea, Dicyandiamid or Tetraacetylenediamine, Acetyl Caprolactam; pigments, preferably pigmented Sulfur Black 1 with a particle size less than 150µm or Titanium Dioxide with a particle size less than 150µm; and, combinations thereof.

**[0010]** If used within pre-determined parameters, described in greater detail hereinafter, none to a very acceptable degree of damage of cellulosic, or cellulosic blends with synthetic fiber, substrate is expected. Use of earth-alkaline salts, preferably Mg salts (e.g., MgSO<sub>4</sub>) have a stabilizing function. For example, the earth-alkaline salts prevent premature and uncontrolled destruction of hydrogen peroxide that could lead to insufficient bleach results and fiber damage.

**[0011]** The present invention is a process for pre-treating a cellulosic, or cellulosic blends with synthetic fiber, substrate having the steps of: providing a vessel; providing the cellulosic, or cellulosic blends with synthetic fiber, substrate; providing a water bath; adding an active amount of an activating compound selected from the group of: salts of organic acids, organic amine derivatives, transitional metals, pigments with a particle size less than 150µm, and combinations thereof; adding an active amount of caustic soda; adding an active amount of hydrogen peroxide during a bleaching cycle; achieving a pH from about 6.0 to about 9.0 at the end of the bleaching cycle; heating the water bath to a temperature in excess of 50 degrees centigrade for a period of time; and dropping the bath.

**[0012]** When the activating compound is a salt of an organic acid, some examples that have been found to work well include, but are not limited to: sodium salts of citric acid; sodium stearate; sodium salts of gluconic acid; sodium oleate; potassium salt of citric acid; potassium stearate; potassium salt of gluconic acid; potassium oleate; ammonium salts of citric acid; ammonium stearate; ammonium salts of gluconic acid; ammonium oleate; and, combinations thereof. Preferably about 0.2 to about 5.0% based on the weight of the substrate, hereinafter referred to as "owg". of the salt of an organic acid is used.

**[0013]** When the activating compound is an organic amine derivative, some examples that have been found to work well include, but are not limited to: urea; dicyandiamid; tetra-acetyl-ethylene-di-amine; acetyl-caprolactam; and, combinations thereof. Preferably about 0.2 to about 5.0% owg of the organic amine derivative is used.

**[0014]** When the activating compound is a transitional metal salt or complex, some examples that have been found to work well include, but are not limited to: copper gluconate; copper sulfate; copper acetate; copper carbonate; copper citrate; copper nitrate; copper EDTA; copper complexes; and, combinations thereof. When Copper compounds are used as the transitional metal salt or complex, preferably about 0.1 to about 10ppm based on the weight of the bath, hereinafter referred to as "owb", based on the element Copper is used.

**[0015]** When the activating compound is a pigment, some examples that have been found to work well include, but are not limited to: pigmented Sulfur Black 1 with a particle size less than 150µm; fully pre-oxidized sulfur dyes, such as Diresul Black 4G-EV or Titanium Dioxide and, combinations thereof. Fully pre-oxidized sulfur dyes or Titanium Dioxide are preferably selected because the bleach-white base as well as the visual white appearance of the substrate is synergistically improved by the use thereof. Preferably about 1 to about 200ppm owb of pigment is used.

[0016] When caustic soda is added, from about 0.1 to about 1.0% owg is preferably used. When hydrogen peroxide is added, the amount depends on the desired whitening effects but preferably ranges between about 0.5 to about 5.0% owg.

[0017] In the invented process for pre-treating a cellulosic, or cellulosic blends with synthetic fiber, substrate, the water bath is preferably heated to a temperature ranging from about 80 degrees centigrade to about 140 degrees centigrade. The substrate is held within this temperature range for a period ranging from about 0.5 second to about one hour. In an alternative procedure, a temperature point may be pre-determined, and the bath heated until such point is reached. Then the bath is simply cooled. In this alternative procedure, the length of time in the temperature range would be greater than 0.5 seconds.

[0018] In the invented process, an active amount, for example from about 0.1 to about 1.5% owg, of a wetting and/or scouring compound is optionally used. An example of a wetting agent is an ethoxylated and/or propoxylated fatty alcohol, and an example of a scouring agent is an ethoxylated and/or propoxylated fatty alcohol. While this type of scouring or wetting agent has been found to perform well, many other types of conventional scouring or wetting agents may also be employed. An active amount, for example from about 0.1 to about 1.5% owg, of a peroxide stabilizing compound is preferably added to the bath. Examples of peroxide stabilizing agents include, but are not limited to: organo-phosphate based agents (e.g., Diethylenetriamine penta(methylene phosphonic acid)); amino-organic acid based agents (e.g., Diethylenetriamine pentaacetic acid); organic acid based agents (e.g., Sodium salt of Gluconic Acid); polyacrylic acid based agents; earth alkaline salts (e.g., Mg<sup>2+</sup> salts); and, combinations thereof.

[0019] In the invented process for pre-treating cellulosic, or cellulosic blends with synthetic fiber, substrate, achieving a near neutral pH enables a reduction or elimination of the need for subsequent water baths. During the invented process, the bath starts with a slightly alkali pH. As the invented process progresses, a pH of about 6.0 to about 9.0, and preferably from about 6.5 to about 8.5, is achieved.

**EXAMPLES**

[0020] Typical examples for the new process are:

1 kg of 100% cotton knit material was loaded in a laboratory jet-dyeing machine. The machine was filled with water, non-foaming wetting agent/detergent before the load. Chosen liquor ratio was 1:10 such that 10l treatment liquor were used. Subsequent to loading the machine remaining chemicals were added and bath was heated up to 110°C (4°C/min). Treatment time at this temperature was 20 minutes followed by a cooling phase to 75°C (4°C/min). Finally, the bath was dropped and the fabric was centrifuged, dried and analyzed.

