

This invention relates to mercerizing and/or causticizing wetting agents containing alkali metal and/or ammonium salts of 2-ethyl hexyl sulfate, end-capped polyethers and, optionally, trialkyl phosphates.

5 The object of mercerizing and causticizing cotton, i.e. the treatment of cellulose with lyes of different concentration, is to provide the fibers with better gloss effects, with increased receptivity for dyes, greater
10 tensile strengths and a softer, fuller feel. However, the concentrated caustic soda used in practice does not have a particularly high wetting power. In addition, wetting is complicated by the hydrophobic substances adhering to the cellulose fibers. Accordingly, wetting agents are frequently added to the mercerizing and/or causticizing
15 liquors to obtain a fast and uniform reaction of the lye with the cellulose (Chwala/Anger: "Handbuch der Textilhilsmittel", pages 347 - 350, 1977).

Besides having high wetting power, the wetting agents must not cause any foaming in the liquors, must be stable
20 over a wide alkali metal concentration range and must show very good biodegradability. However, most of the known mercerizing and/or causticizing wetting agents do not effectively satisfy these stringent requirements. For example, the salts of 2-ethyl hexyl sulfate frequently used
25 as wetting agents tend to cause excessive foaming in mercerizing and/or causticizing liquors.

DE-A-12 45 898 describes mixtures of branched-chain carboxylic acid esters, phosphoric acid esters of aliphatic alcohols, fatty acids or soaps and alkoxyated compounds or
30 alkyl sulfuric acid esters as low-foaming wetting agents. In addition, it is known from Chemical Abstracts, Abstract Ca 93, 115887 g - (1980) that fatty alcohols alkoxyated with 20 mol ethylene oxide in combination with 2-ethyl hexyl sodium sulfate improve the wetting power of mercerizing liquors and reduce foaming therein.
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End-capped alkyl alcohol ethoxylates are known as foam-inhibiting additives in low-foaming cleaning preparations from DE-A-33 15 951, DE-A-37 27 378 and DE-A-38 00 490.

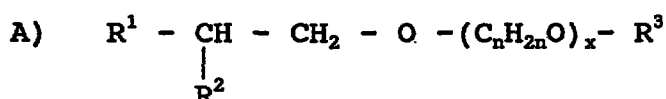
5 The problem addressed by the present invention was to provide low-foaming, alkali-stable and readily biodegradable mercerizing and/or causticizing wetting agents of high shrinkage wetting power.

10 It has now surprisingly been found that wetting agents containing alkali metal and/or ammonium salts of 2-ethyl hexyl sulfate, special end-capped polyethers and, optionally, trialkohol phosphates largely satisfy the stringent demands made of them.

15 Accordingly, the present invention relates to mercerizing and/or causticizing wetting agents containing 15 to 30% by weight alkali metal and/or ammonium salts of 2-ethylhexyl sulfate,

1 to 10% by weight of a mixture of end-capped polyethers corresponding to the following general formulae

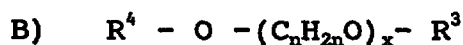
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in which R^1 is a linear C_{1-20} alkyl radical, R^2 is a linear C_{1-20} alkyl radical and R^3 is a C_{1-22} alkyl, alkenyl, cycloalkyl, aryl, arylalkyl or alkylaryl radical and n is the number 2 and/or 3 and/or 4 and x is a number of 2 to 25, and

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in which R^4 is a C_{8-22} alkyl or alkenyl radical and R^3 is a C_{1-22} alkyl, alkenyl, cycloalkyl, aryl, arylalkyl or alkylaryl radical and n is the number and 2 and/or 3 and/or 4 and x is a number of 2 to 25,

0 to 5% by weight trialkyl phosphates containing 1 to 6

carbon atoms in the alkyl groups.

The mercerizing and/or causticizing wetting agents according to the invention contain end-capped polyethers corresponding to general formulae A and B, in which R^1 is a linear C_{8-12} alkyl radical, R^2 is a linear C_{6-10} alkyl radical and R^3 is a C_{1-6} alkyl radical and R^4 is a C_{12-18} alkyl or alkenyl radical, n is the number 2 and x is a number of 2 to 10. The ratio by weight between the end-capped polyethers A and B is preferably between 0.1 and 10 and more preferably between 0.9 and 1.1.

The end-capped polyethers corresponding to general formula A may be obtained by known methods. Guerbert alcohols prepared by reaction of saturated primary C_{1-20} alcohols are alkoxyated with ethylene oxide, propylene oxide and/or butylene oxide, preferably ethoxylated ("Chemische Technologie", Vol. 7, pages 131 - 132, Carl-Hanser-Verlag, München-Wien (1986)), in the presence of alkali (cf. for example Angew. Chem. 64, 213 - 220 (1952)) and are then preferably reacted with C_{1-6} alkyl halides, for example methyl chloride, ethyl chloride, propyl chloride, butyl chloride, butyl bromide, pentyl chloride and/or hexyl chloride at temperatures in the range from 60 to 140°C (DE-OS 37 44 525). Examples of the alcohols required for the preparation of Guerbet alcohols are capryl, capric, lauryl, myristyl, cetyl, stearyl alcohol and mixtures of these alcohols.

The end-capped polyethers corresponding to general formula B may also be prepared by methods known from the literature. The educts used are aliphatic, saturated and/or unsaturated C_{8-22} alcohols of natural and/or synthetic origin, for example octyl, decyl, dodecyl, myristyl, cetyl, stearyl, oleyl, linoleyl, behenyl, erucyl alcohol or mixtures of these alcohols. C_{12-18} alcohols, for example lauryl, myristyl, cetyl, stearyl, oleyl alcohol and mixtures thereof are preferred. The alkoxylation of these

alcohols with ethylene oxide, propylene oxide and/or butylene oxide, preferably with ethylene oxide, is carried out by known industrial methods (cf. for example "Chemische Technologie" Vol. 7, pages 131 - 132, Carl-Hanser-Verlag, München-Wien (1986)). The alkoxyated alcohols are then reacted in known manner with, preferably, C₁₋₆ alkyl halides, for example methyl chloride, ethyl chloride, propyl chloride, butyl chloride, butyl bromide, pentyl chloride and/or hexyl chloride to form the end-capped polyethers.

The alkali metal and/or ammonium salts of 2-ethyl hexyl sulfate present in the wetting agents according to the invention are commercially available products, for example Texapon^(R) EHS, a product of Henkel KGaA.

The trialkyl phosphates optionally present in the wetting agents according to the invention are prepared in known manner, for example by reaction of phosphorus oxychloride with alkyl alcohols (Chem. and Ind. 1962, 1032 - 1035). The alcohols used are, for example, methanol, ethanol, propanol, n-butanol, n-pentanol or n-hexanol. The wetting agents according to the invention preferably contain from 0.5 to 5% by weight trialkyl phosphates.

The wetting agents according to the invention are prepared at 18 to 50°C by stirring the individual components, optionally after melting, to form a homogeneous solution.

The wetting agents according to the invention, which are present in the form of aqueous solutions, are distinguished by high shrinkage wetting power and minimal foaming. In addition, they show very good biodegradability. The wetting agents according to the invention contain only small quantities, if any, of solvents, such as polyalcohols, for example hexylene glycol and/or butylene glycol.

By virtue of their stability over a wide alkali metal concentration range, the wetting agents according to the invention may be used both for mercerizing and for causticizing, for example, cellulose-containing woven fabrics,

knitted fabrics and/or yarns. The content of wetting agents according to the invention in the mercerizing and/or causticizing liquors is between 2 and 12 g/l liquor.

5 **EXAMPLES**

1. Preparation of mercerizing and/or causticizing wetting agents according to the invention

- 10 a) 81 % by weight 2-ethyl hexyl sulfate sodium salt
(active substance content: 30% by weight,
water content: 70% by weight)
2.5 % by weight C₁₂₋₁₈ fatty alcohol x 5 mol ethylene
oxide butyl ether
2.5 % by weight 2-hexyl-1-decanol x 6 mol ethylene
oxide butyl ether
15 2 % by weight n-tributyl phosphate
4 % by weight hexylene glycol
8 % by weight water
- 20 b) 81 % by weight 2-ethyl hexyl sulfate sodium salt
(active substance content 30% by weight,
water content 70% by weight)
2.5 % by weight C₁₂₋₁₈ fatty alcohol x 5 mol ethylene
oxide butyl ether
2.5 % by weight 2-octyl-1-dodecanol x 8 mol ethylene
oxide butyl ether
25 2 % by weight n-tributyl phosphate
4 % by weight hexylene glycol
8 % by weight water
- 30 c) 81 % by weight 2-ethyl hexyl sulfate sodium salt
(active substance content 30% by weight,
water content 70% by weight)
2.5 % by weight C₁₂₋₁₈ fatty alcohol x 7 mol ethylene
oxide butyl ether
35 2.5 % by weight 2-octyl-1-dodecanol x 8 mol ethylene

oxide butyl ether

- 2 % by weight n-tributyl phosphate
- 4 % by weight hexylene glycol
- 8 % by weight water

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Shrinkage wetting power of the wetting agents according to the invention

10 The shrinkage wetting power was determined in accordance with DIN 53987 (Landolt method) at 18°C using a quantity of wetting agent of 5 g/l (shrinkage power expressed in %).

	Time in seconds						
	10	20	30	40	50	60	90
Caustic soda 20°Bé (16.7% by weight)							
Wetting agent 1a	13	17.5	20	21	22	23	25
Wetting agent 1b	11.5	16	18	20	21.5	22	23
Wetting agent 1c	11	15.5	18	19	20.5	21.5	23
Commercial product based on							
2-ethyl hexyl sulfate, sodium salt	10	15	17	19	20	20.5	20.5
Caustic soda 22°Bé (25.6% by weight)							
Wetting agent 1a	14	18	20.5	22	22.5	23	24
Wetting agent 1b	15	19	21	23	24	24.5	26
Wetting agent 1c	13	17	19	21	22	23	24
Commercial product based on							
2-ethyl hexyl sulfate, sodium salt	14	17.5	19	20.5	21	22	23.5

	Time in seconds						
	10	20	30	40	50	60	90
Caustic soda 25°Bé (29.7% by weight)							
Wetting agent 1a	17	22	24	24	25	25	26
Wetting agent 1b	19	24	26	27	28	28.5	29
Wetting agent 1c	16	22	24	25	25.5	26	27
Commercial product based on 2-ethyl hexyl sulfate, sodium salt	18	23	24	25	25	26	26
Caustic soda 28°Bé (35 % by weight)							
Wetting agent 1a	15	21	23	23.5	24	24	25
Wetting agent 1b	17	25.5	27	28	28	28.5	29
Wetting agent 1c	11	17	20	21.5	22	23	24
Commercial product based on 2-ethyl hexyl sulfate, sodium salt	14	20	22	22	23	23	24

	Time in seconds						
	10	20	30	40	50	60	90
Caustic soda 30°Bé (38.9% by weight)							
Wetting agent 1a	4	12	17	20	22	23	24
Wetting agent 1b	5	12.5	18	20	22	22.5	24.5
Wetting agent 1c	5	12	15.5	20	21	22	24
Commercial product based on 2-ethyl hexyl sulfate, sodium salt	10	21	22	24	25	25	25.5
Caustic soda 32°Bé (42.8% by weight)							
Wetting agent 1a	2	5	8	10	13	14	18
Wetting agent 1b	4	7	11	14	16	16.5	20
Wetting agent 1c	5	12	15.5	20	21	22	24
Commercial product based on 2-ethyl hexyl sulfate, sodium salt	3	9	14.5	17	19.5	21	22

Foaming power of the wetting agents according to the invention

Foaming power was determined at 18°C by the glass frit method described in Melliand Textilberichte 48, 311 - 315 and 450 - 456 using a quantity of wetting agent of 5 g/l. The foam height is expressed in mm (200 mm is the maximum height under the test conditions).

Table 2

	Time in minutes	
	1	60
Caustic soda 20°Bé (16.7% by weight)		
Wetting agent 1a	35	40
Wetting agent 1b	35	60
Wetting agent 1c	30	35
Commercial product based on 2-ethyl hexyl sulfate, sodium salt	>200	-
Caustic soda 22°Bé (25.6% by weight)		
Wetting agent 1a	40	100
Wetting agent 1b	50	125
Wetting agent 1c	35	50
Commercial product based on 2-ethyl hexyl sulfate, sodium salt	>200	-
Caustic soda 25°Bé (29.7% by weight)		
Wetting agent 1a	50	200
Wetting agent 1b	40	200
Wetting agent 1c	50	120
Commercial product based on 2-ethyl hexyl sulfate, sodium salt	>200	-

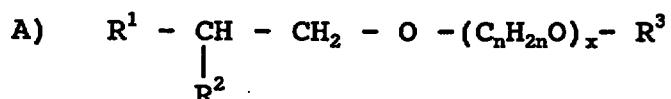
	Time in minutes	
	1	60
Caustic soda 28°Bé (35 % by weight)		
Wetting agent 1a	35	35
Wetting agent 1b	35	35
Wetting agent 1c	30	70
Commercial product based on		
2-ethyl hexyl sulfate,	100	>200
sodium salt		
Caustic soda 30°Bé (38.9% by weight)		
Wetting agent 1a	35	35
Wetting agent 1b	35	35
Wetting agent 1c	35	35
Commercial product based on		
2-ethyl hexyl sulfate,	100	200
sodium salt		
Caustic soda 32°Bé (42.8% by weight)		
Wetting agent 1a	35	35
Wetting agent 1b	35	35
Wetting agent 1c	35	35
Commercial product based on		
2-ethyl hexyl sulfate,	20	120
sodium salt		

CLAIMS

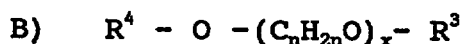
1. Mercerizing and/or causticizing wetting agents containing

15 to 30% by weight alkali metal and/or ammonium salts of 2-ethyl hexyl sulfate,

1 to 10% by weight of a mixture of end-capped polyethers corresponding to the following general formulae



in which R^1 is a linear C_{1-20} alkyl radical, R^2 is a linear C_{1-20} alkyl radical and R^3 is a C_{1-22} alkyl, alkenyl, cycloalkyl, aryl, arylalkyl or alkylaryl radical and n is the number 2 and/or 3 and/or 4 and x is a number of 2 to 25, and



in which R^4 is a C_{8-22} alkyl or alkenyl radical and R^3 is a C_{1-22} alkyl, alkenyl, cycloalkyl, aryl, arylalkyl or alkylaryl radical and n is the number 2 and/or 3 and/or 4 and x is a number of 2 to 25,

0 to 5% by weight trialkyl phosphates containing 1 to 6 carbon atoms in the alkyl groups.

2. Mercerizing and/or causticizing wetting agents as claimed in claim 1, characterized in that, in general formulae A and B, R^1 is a linear C_{8-12} alkyl radical, R^2 is a linear C_{6-10} alkyl radical, R^3 is a C_{1-6} alkyl radical and R^4 is a C_{12-18} alkyl or alkenyl radical, n is the number 2 and x is a number of 2 to 10.

3. Mercerizing and/or causticizing wetting agents as claimed in claim 1 or 2, characterized in that the ratio by weight of A to B is between 0.1 and 10 and preferably between 0.9 and 1.1.



4. Mercerizing and/or causticizing wetting agents as claimed in one or more of claims 1 to 3, characterized in that the trialkyl phosphate content is from 0.5 to 5% by weight.



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