

[54] WASHING OR DYEING WITH A
NON-TERMINAL, VICINAL ALKANEDIOL
ADDUCT OF ETHYLENE AND PROPYLENE
OXIDES

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[58] Field of Search..... **8/137, 93, 169**

[56] **References Cited**
UNITED STATES PATENTS

2,671,811 3/1954 Baird 252/358

2,679,522 5/1954 DeGroot 252/358

OTHER PUBLICATIONS

Chem. & Eng. News, 1/30/1956, Vol. 34, No. 5,
pages P-477-480.

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[57] **ABSTRACT**

In the process of treating textiles which comprises
contacting said textiles with an aqueous textile treat-
ing bath containing a wetting agent and recovering
said treated textiles, the improvement which consists
in utilizing from 0.1 to 5 gm/liter of a poorly foaming
alkylene oxide adduct of from 5 to 20 mols of ethyl-
ene oxide and from 1 to 10 mols of propylene oxide in
a mol ratio of 1:0.1 to 1 to a nonterminal, vicinal al-
kanediol having from 10 to 20 carbon atoms, as said
wetting agent.

7 Claims, No Drawings

WASHING OR DYEING WITH A NON-TERMINAL, VICINAL ALKANEDIOL ADDUCT OF ETHYLENE AND PROPYLENE OXIDES

PRIOR ART

In the many washing and wetting processes used in the treatment of textiles, including dyeing, considerable foaming problems occur with the usual wetting and washing agents, which up to now could only be partly solved by the addition of anti-foaming substances. Spots are frequently formed on the goods on separation of the anti-foaming agents. On the other hand, the addition of special poorly foaming non-ionic wetting and washing agents has not hitherto led to the desired results since, when the foaming was sufficiently reduced, the wetting and washing action was not satisfactory.

OBJECTS OF THE INVENTION

It is an object of the present invention to provide a development in the process of treating textiles which comprises contacting said textiles with an aqueous textile treating bath containing a wetting agent and recovering said treated textiles, wherein the improvement consists in utilizing from 0.1 to 5 gm/liter of a poorly foaming alkylene oxide adduct of from 5 to 20 mols of ethylene oxide and from 1 to 10 mols of propylene oxide in a mol ratio of 1:0.1 to 1 to a non-terminal, vicinal alkanediol having from 10 to 20 carbon atoms, as said wetting agent.

It is a further object of the present invention to provide a poorly foaming wetting agent for textile treatment comprising an alkylene oxide adduct of from 5 to 20 mols of ethylene oxide and from 1 to 10 mols of propylene oxide in a mol ratio of 1:0.1 to 1 to a non-terminal, vicinal alkanediol having from 10 to 20 carbon atoms.

These and other objects of the invention will become apparent as the description thereof proceeds.

DESCRIPTION OF THE INVENTION

According to the present invention there is provided a method of carrying out textile treatment processes using an aqueous bath containing a wetting or washing agent, and wherein the said wetting or washing agent is a poorly foaming alkylene oxide adduct of from 5 to 20 mols of ethylene oxide and from 1 to 10 mols of propylene oxide adducted to alkanediols containing 10 to 20 carbon atoms, the hydroxyl groups of which are substantially non-terminal and adjacent to one another, as the said wetting and washing agents. By "non-terminal" are meant diols in which the adjacent hydroxyl groups are distributed almost statistically over the whole alkyl chain.

More particularly, the present invention provides an improvement in the process of treating textiles which comprises contacting said textiles with an aqueous textile treating bath containing a wetting agent and recovering said treated textiles, the said improvement consists in utilizing from 0.1 to 5 gm/liter of a poorly foaming alkylene oxide adduct of from 5 to 20 mols of ethylene oxide and from 1 to 10 mols of propylene oxide in a mol ratio of 1:0.1 to 1 to a non-terminal, vicinal alkanediol having from 10 to 20 carbon atoms, as said wetting agent.

The present invention is further directed to a poorly foaming wetting agent for textile treatment comprising

an alkylene oxide adduct of from 5 to 20 mols of ethylene oxide and from 1 to 10 mols of propylene oxide in a mol ratio of 1:0.1 to 1 to a non-terminal, vicinal alkanediol having from 10 to 20 carbon atoms.

The starting materials for the preparation of the adducts are aliphatic diols, usually alkanediols of 12 to 20 carbon atoms, with non-terminal and adjacent or vicinal hydroxyl groups. Such diols are prepared in known way from linear aliphatic olefins with isomeric double bonds, for example by epoxidation with per-acids or hydrogen peroxide and lower carboxylic acids forming per-acids, and subsequent saponification of the epoxides. The products obtained are normally mixtures of various diols, the diol group being statistically distributed over the carbon chain. The amount of 1,2-diols in the mixture is very small.