[0021] In a production process, the bath would be refilled after the drop, and a peroxidase (catalase) (enzymatic peroxide eliminator) would be added to remove residual peroxide. The subsequent process (e.g., dyeing) can start in the same bath.

[0022] Formulas for the treatment bath (concentrations in % on the weight of the substrate (owg) if not stated otherwise):

TABLE 1

	1	2	3	4	5	Untreated goods
Non-foaming scouring/wetting agent	0.7	0.7	0.7	0.7	0.7	
Peroxide Stabilizer	0.5	0.5	0.5	0.5	0.5	
Trisodium Citrate	2	2	1			
Copper Gluconate (ppm Cu owb)		0.8				
Urea			5			
Hydrogen Peroxide (50%)	3	3	3	3	3	
Caustic Soda (50%)	0.4	0.4	0.3	4	0.4	
Initial Ph of bath	10.5	10.2	9.7	11.5	10.1	
Final Ph of bath	7.9	7.2	7.8	10.5	7.9	

[0023] Treatment 4 (Table 1) represents a typical prior art bleach. A final pH of 10.5, such as in the prior art bleach of Treatment 4 (Table 1), requires multiple rinses. Treatment 5 (Table 1) represents a low alkali pretreatment without any activator. The following results, shown in Table 2, demonstrate that the presence of various activators allows

bleaching with an excellent level of absorbency and a suitable level of clearness while using significantly lower amounts of alkali than conventional processes.

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**TABLE 2**

Results for different fabric styles:

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- a) 100% cotton interlock knit
- b) 100% cotton haring-bone knit
- c) 100% cotton jersey knit
- d) 100% cotton piquet knit

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Results of Treatment Nos. from Table 1:	1	2	3	4	5	Un- treated goods
<b>Fabric a:</b>	MG 1-2	MG 1-3		MG 1-1		
Whiteness (CIE)	64	69		70		7
Visual Cleanliness (Seeds, etc.)	Very clean	Very clean		Very clean		Not clean
Water drop absorbency	Very high	Very high		High		None
Burst Strength (lbs./in <sup>2</sup> )	124	115		124		137
Average degree of polymerization EWN-method	3000	2300		3000		3000
<b>Fabric b:</b>	EK 19-2	EK 19-4	EK 19-3	EK 19-1	EK 30-1	
Whiteness (CIE)	60	66	63	72	54	8
Visual Cleanliness (Seeds, etc.)	Very clean	Very clean	Very clean	Very clean	Not clean	Not clean
Water drop absorbency	Very high	Very high	Very high	High	None/ Low	None
Average degree of polymerization EWN-method	2700	2200	2700	2900		3000
<b>Fabric c:</b>	EK 19-2	EK 19-4	EK 19-3	EK 19-1		
Whiteness (CIE)	61	69	64	71		28
Visual Cleanliness (Seeds, etc.)	Very clean	Very clean	Very clean	Very clean		Not Clean
Water drop absorbency	Very high	Very high	Very high	High		None
Average degree of polymerization EWN-method	2600	2300	2600	2700		3000
<b>Fabric d:</b>	EK 19-2	EK 19-4	EK 19-3	EK 19-1		
Whiteness (CIE)	57	66	62	68		
Visual Cleanliness (Seeds, etc.)	Very clean	Very clean	Very clean	Very clean		Not Clean
Water drop absorbency	Very high	Very high	Very high	High		None

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Average degree of polymerization EWN-method	2700	2300	2500	2700		Est. 3000
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Average value of polymerization (DP):

< 1800	Poor	Depending on greige fabric DP
1800-2000	Good	
2000-2400	Very good	
> 2400	Excellent	

(concentrations in % owg if not stated otherwise)

TABLE 3

	MG 11-7	MG 11-2	MG 11-3	MG 11-4
Non-foaming scouring/wetting agent	0.7	0.7	0.7	0.7
Peroxide Stabilizer	0.5	0.5	0.5	0.5
Trisodium Citrate		2		2
Tetra Acetyl Ethylene Diamine (TAED)				1
Hydrogen Peroxide (50%)	3	3	3	3
Caustic Soda (50%)	4	0.4	0.4	1(*)
Treatment Time at 110°C (min.)	15	15	15	15
Initial pH of bath	12.0	11.0	10.7	11.0
Final pH of bath	11.0	7.8	7.3	7.4
Results on 100% cotton interlock knit:				
Whiteness (CIE) after treatment	72.7	57.9	54.7	65.9
Hydrophilicity	High	Very high	poor	Very high

(\*) more alkali was used to compensate for the acid nature of TAED. Final pH was still in a range where nearly all cotton dye-procedures can be started without the need for prior rinses.

**[0024]** Treatment MG 11-7 (Table 3) represents a typical prior art bleach. The final pH of 11.0 of the prior art bleach (Treatment MG 11-7, Table 3) requires multiple rinses. Treatment MG 11-3 (Table 3) represents a bleach without the addition of any described activating compounds. Treatment MG 11-3 (Table 3) expectedly yields unacceptable whiteness and absorbency levels. The addition of activating compound Trisodium Citrate (Treatment MG 11-2, Table 3) and Trisodium Citrate plus Tetra Acetylen Ethylene Diamine (Treatment MG 11-4, Table 3) results in a preparation of cotton substrate in accordance with the present invention that is suitable for subsequent dyeing operations without additional

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rinse requirement.

**[0025]** Formulas for the treatment bath (concentrations in % owg if not stated otherwise):

TABLE 4

	1	2	3	4	5	6	7	8	9
Non-foaming scouring/wetting agent	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7
Peroxide Stabilizer	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Trisodium Citrate	2	1.5	2	2	1.5	1.5		2	
Copper Gluconate (ppm Cu owb)	0.5	0.5						0.8	0.5
Sulfur Black 1 (ppm owb)	5	5		5	5				5
Hydrogen Peroxide (50%)	3	3	3	3	3	3	3	3	3
Caustic Soda (50%)	0.4	0.4	0.4	0.4	0.4	0.4	4	0.4	0.4
Treatment Time at 110°C (min.)	30	30	30	30	30	30	30	30	30
Initial pH of bath	10.4	9.7	9.7	9.7	9.7	9.7	11.0	9.7	10.0
Final pH of bath	6.9	6.9	7.2	7.3	7.6	7.5	10.5	7.0	6.9
Residual Hydrogen Peroxide (%)	43	43	69	75	75	70	75	34	46
Results on 100% cotton interlock knit:									
Whiteness (CIE) after treatment	66.9	66.5	60.3	61.2	59.4	60.4	-	67.7	67.6
Whiteness (CIE) after one rinse with water	68.6	65.8	60.6	62.8	61.3	60.7	73.4	67.5	69.0
Comments									
Hydrophilicity	Very high	Very high	High	Fair	Fair	High	High	Very high	Very high

**[0026]** Treatment 7 (Table 4) represents a typical prior art bleach. The final pH of 10.5 of Treatment 7 (Table 4) requires multiple rinses. All other treatments in accordance with the present invention, shown in Table 4, demonstrate sufficient preparation for most dye processes without the need for rinsing. The addition of Sulfur Black 1 (Treatment 4, Table 4) has improved whiteness levels in comparison to the sole use of Trisodium citrate (Treatment 3, Table 4). Further addition of copper gluconate (Treatments 1, 2, 8 and 9, Table 4) enhances whiteness more and creates a very absorbent substrate. The hydrogen peroxide utilization increases significantly with the use of copper gluconate. (concentrations in % owg if not stated otherwise)

TABLE 5

	SS-3-13-1	SS-3-13-2	SS-3-13-3	SS-3-13-4
Non-foaming scouring/wetting agent	0.7	0.7	0.7	0.7



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TABLE 5 (continued)

	<b>SS-3-13-1</b>	<b>SS-3-13-2</b>	<b>SS-3-13-3</b>	<b>SS-3-13-4</b>
Peroxide Stabilizer	0.5	0.5	0.5	0.5
Trisodium Citrate	2	2	2	2
Copper Gluconate (ppm Cu owb)		0.5	0.5	
Sulfur Black 1 (ppm owb)			5	5
Hydrogen Peroxide (50%)	3	3	3	3
Caustic Soda (50%)	0.4	0.4	0.4	0.4
Treatment Time at 110°C (min.)	30	30	30	30
Initial pH of bath	10.4	9.8	9.8	9.9
Final pH of bath	7.4	7.0	7.2	7.6
Residual Hydrogen Peroxide (%)	65	51	36	68
Results on 100% cotton interlock knit:				
Whiteness (CIE) after treatment	57.1	59.7	63.2	58.6
Hydrophilicity	High	Very high	Very High	Fair

**[0027]** The addition of Sulfur Black 1 (Treatments SS-3-13-3 and SS-3-13-4, Table 5) in accordance with the present invention improves whiteness levels. Addition of copper gluconate (Treatment 2, Table 5) in accordance with the present invention enhances whiteness more and creates a very absorbent substrate. The combination of Sulfur Black 1 and copper gluconate demonstrates optimized conditions (Treatment SS 3-13-3, Table 5).  
(concentrations in % owg if not stated otherwise)

TABLE 6

	<b>EK-4-87-1</b>	<b>EK-4-87-2</b>	<b>EK-4-87-3</b>
Non-foaming scouring/wetting agent	0.7	0.7	0.7
Peroxide Stabilizer	0.5	0.5	0.5
Trisodium Citrate	2	2	2
Copper Gluconate (ppm Cu owb)		0.5	
Titanium Dioxide (ppm owb)			1
Sulfur Black 1 (ppm owb)	5		
Hydrogen Peroxide (50%)	3	3	3
Caustic Soda (50%)	0.4	0.4	0.4
Treatment Time at 110°C (min.)	20	20	20
Initial pH of bath	10.7	10.4	10.4

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TABLE 6 (continued)

	EK-4-87-1	EK-4-87-2	EK-4-87-3
Final pH of bath	8.3	7.4	8.2
Results on 100% cotton interlock knit:			
Whiteness (CIE) after treatment	59.6	62.4	62.1
Hydrophilicity	Poor	Very high	Poor

**[0028]** As previously mentioned hereinabove, the addition of Sulfur Black 1 in accordance with the present invention improves whiteness levels. As shown by Treatments EK-4-87-1 (Table 6) and EK-4-87-3 (Table 6), replacement of 5 ppm Sulfur Black 1 pigment with 1 ppm Titanium Dioxide pigment enhances whiteness further in accordance with the present invention.  
(concentrations in % owg if not stated otherwise)

TABLE 7

	EK-4-90-1	EK-4-90-2	EK-4-90-3	EK-4-90-4
Non-foaming scouring/wetting agent	0.7	0.7	0.7	0.7
Peroxide Stabilizer	0.5	0.5	0.5	0.5
Trisodium Citrate	2	2	2	2
Copper Gluconate (ppm Cu owb)	0.5	0.5	0.5	0.5
Titanium Dioxide (ppm owb)		2.5	5	3.3
Sulfur Black 1 (ppm owb)	5	2.5		1.7
Hydrogen Peroxide (50%)	3	3	3	3
Caustic Soda (50%)	0.4	0.4	0.4	0.4
Treatment Time at 110°C (min.)	20	20	20	20
Initial pH of bath	10.4	10.2	10.2	10.5
Final pH of bath	7.4	7.6	7.8	7.6
Results on 100% cotton interlock knit:				
Whiteness (CIE) after treatment	65.2	65.9	67.9	67.2
Hydrophilicity	Very high	Very high	Very high	Very high

**[0029]** Replacement of Sulfur Black 1 pigment (Treatment EK-4-90-1, Table 7) with Titanium Dioxide pigment (Treatment EK-4-90-3, Table 7) enhances whiteness levels. All treatments in accordance with the present invention, as shown in Table 7, result in perfectly prepared cotton substrates.  
(concentrations in % owg if not stated otherwise)

TABLE 8

	EK-4-95-1	EK-4-95-2/9	EK-4-95-6	EK-4-95-7	EK-4-95-8
5 Non-foaming scouring/wetting agent	0.5	0.5	0.5	0.5	0.5
Peroxide Stabilizer	0.7				
Sodium Gluconate (60%)		0.2	0.2	0.2	0.2
10 Trisodium Citrate		0.5	0.5	0.5	0.5
MgSO <sub>4</sub> x 6 H <sub>2</sub> O		0.25	0.25	0.25	0.25
Sulfur Black 1 (ppm owb)		8			2.2
15 Titanium Dioxide (ppm Ti owb)			4.4	13.2	8.8
Copper Gluconate (ppm Cu owb)		0.54	0.54	0.54	0.54
Urea		0.2	0.2	0.2	0.2
Hydrogen Peroxide (50%)	3	3	3	3	3
20 Caustic Soda (50%)	4	0.8	0.8	0.8	0.8
Treatment Time at 110°C (min.)	20	20	20	20	20
25 Initial pH of bath	12.0	11.4	11.2	11.0	11.0
Final pH of bath	11.0	8.3	8.3	8.3	8.3
30 Results on 100% cotton interlock knit:					
Whiteness (CIE) after treatment	70.7	59.6	62.7	62.7	62.3
35 Hydrophilicity	High	Very high	Very high	Very high	Very high

[0030] Treatment EK-4-95-1 (Table 8) represents a typical prior art bleach. The final pH of 11.0 of Treatment EK-4-95-1 (Table 8) requires multiple rinses. All other treatments in accordance with the present invention shown in Table 8 lead to highly acceptable preparation results without the need for rinsing.

## Claims

1. A process for pre-treating a cellulosic, or cellulosic blends with synthetic fiber, substrate comprising the steps of:

- providing a vessel;
- providing a cellulosic, or cellulosic blends with synthetic fiber, substrate;
- providing a water bath;
- adding an active amount of an activating compound selected from the group of: salts of organic acids, organic amine derivatives, transitional metal salts, transitional metal complexes pigments, and combinations thereof;
- adding an active amount of caustic soda to achieve a starting bath having a slightly alkali pH;
- adding an active amount of hydrogen peroxide;
- heating the water bath to a temperature in excess of 50 degrees centigrade for a period of time;
- achieving a pH from about 6.0 to about 9.0 at the end of a bleaching cycle; and
- dropping the bath; wherein said process is **characterized by** the absence of a rinsing cycle.

2. The process for pre-treating a cellulosic, or cellulosic blends with synthetic fiber, substrate according to claim 1 wherein said activating compound adding step is performed with about 0.2 to about 5.0% based on the weight of

the substrate of the salt of an organic acid where a salt of an organic acid is selected from: sodium salts of citric acid, sodium stearate, sodium salts of gluconic acid, sodium oleate, potassium salts of citric acid, potassium stearate, potassium salts of gluconic acid, potassium oleate, ammonium salts of citric acid, ammonium stearate, ammonium salts of gluconic acid, ammonium oleate, and combinations thereof, and where an organic amine derivative is selected from: Urea, Dicyandiamide, Tetraacetyl Ethylene Diamine, Acetyl Caprolactam and combinations thereof.

3. The process for pre-treating a cellulosic, or cellulosic blends with synthetic fiber, substrate according to claim 1 wherein said activating compound adding step is performed with about 0.1 to about 10ppm based on the weight of the bath of the transitional metal where said transitional metal complex is selected from: copper gluconate, copper sulfate, copper acetate, copper carbonate, copper citrate, copper nitrate, copper EDTA, copper complexes and combinations thereof.

4. The process for pre-treating a cellulosic, or cellulosic blends with synthetic fiber, substrate according to claim 1 wherein said activating compound adding step is performed with about 1 to about 200ppm based on the weight of the bath of the pigment, where said pigment is selected from from: Sulfur Black 1 with a particle size less than 150 $\mu$ m, fully pre-oxidized sulfur dyes, titanium Dioxide with a particle size less than 150 $\mu$ m and combinations thereof.

5. The process for pre-treating a cellulosic, or cellulosic blends with synthetic fiber, substrate according to claim 1 wherein the water bath is heated to a temperature ranging from about 80 degrees centigrade to about 140 degrees centigrade for a period ranging from about 0.5 second to about one hour.

6. The process for pre-treating a cellulosic, or cellulosic blends with synthetic fiber, substrate according to claim 1 further comprising the step of:

- adding an active amount of a wetting and/or scouring compound; or
- adding an active amount of a peroxide stabilizing compound; or adding both.

7. A composition for pre-treating a cellulosic, or cellulosic blends with synthetic fiber, substrate prior to dyeing comprising:

- a non-foaming scouring/wetting agent;
- a first activating compound selected from the group consisting of a transitional metal, transitional metal salts, transitional metal complexes, salts of organic acids, and combinations thereof, said salt of organic acids being selected from sodium salts of citric acid, sodium stearate, sodium salts of gluconic acid, sodium oleate, potassium salt of citric acid, potassium stearate, potassium salt of gluconic acid, potassium oleate, ammonium salts of citric acid, ammonium stearate, ammonium salts of gluconic acid, ammonium oleate, and combinations thereof, where said salt of organic acid is about 0.2 to about 5.0% based on the weight of the substrate ("owg").
- optionally, a second activating compound selected from the group of: organic amine derivatives, pigments, and combinations thereof;
- caustic soda; and
- hydrogen peroxide,

wherein said composition has a slightly alkali pH.

8. A composition according to claim 7, wherein said second activating compound is an organic amine-derivative selected from urea, dicyandiamide, tetra-acetyl-ethylene-diamine, acetyl-caprolactam, and combinations thereof, where said organic amine derivative is about 0.2 to about 5.0% owg.

9. A composition according to claim 7, wherein said first activating compound is a transitional metal complex selected from copper gluconate, copper sulfate, copper acetate, copper carbonate, copper citrate, copper nitrate, copper EDTA, copper complexes, and combinations thereof, where said transitional metal is about 0.1 to about 10ppm based on the weight of the bath ("owb").

10. A composition according to claim 7, wherein said second activating compound is a pigment selected from pigmented Sulfur Black I with a particle size less than 150 $\mu$ m, fully pre-oxidized sulfur dyes, and combinations thereof, where said pigment is about 1 to about 200ppm owb.

11. A composition according to claim 7, wherein said non-foaming scouring/wetting agent is selected from ethoxylated fatty alcohol and propoxylated fatty alcohol; and wherein said non-foaming scouring/wetting agent is about 0.1 to about 1.5% owg.

12. A composition according to claim 7 further comprising a peroxide stabilizer, where said peroxide stabilizer is selected from an organo-phosphate based agent, an amino-organic acid based agent, an organic acid based agent, a polyacrylic acid based agent, an earth alkaline salt, and combinations thereof, where said organo-phosphate based agent is Diethylenetriamine penta(methylene phosphonic acid), said amino-organic acid based agent is Diethylenetriamine pentaacetic acid, said organic acid based agent is Sodium salt of Gluconic Acid, and said earth alkaline salt is Mg<sup>+2</sup> salt.

13. A process for pre-treating a cellulosic, or cellulosic blends with synthetic fiber, substrate comprising the steps of:

- providing a vessel;
- providing a cellulosic, or cellulosic blends with synthetic fiber, substrate;
- providing a water bath;
- heating said water bath to a temperature between 80°C to 140°C;
- adding an active amount of an activating compound selected from the group of: salts of organic acids, organic amine derivatives, transitional metal salts, transitional metal complexes pigments, and combinations thereof;
- adding an active amount of caustic soda to achieve a starting bath having a slightly alkali pH;
- adding an active amount of hydrogen peroxide;
- heating the water bath to a temperature in excess of 50 degrees centigrade for a period of time;
- achieving a pH from about 6.0 to about 9.0 at the end of a bleaching cycle; and
- dropping the bath.

14. A composition for pre-treating a cellulosic, or cellulosic blends with synthetic fiber, substrate prior to dyeing comprising:

- a non-foaming scouring/wetting agent;
- an activating compound formed of a mixture of urea and copper gluconate;
- caustic soda; and
- hydrogen peroxide.

## Patentansprüche

1. Verfahren zur Vorbehandlung eines Cellulose- oder Cellulose-/Synthetikfasergemischs, das folgende Schritte umfasst:

- Bereitstellung eines Behälters;
- Bereitstellung eines Cellulose- oder Cellulose-/Synthetikfasergemischsubstrats;
- Bereitstellung eines Wasserbades;
- Hinzufügen einer aktiven Menge einer Aktivierungsverbindung, ausgewählt aus der Gruppe, bestehend aus: Salzen organischer Säuren, organischen Aminderivaten, Übergangsmetallsalzen, Übergangsmetallkomplexen, Pigmenten, und Kombinationen davon;
- Hinzufügen einer aktiven Menge Ätznatron, um ein Ausgangsbad mit einem leicht basischen pH zu erhalten;
- Hinzufügen einer aktiven Menge Wasserstoffperoxid;
- Erwärmen des Wasserbads auf eine Temperatur über 50 Grad Celsius während eines Zeitraums;
- Erzielen eines pH von ungefähr 6,0 bis ungefähr 9,0 am Ende des Bleichzyklus; und
- Ablassen des Bades; wobei das Verfahren **gekennzeichnet ist durch** die Abwesenheit eines Spülzyklus.

2. Verfahren zur Vorbehandlung eines Cellulose- oder Cellulose-/Synthetikfasergemischsubstrats nach Anspruch 1, wobei der Schritt des Hinzufügens der Aktivierungsverbindung ausgeführt wird mit ungefähr 0,2 bis ungefähr 5,0 %, basierend auf dem Gewicht des Substrats, des Salzes einer organischen Säure, wobei ein Salz einer organischen Säure ausgewählt ist unter: Natriumsalzen von Citronensäure, Natriumstearat, Natriumsalzen von Gluconsäure, Natriumoleat, Kaliumsalzen von Citronensäure, Kaliumstearat, Kaliumsalzen von Gluconsäure; Kaliumoleat, Ammoniumsalzen von Citronensäure, Ammoniumstearat, Ammoniumsalzen von Gluconsäure, Ammoniumoleat, und Kombinationen davon, und wobei ein organisches Aminderivat ausgewählt ist unter: Harnstoff, Dicyan-

diamid, Tetraacetylenylendiamin, Acetylcaprolactam und Kombinationen davon.

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3. Verfahren zur Vorbehandlung eines Cellulose- oder Cellulose-/Synthetikfasergemischsubstrats nach Anspruch 1, wobei der Schritt des Hinzufügens der Aktivierungsverbindung ausgeführt wird mit ungefähr 0,1 bis ungefähr 10 ppm, basierend auf dem Gewicht des Bades, des Übergangsmetalls, wobei der Übergangsmetallkomplex ausgewählt ist unter: Kupfergluconat, Kupfersulfat, Kupferacetat, Kupfercarbonat, Kupfercitrat, Kupfernitrat, Kupfer-EDTA; Kupferkomplexen und Kombinationen davon.
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4. Verfahren zur Vorbehandlung eines Cellulose- oder Cellulose-/Synthetikfasergemischsubstrats nach Anspruch 1, wobei, basierend auf dem Gewicht des Bades, der Schritt des Hinzufügens der Aktivierungsverbindung ausgeführt wird mit ungefähr 1 bis ungefähr 200 ppm des Pigments, wobei das Pigment ausgewählt ist unter: Schwefelschwarz 1 mit einer Teilchengröße unter 150 µm, vollständig voroxidierten Schwefelfarbstoffen, Titandioxid mit einer Teilchengröße unter 150 µm, und Kombinationen davon.
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5. Verfahren zur Vorbehandlung eines Cellulose- oder Cellulose-/Synthetikfasergemischsubstrats nach Anspruch 1, wird das Wasserbad auf eine Temperatur im Bereich von ungefähr 80 Grad Celsius bis ungefähr 140 Grad Celsius während eines Zeitraums im Bereich von ungefähr 0,5 Sekunden bis ungefähr einer Stunde erwärmt wird.
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6. Verfahren zur Vorbehandlung eines Cellulose- oder Cellulose-/Synthetikfasergemischs nach Anspruch 1, das weiterhin folgenden Schritt umfasst:
- Hinzufügen einer aktiven Menge eines Netz- und/oder Scheuermittels; oder
  - Hinzufügen einer aktiven Menge eines Peroxidstabilisators; oder Hinzufügen beider.
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7. Zusammensetzung zur Vorbehandlung eines Cellulose- oder Cellulose-/Synthetikfasergemischsubstrats vor dem Färben, umfassend:
- ein nicht schäumendes Scheuer-/Netzmittel;
  - eine erste Aktivierungsverbindung, ausgewählt aus der Gruppe, bestehend aus einem Übergangsmetall, Übergangsmetallsalzen, Übergangsmetallkomplexen, Salzen organischer Säuren, und Kombinationen davon, wobei das Salz organischer Säuren ausgewählt ist unter Natriumsalzen von Citronensäure, Natriumstearat, Natriumsalzen von Gluconsäure, Natriumoleat, Kaliumsalz von Citronensäure, Kaliumstearat, Kaliumsalz von Gluconsäure, Kaliumoleat, Ammoniumsalzen von Citronensäure, Ammoniumstearat, Ammoniumsalzen von Gluconsäure, Ammoniumoleat, und Kombinationen davon, wobei das Salz der organischen Säure, basierend auf dem Gewicht des Substrats ("owg"), ungefähr zwischen 0,2 und ungefähr 5,0 % ist.
  - optional Auswählen einer zweiten Aktivierungsverbindung aus der Gruppe, bestehend aus: Organischen Aminderivaten, Pigmenten, und Kombinationen davon;
  - Ätznatron; und
  - Wasserstoffperoxid,
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- wobei die Zusammensetzung einen leicht basischen pH aufweist.
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8. Zusammensetzung nach Anspruch 7, wobei es sich bei der zweiten Aktivierungsverbindung um ein organisches Aminderivat handelt, ausgewählt unter Harnstoff, Dicyandiamid, Tetraacetylenylendiamin, Acetylcaprolactam, und Kombinationen davon, wobei das organische Aminderivat ungefähr 0,2 bis ungefähr 5,0 % owg ist.
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9. Zusammensetzung nach Anspruch 7, wobei die erste Aktivierungsverbindung ein Übergangsmetallkomplex ist, ausgewählt unter Kupfergluconat, Kupfersulfat, Kupferacetat, Kupfercarbonat, Kupfercitrat, Kupfernitrat, Kupfer-EDTA, Kupferkomplexen, und Kombinationen davon, wobei das Übergangsmetall ungefähr 0,1 bis ungefähr 10 ppm ist, basierend auf dem Gewicht des Bades ("owb").
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10. Zusammensetzung nach Anspruch 7, wobei die zweite Aktivierungsverbindung ein Pigment ist, ausgewählt unter pigmentiertem Schwefelschwarz 1 mit einer Teilchengröße unter 150 µm, vollständig voroxidierten Schwefelfarbstoffen, und Kombinationen davon, wobei das Pigment ungefähr 1 bis ungefähr 200 ppm owb ist
11. Zusammensetzung nach Anspruch 7, wobei das nicht schäumende Scheuer-/Netzmittel ausgewählt ist unter ethoxyliertem Fettalkohol und propoxyliertem Fettalkohol; und wobei das nicht schäumende Scheuer-/Netzmittel ungefähr 0,1 bis ungefähr 1,5 % owg ist.

12. Zusammensetzung nach Anspruch 7, ausserdem umfassend einen Peroxidstabilisator, wobei der Peroxidstabilisator ausgewählt ist unter einem Mittel auf der Basis von Organophosphat, einem Mittel auf der Basis einer organischen Aminsäure, einem Mittel auf der Basis einer organischen Säure, einem Mittel auf der Basis von Polyacrylsäure, einem Erdalkalisalz, und Kombinationen davon, wobei das Mittel auf der Basis von Organophosphat die Diethylentriaminpenta(methylenphosphonsäure) ist, das Mittel auf der Basis von organischer Aminsäure die Diethylentriaminpentaessigsäure ist, das Mittel auf der Basis einer organischen Säure das Natriumsalz von Glucosäure ist, und das Erdalkalisalz das  $Mg^{+2}$ -Salz ist.

13. Verfahren zur Vorbehandlung eines Cellulose- oder Cellulose-/Synthetikfasergemischs, das folgende Schritte umfasst:

- Bereitstellung eines Behälters;
- Bereitstellung eines Cellulose- oder Cellulose-/Synthetikfasergemischsubstrats;
- Bereitstellung eines Wasserbades;
- Erwärmen des Wasserbades auf eine Temperatur zwischen 80 °C und 140 °C;
- Hinzufügen einer aktiven Menge einer Aktivierungsverbindung, ausgewählt aus der Gruppe, bestehend aus Salzen organischer Säuren, organischen Aminderivaten, Übergangsmetallsalzen, Übergangsmetallkomplexen, Pigmenten, und Kombinationen davon;
- Hinzufügen einer aktiven Menge Ätznatron, um ein Ausgangsbad mit einem leicht basischen pH zu erhalten;
- Hinzufügen einer aktiven Menge Wasserstoffperoxid;
- Erwärmen des Wasserbades auf eine Temperatur über 50 Grad Celsius während eines Zeitraums;
- Erzielen eines pH von ungefähr 6,0 bis ungefähr 9,0 am Ende des Bleichzyklus; und
- Ablassen des Bades.

14. Zusammensetzung zur Vorbehandlung eines Cellulose- oder Cellulose-/Synthetikfasergemischsubstrats vor dem Färben, umfassend:

- ein nicht schäumendes Scheuer-/Netzmittel;
- eine Aktivierungsverbindung, gebildet aus einem Gemisch aus Harnstoff und Kupfergluconat;
- Ätznatron; und
- Wasserstoffperoxid.

## Revendications

1. Procédé permettant de prétraiter un substrat cellulosique, ou de mélanges cellulosiques avec une fibre synthétique, comprenant les étapes consistant à :

- se procurer un récipient;
- se procurer un substrat cellulosique, ou de mélanges cellulosiques avec une fibre synthétique;
- se procurer un bain d'eau;
- ajouter une quantité active d'un composé activant choisi dans le groupe des sels d'acides organiques, des dérivés d'amine organiques, des sels de métaux de transition, des complexes de métaux de transition, des pigments et de leurs combinaisons;
- ajouter une quantité active de soude caustique pour obtenir un bain de départ ayant un pH légèrement alcalin;
- ajouter une quantité active de peroxyde d'hydrogène;
- chauffer le bain d'eau à une température supérieure à 50 degrés centigrades pendant un certain temps;
- obtenir un pH d'environ 6,0 à environ 9,0 à la fin d'un cycle de blanchiment; et
- abaisser le bain; ledit procédé étant **caractérisé par** l'absence de cycle de rinçage.

2. Procédé permettant de prétraiter un substrat cellulosique, ou de mélanges cellulosiques avec une fibre synthétique, selon la revendication 1 dans lequel ladite étape d'addition d'un composé activant est réalisée avec environ 0,2 à environ 5,0%, par rapport au poids du substrat, de sel d'un acide organique, le sel d'acide organique étant choisi parmi les sels de sodium d'acide citrique, le stéarate de sodium, les sels de sodium d'acide gluconique, l'oléate de sodium, les sels de potassium d'acide citrique, le stéarate de potassium, les sels de potassium d'acide gluconique, l'oléate de potassium, les sels d'ammonium d'acide citrique, le stéarate d'ammonium, les sels d'ammonium d'acide gluconique, l'oléate d'ammonium, et leurs combinaisons; et un dérivé d'amine organique étant choisi parmi l'urée, le dicyandiamide ou la tétraacétyléthylènediamine, l'acétylcaprolactame, et leurs combinaisons.