The reaction of these non-terminal, vicinal alkanediols with the alkylene oxides is effected in known way in the presence of acid or alkaline catalysts, preferably using elevated temperatures and pressures. Firstly, the desired amount of ethylene oxide is added onto the diol followed by the amount of propylene oxide. In practice, the reaction with ethylene oxide is preferably carried out in a two-stage process, in which 1 mol of a diol or diol mixture is first reacted with 1 mol of ethylene oxide. After separating any unreacted diol, the further reaction with the desired amount of ethylene oxide is effected. Subsequent to the addition of the ethylene oxide, the further reaction with propylene oxide is carried out in the usual way. In the reaction preferably only one polyalkylene glycol chain is formed per diol molecule, since substantially only the primary hydroxyl groups resulting from the first reaction stage react further with the alkylene oxides at sufficient speed.

Suitable products according to the present invention are in particular those which contain from 5 to 10 mols of ethylene oxide and from 3 to 10 mols of propylene oxide added onto the diol and in which the molar amounts of the ethylene oxide to propylene oxide added onto the diol are in the ratio of from 1:0.1-1, preferably from 1:0.3-1. Diols or diol mixtures having alkyl chain lengths of from 14 to 18 carbon atoms are preferred as starting materials. Examples of suitable substances are the adducts of 5 mol of ethylene oxide and 3 mol of propylene oxide, or 7 mol of ethylene oxide and 3 mol of propylene oxide or 7 mol of ethylene oxide and 5 mol of propylene oxide to diols or diol mixtures of an average chain length of 15 to 17 carbon atoms.

The said products are marked by very little foaming and have a high wetting and washing action. These products may be utilized at temperatures ranging from about 25°C to 95°C while maintaining the properties of non-foaming with excellent wetting and washing capabilities.

The adducts described are very suitable as wetting and washing agents in many processes of textile manufacture. For example, they are used for the improvement of the penetration of finishing baths; in the sizing of material which contains difficulty wettable waxes or preparations; as wetting agents in carbonizing liquors; for continuous scouring, dyeing, washing, desizing and bleaching processes, where they ensure safety and acceleration of the operations; in the pigment pad process of cotton on foulard machines; for cross coil dyeing in a backwashing machine of worsted tops; in pre-scouring and dyeing in high temperature reel dyeing

machines; in dyeing in high temperature apparatus; in nozzle washing machines; for jig processes and so forth. In all such cases the products are marked by extremely low foaming, so that the use of special antifoaming agents is unnecessary. It is to be noted that the tendency of the adducts to foam decreases still further as the temperature rises, so that the adducts of the present invention are particularly suitable for use in baths which are operated at elevated temperatures.

The quantity of the products to be added to the baths depends upon the intended use and upon the desired results. In general, the amount added is between about 0.1 and 5 gm/liter, preferably between about 0.5 and 3 gm/liter. For example, dyebaths require a concentration of about 1 to 3 gm/liter; yarn washing liquors require a concentration of about 1 to 2 gm/liter; sizing and finishing liquors require a concentration of about 0.5 to 1 gm/liter and carbonizing liquors require a concentration of about 0.5 to 1.5 gm/liter. The baths have also a good stability at higher temperatures or with a higher concentration of electrolyte. The adducts are normally soluble in water at low temperatures giving a clear solution; at higher temperatures above the so-called turbidity point the solutions become cloudy without, however, any separation occurring. The superior wetting and washing properties are also maintained above the turbidity point, while the already minimal foaming tendency decreases practically to zero in this temperature region.

The following examples are merely illustrative of the present invention without being deemed limitative in any manner thereof.

EXAMPLES

The following Table contains data on important technical application properties of the substances used according to the present invention as well as some commercial products for comparison. It can be observed from the Table that the said products are practically non-foaming with excellent wetting and washing properties.

Technical application properties of the substances according to the invention as well as of some comparative products.

Product	Wetting action acc. to DIN 53901 (wetting times in sec.)			Washing action in % brightening with 1 gm/l of surface-active product			Foaming test with 1g/l of surface-active product at 60°C.	
	0.5 gm/l	1.0 gm/l	1.5 gm/l	Wool at 30°C Bath ratio 1:50	Cotton at 90°C Bath ratio 1:12	Diolen at 40°C Bath ratio 1:30	Beating method acc. to DIN 53902 ml foam	In the cross coil dyeing machine cm. foam
Products according to the invention:								
C_{16} diol + 5EO + 3PO	84	34	12	80	65	51	0	0
C_{16} diol + 7EO + 3PO	72	71	10	67	65	53	0	0
C_{16} diol + 7EO + 5PO	79	21	9	36	59	42	0	0
Commercial surface-active compounds as products for comparison:								
Decyl alcohol + 3EO	78	21	8	45	50	29	20	10
C_{16} -fatty alcohol + 10EO	143	72	47	70	66	60	250	foams over (> 30 cm)
lonylphenol + 9EO	93	32	13	71	75	72	200	foams over (> 30 cm)

EXAMPLE 1

Cross-wound bobbins of cotton were boiled for 30 min. at 95°C with 1 gm/liter of the adduct of 7 mols of ethylene oxide and 3 mols of propylene oxide to a diol

mixture having chain lengths of 15 to 18 carbon atoms. Then without changing the bath, a substantive dyeing with Sirius light red 4 BL (Registered Trade Mark) was effected with the addition of sodium sulfate. The addition of a further dispersing agent was not necessary, since the diol adduct has a simultaneous dispersing action.