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3. Procédé permettant de prétraiter un substrat cellulosique, ou de mélanges cellulosiques avec une fibre synthétique, selon la revendication 1 dans lequel ladite étape d'addition d'un composé activant est réalisée avec environ 0,1 à environ 10 ppm, par rapport au poids du bain, du métal de transition, ledit complexe de métal de transition étant choisi parmi le gluconate de cuivre, le sulfate de cuivre, l'acétate de cuivre, le carbonate de cuivre, le citrate de cuivre, le nitrate de cuivre, l'EDTA de cuivre, les complexes de cuivre, et leurs combinaisons.
4. Procédé permettant de prétraiter un substrat cellulosique, ou de mélanges cellulosiques avec une fibre synthétique, selon la revendication 1 dans lequel ladite étape d'addition d'un composé activant est réalisée avec environ 1 à environ 200 ppm, par rapport au poids du bain, du pigment, le pigment étant choisi parmi le noir de soufre 1 ayant une granulométrie inférieure à 150 µm, les colorants soufrés entièrement préoxydés, le dioxyde de titane ayant une granulométrie inférieure à 150 µm, et leurs combinaisons.
5. Procédé permettant de prétraiter un substrat cellulosique, ou de mélanges cellulosiques avec une fibre synthétique, selon la revendication 1 dans lequel le bain d'eau est chauffé à une température s'échelonnant d'environ 80 degrés centigrades à environ 140 degrés centigrades pendant une durée s'échelonnant d'environ 0,5 seconde à environ une heure.
6. Procédé permettant de prétraiter un substrat cellulosique, ou de mélanges cellulosiques avec une fibre synthétique, selon la revendication 1 comprenant en outre l'étape consistant à :
- ajouter une quantité active d'un composé mouillant et/ou lavant; ou
  - ajouter une quantité active d'un composé stabilisant le peroxyde; ou ajouter les deux.
7. Composition permettant de prétraiter un substrat cellulosique, ou de mélanges cellulosiques avec une fibre synthétique, avant de le teindre, comprenant:
- un agent mouillant et/ou lavant non moussant;
  - un premier composé activant choisi dans le groupe constitué d'un métal de transition, des sels de métaux de transition, des complexes de métaux de transition, des sels d'acides organiques, et de leurs combinaisons, ledit sel d'acide organique étant choisi parmi les sels de sodium d'acide citrique, le stéarate de sodium, les sels de sodium d'acide gluconique, l'oléate de sodium, le sel de potassium d'acide citrique, le stéarate de potassium, le sel de potassium d'acide gluconique, l'oléate de potassium, les sels d'ammonium d'acide citrique, le stéarate d'ammonium, les sels d'ammonium d'acide gluconique, l'oléate d'ammonium, et leurs combinaisons, ledit sel d'acide organique représentant environ 0,2 à environ 5,0% par rapport au poids du substrat ("pds");
  - le cas échéant, un second composé activant choisi dans le groupe constitué des dérivés d'amines organiques, des pigments et de leurs combinaisons;
  - de la soude caustique; et
  - du peroxyde d'hydrogène;
- ladite composition ayant un pH légèrement alcalin.
8. Composition selon la revendication 7, dans laquelle ledit second composé activant est un dérivé d'amine organique choisi parmi l'urée, le dicyandiamide, la tétraacétyléthylènediamine, l'acétylcaprolactame, et leurs combinaisons, ledit dérivé d'amine organique représentant environ 0,2 à environ 5,0% pds.
9. Composition selon la revendication 7, dans laquelle ledit premier composé activant est un complexe de métal de transition choisi parmi le gluconate de cuivre, le sulfate de cuivre, l'acétate de cuivre, le carbonate de cuivre, le citrate de cuivre, le nitrate de cuivre, l'EDTA de cuivre, les complexes de cuivre, et leurs combinaisons, ledit métal de transition représentant environ 0,1 à environ 10 ppm, par rapport au poids du bain ("pdb").
10. Composition selon la revendication 7, dans laquelle ledit second composé activant est un pigment choisi parmi le noir de soufre 1 pigmenté ayant une granulométrie inférieure à 150 µm les colorants soufrés entièrement préoxydés, et leurs combinaisons, ledit pigment représentant environ 1 à environ 200 ppm pdb.
11. Composition selon la revendication 7, dans laquelle ledit agent lavant/mouillant non moussant est choisi parmi un alcool gras éthoxylé et un alcool gras propoxylé; et dans laquelle ledit agent lavant/ mouillant non moussant représente environ 0,1 à environ 1,5% pds.



5 12. Composition selon la revendication 7 comprenant en outre un stabilisant de peroxyde, ledit stabilisant de peroxyde étant choisi parmi un agent à base d'organophosphate, un agent à base d'acide organique aminé, un agent à base d'acide organique, un agent à base d'acide polyacrylique, un sel alcalino-terreux, et leurs combinaisons, ledit agent à base d'organophosphate étant l'acide diéthylènetriaminepentaméthylène phosphonique, ledit agent à base d'acide organique aminé étant l'acide diéthylènetriaminepentaacétique, ledit agent à base d'acide organique étant le sel de sodium d'acide gluconique et ledit sel alcalino-terreux étant un sel de  $Mg^{2+}$ .

10 13. Procédé permettant de prétraiter un substrat cellulosique, ou de mélanges cellulosiques avec une fibre synthétique, comprenant les étapes consistant à :

- se procurer un récipient;
- se procurer un substrat cellulosique, ou de mélanges cellulosiques avec une fibre synthétique;
- se procurer un bain d'eau;
- chauffer ledit bain d'eau à une température comprise entre 80°C et 140°C;
- 15 - ajouter une quantité active d'un composé activant choisi dans le groupe des sels d'acides organiques, des dérivés d'amine organiques, des métaux de transition, des sels de métaux de transition, des complexes de métaux de transition, des pigments, et de leurs combinaisons;
- ajouter une quantité active de soude caustique pour obtenir un bain de départ ayant un pH légèrement alcalin;
- ajouter une quantité active de peroxyde d'hydrogène;
- 20 - chauffer le bain d'eau à une température supérieure à 50 degrés centigrades pendant un laps de temps;
- obtenir un pH d'environ 6,0 à environ 9,0 à la fin d'un cycle de blanchiment; et
- abaisser le bain.

25 14. Composition permettant de prétraiter un substrat cellulosique, ou de mélanges cellulosiques avec une fibre synthétique, avant de le teindre, comprenant:

- un agent mouillant et/ou lavant non moussant;
- un composé activant formé d'un mélange d'urée et de gluconate de cuivre;
- de la soude caustique; et
- 30 - du peroxyde d'hydrogène.

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