Both during the boiling and also during the dyeing processes, no objectionable foam occurred. A good even dyeing was obtained.

EXAMPLE 2

Cotton piece goods were padded on the foulard at a speed of 60 m/min. with 20 gm/liter of Indanthrene blue GCD (Registered Trade Mark) with addition of 3 gm/liter of an adduct of 77 mols of ethylene oxide and 5 mols of propylene oxide to a diol mixture of chain lengths 15 to 18 carbon atoms. After intermediate drying the goods were vatted as usual with hydrogensulfite in alkaline liquor. The dyeing was developed with steam and again scoured with the adduct of 5 mols of ethylene oxide and 3 mols of propylene oxide to a diol mixture of chain lengths 15 to 18 carbon atoms.

Both the padding process and the further treatment stages proceeded completely free from trouble, since practically no foaming occurs. The result of the dyeing was satisfactory.

EXAMPLE 3

The piece goods dyed according to Example 2 were passed at 70 m/min through the following baths of a continuous washing plant:

1st Bath	40°C	0.5 gm/liter H ₂ O ₂
2nd Bath	40°C	0.5 ml/liter acetic acid 60%

3rd Bath	95°C	2 gm/liter of the adduct from 5 mols of ethylene oxide and 3 mols of propylene oxide to an alkanediol mixture having from 15 to 18 carbon atoms
4th Bath	60°C	Water
5th Bath	25°C	Water

The fine-pored stable foam occurring during use of commercial washing agents was completely lacking in this case. The plant ran for several days free from trouble when the above-mentioned poorly foaming adduct was used. The cleaning result was satisfactory.

Although the present invention has been disclosed in connection with a few preferred embodiments thereof, variations and modifications may be resorted to by those skilled in the art without departing from the principles of the new invention. All of these variations and modifications are considered to be within the true spirit and scope of the present invention as disclosed in the foregoing description and defined by the appended claims.

We claim:

1. In the process of treating textiles which comprises contacting said textiles with an aqueous textile treating bath containing a wetting agent and recovering said treated textiles, the improvement which consists in utilizing from 0.1 to 5 gm/liter of a poorly foaming alkyl-ene oxide adduct of from 5 to 20 mols of ethylene oxide and from 1 to 10 mols of propylene oxide in a mol ratio of 1:0.1 to 1 to a nonterminal, vininal alkanediol having from 10 to 20 carbon atoms, as said wetting agent.

2. The process as claimed in claim 1, in which the adduct is from 5 to 10 mols of ethylene oxide and from 3 to 10 mols of propylene oxide adducted to said alkanediol having from 14 to 18 carbon atoms.

3. The process as claimed in claim 1, in which the diol adduct has a molar ratio of ethylene oxide to propylene oxide of from 1:0.3 to 1:1.

4. The process as claimed in claim 1, in which the

aqueous bath contains from about 0.5 to 3 gm/liter of solution of said poorly foaming adduct.

5. The process as claimed in claim 1, in which said poorly foaming adduct is selected from the group consisting of the adducts of 5 mols of ethylene oxide and 3 mols of propylene oxide to an alkanediol or alkanediol mixture of 15 to 17 carbon atoms, the adducts of 7 mols of ethylene oxide and 3 mols of propylene oxide to an alkanediol or alkanediol mixture of 15 to 17 carbon atoms, and the adducts of 7 mols of ethylene oxide and 5 mols of propylene oxide to alkanediols or alkanediol mixtures of 15 to 17 carbon atoms.

6. The process as claimed in claim 1, in which said poorly foaming adduct is selected from the group consisting of the adducts of 5 mols of ethylene oxide and 3 mols of propylene oxide to a hexadecanediol, the adducts of 7 mols of ethylene oxide and 3 mols of propylene oxide to a hexadecanediol, and the adducts of 7 mols of ethylene oxide and 5 mols of propylene oxide to a hexadecanediol.

7. The process as claimed in claim 1, in which said poorly foaming adduct is selected from the group consisting of the adducts of 7 mols of ethylene oxide and 3 mols of propylene oxide to an alkanediol mixture of 15 to 18 carbon atoms, the adducts of 7 mols of ethylene oxide and 5 mols of propylene oxide to an alkanediol mixture of 15 to 18 carbon atoms and the adducts of 5 mols of ethylene oxide and 3 mols of propylene oxide to an alkanediol mixture of 15 to 18 carbon atoms.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,892,522 Dated July 1, 1975

Inventor(s) Wolfgang Schade et al.

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

On the Cover Sheet, the following should be added:

-- [30] Foreign Application Priority Data

Dec. 17, 1971 Germany -----2162672

Column 4, line 18, "77 mols" should read

-- 7 mols --.

Signed and Sealed this

fourth Day of May 1976

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks