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(54) Alkalizer for fibre reactive dyes in cotton.

(57) A premixed liquid alkalizer, suitable for use as a direct textile bath additive, comprises a water solution containing tro-potassium phosphate, potassium hydroxide and/or sodium hydroxide and potassium carbonate and/or sodium carbonate.

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ALKALIZER FOR FIBER REACTIVE DYES ON COTTON

Background of the Invention

In the dyeing of cellulosic materials using fibre-reactive dyes, it is the practice to add the cellulosic material and dye to the dyebath first. Next a neutral salt (e.g. sodium sulfate or sodium chloride) is added and the bath is stirred and heated to a prescribed temperature for a prescribed period of time. During this period of time, the dye becomes absobed by the material as the result of the "salting out" effect of the salt which had been added. This "salting out" step is essential for placing dye molecules in very close proximity to the macromolecules of the cellulosic materials. At this point, the material has absorbed the dye, but the dye is not held fast to the fibre.

After the salt addition and stirring as prescribed, an alkaline material is added to promote a chemical reaction between the dye molecules and atoms of the cellulosic fibres. As a result of this reaction, the dye combines chemically with the cellulosic material. The dye, so bonded, will not wash out of the cellulose. This is known as a "wash-fast" dyeing. Without the "salting out", which increases the substantivity of unreacted dye molecules with the fibre, much of the dye would not have reacted with the fibre and the colour would have been lost.

A fibre-reactive dye molecule is made up of a colour portion and a chemically-reactive portion. When used to dye cellulosic materials, the chemically-reactive portion reacts with an hydroxyl group on the celluiose macromolecule under alkaline conditions to form a chemical bond between the cullulosic material and the colour portion of the dye molecule.

In order to ensure a good yield of dyeing, electrolytes in the form of neutral salts are added to the dyebath to enhance the substantivity of the dye molecules on to the cellulosic material fibres, before chemical reaction between the hydroxyl groups and the chemically-reactive portion of the dye takes place.

Addition of an alkaline material then, under proper conditions of time and temperature, casues the absorbed dye molecules to react with the hydroxyl groups of the cellulosic fibres to form chemical bonds between the dye molecule and the macromolecules of cellulose. There is also a tendency for the dye molecule to react with other hydroxyl groups, such as are present in water. This is known as hydrolysis. When this occurs, the water molecule becomes chemicaly bonded to the colour portion of the dye molecule, and hence, the dye molecule is lost for combining with the cellulose. The colour washes out of the cellulosic material. Hydrolysis thus results in a lower colour yield on the cellulosic material.

A good yield in dyeing, then, depends on causing a large proportion of the dye molecules to absorb, or "salt out", on to the cellulosic macromolecules, before chemical reaction occurs. This must then be followed by the addition of an alkaline material which promotes the chemical reaction between the cellulosic fibre and the dye molecule and minimizes the reaction between dye molecules and water molecules.

In the past, a number of chemicals have been used as the alkalizing material. For example, sodium hydroxide alone or in combination with soda ash (sodium carbonate); sodium carbonate alone; tri-sodium phosphate; sodium bicarbonate; tri-potassium phosphate alone or in combination with a strong base such as potassium hydroxide and/or sodium hydroxide.

Summary of the Invention

It has been discovered that a combination of tri-potassium phosphate (K_3PO_4), potassium hydroxide (KOH) [or sodium hydroxide (NaOH)] plus potassium carbonate (K_2CO_3) [or sodium caronate (Na_2CO_3)],when used as an alkalizer for dyeing cellulosic material with fibre-reactive dyes, yields dyeings with less hydrolysis and therefore increased yield of colour on the cellulose.

It has been further discovered that the combination of these ingredients can be made as a convenient direct textile bath additive in the form of an aqueous solution with the total solids ranging from about 10% up to approximately 50% by weight of the solution. The limit of lower percentage is dictated by the economics of having a more concentrated solution for transportation. The upper limit of concentration is determined by the temperature at which a mixture can be stored. For storage above 10°C, the concentration can even be higher.

Detailed Description of the Invention

The liquid alkalizer of the present invention can be formulated over a wide range of concentration. It has been found that the combination of the ingredients of the present invention can be made in aqueous solutions with the total solids ranging from about 10%to up to approximately 50% by weight of the solution. The lower limit is dictated mainly by the economics of having a more concentrated solution for transportation, while the upper limit of concentration is determined by the temperature at which a mixture can be stored. For storage above 10°C, the concentration can be higher. It has been found that a combination of K_3PO_4, KOH (or NaOH) plus K_2CO_3 (or Na_2CO_3), when used in combination as an alkalizer for dyeing a cellulosic material with fibre-reactive dyes, provides dyeings with less hydrolysis and therefore increased yield of colour on the cellulose.

The broad and preferred concentration ranges for the K_3PO_4, KOH (or NaOH), and K_2CO_3 (or Na_2CO_3) for liquid alkalizers of the present invention are tabulated below in weight percent of the solution:

	Broad Range	Preferred Range
K_3PO_4	5-45%	10-35%
KOH	0-10%	2-8%
K_2CO_3	2-10%	3-8%
Water:	Balance	Balance

Although the potassium hyroxide (or equivalent sodium hydroxide) is optional, it is a preferred component in that it functions in the dye-bath to neutralize acidity.

NaOH may be substituted in equal amount for KOH and Na_2CO_3 may be substituted in equal amounts for the K_2CO_3.

A typical method used for making the liquid alkalizers of the present invention is as follows: To prepare a formulation of the present invention, the appropriate amount of water is first added to a vessel. The K_3PO_4 can be added with stirring and, when dissolved, the desired amount of KOH is then added. Next, Na_2CO_3 or K_2CO_3 is added. The alkalizer mix is stirred and drained into drums after cooling below 40°C. The drummed material is the alkalizer ready for use or sale.

As an alternative to adding solids, the liquid alkalizer can be prepared using a phosphoric acid (H_3PO_4), a 45% solution of KOH and a 47% solution of K_2CO_3, all of which are commercially available.

For example, formula (A) of TABLE II would be made as follows:

36.30%	Water
46.14%	KOH solution 45%
13.56%	H_3PO_4 75%
4.00%	Na_2CO_3
100.00%	

The ingredients would be added in the same order as listed, using cooling as necessary to keep temperature below 70°C. Cool to 40°C and pack into drums.

Formula (F) of TABLE II would be made as follows:

8.1% Water
61.8% KOH - 45% solution
21.6% H_3PO_4 -75% solution
8.5% K_2CO_3 47% solution

Add ingredients in the order listed, using cooling to keep the temperature below 70°C. Cool finally to

below 40°C and pack into drums.

The alkalizer formulations of the present invention may be used for the dyeing of cellulosic materials with fibre-reactive dyes as follows, using an Ahiba Laboratory dyeing apparatus to carry out the dyeing: Into a suitable dyeing beaker of about 230 ml volume,. 100 ml of water at 80°F (27°C), 7 grams of sodium chloride, 0.05 grams of surfactant, Tanaterge LV (Sybron Chemicals, Inc), and 0.3 grams of Remazol REd F3B Fibre-Reactive Dye (American Hoechst) are added. Place the beaker in the water bath of the Ahiba device. Mount a swatch of 100% woven cotton on an agitator holder and insert it into the beaker. The agitator is capable of simultaneous vertical and rotational motion. The cotton swatch in held below the surface of the liquid at the lowest point of the agitator movement. The Ahiba bath is held at 80°F (27°C) while agitating for 10 minutes. Then about 0.5 gm of liquid alkalizer is added and the bath is agitated for 10 minutes. The bath is then heated to 140°F (60°C), and agitated for 45 minutes. The cotton swatch is then removed from the bath, washed with cold water, then hot water, then soap solution, then hot water and finally cold water. The swatch is then dried and the dyed colour evaluated.

The above dyeing technique is used with three different alkalizers. These three alkalizers are formulas (A), (B), and (G) of TABLE II. Formula (G) is a Sybron Chemicals, Inc. commercial alkalzier sold under the trademark ALKAFLO®. Using the ALKAFLO alkalizer as the reference standard alkalizer, the colours of 100% cotton swatches are compared and tested by means of the ACS Spectro Sensor (Applied Color Systems Inc.).

A specific description of the evaluation of the comparative test data obtained from the results is set forth in TABLE I.

The dry dyed cotton sample was folded over twice and then placed in the sample holder of the ACS Spectro Sensor. A scan of the sample is then run automatically and the spectro data automatically stored in the IBM PC computer which is connected to the ACS Sensor.

The cotton sample which was dyed using ALKAFLO liquid alkalizer as the alkalizer is arbitrarily taken as the standard, with the strength reading set at 100. The spectro data of the other sampels are compared automatically by the IBM PC and strength results are printed out.

Readings of 100 are equal to satandard. Readings above 100 show geater dye strength than standard. Readings below 100 show weaker type stength than standard.

The results of dyeing using Remazol Red F3B with three different alkalizers are listed below in the TABLE I:

TABLE I

ALKALIZER	STRENGTH
Formula G	100
Formula A	105
Formula B	101

It can be seen from the above TABLE I that the formulas of the present invention result in greater dye strength than the standard.

The following formulations in TABLE II below represent typical examples of suitable liquid alkalizer compositions of the present invention in weight percent:

TABLE II

(A)	22.0%	K_3PO_4	(E)	
	3.3	KOH		28.6% K_3PO_4
	4.0%	Na_2CO_3		4.0% Na_2CO_3
	70.7%	Water		67.4% Water
	100.0%			100.0%
(B)	20.7%	K_3PO_4	(F)	
	3.1%	KOH		35.0% K_3PO_4
	8.0%	Na_2CO_3		4.0% K_2CO_3
	68.2%	Water		61.0% Water
	100.0%			100.0%
(C)	31.0%	K_3PO_4	(G)	31.2% K_3PO_4
	4.0%	KOH		8.0% KOH
	8.0%	K_2CO_3		3.9 NaOH
	52.0%	Water		56.9% Water
	100.0%			100.0%
(D)	35.0%	K_3PO_4		
	4.0%	KOH		
	4.0%	K_2CO_3		
	57.0%	Water		
	100.0%			

A small amount, such as 0.1%, of sodium gluconate or other suitable sequestering agent may be added to any of the above formulations, to prevent the formation of precipitates caused by impurities in the water or chemicals used.

Although particular embodiments of the present invention have been disclosed herein for purposes of explanation, further modifications or variations thereof will be apparent to those skilled in the art to which this invention pertains.

Claims

1. An alkalizer, suitable for use as a textile bath additive, which comprises a water solution containing tri-potassium phosphate, characterised in that the alkaliser comprises a premixed liquid composition suitable for direct addition to a textile bath containing, by weight:

Tri-potassium phosphate 5-45%
Potassium hydroxide and/or sodium hydroxide 0-10%
Potassium carbonate and/or sodium carbonate 2-10%
Water Balance

2. A composition according to claim 1, which contains tri-potassium phosphate, potassium hydroxide and potassium carbonate.

3. A composition according to claim 1 which contains tri-potassium phosphate, sodium hydroxide and sodium carbonate.

4. A composition according to any preceding claim, which comprises a water solution containing, by weight:

Tri-potassium phosphate 10-35%
Potassium hydrozide and/or sodium hydroxide 2-8%
Potassium carbonate and/or sodium carbonate 3-8%
Water Balance

5. A method of controlling the alkali content of a reactive dye bath process, which comprises:

(a) providing a liquid reactive dye-bath solution which contains water, surfactant, salt and a dye,

(b) placing in the bath a cellulose-containing textile material to be dyed and heating the dye-bath for a prescribed period of time, and

(c) adding an alkalizer to the bath, characterised in that the alkalizer comprises a premixed liquid solution which contains, by weight, from 5 to 45% of tri-potassium phosphate, up to 10% of potassium hydroxide and/or sodium hydroxide, and from 2 to 10% of potassium carbonate and/or sodium carbonate, the alkalizer being present in an amount sufficient to control the alkali concentration of the dye-bath at a level sufficient to allow the reactive dye to form a firm chemical bond with the cellulose-containing textile material when heated for a prescribed period of time.

6. A method according to claim 5, in which the alkalizer contains tri-potassium phosphate, potassium hydroxide and potassium carbonate.

7. A method according to claim 5, in which the alkalizer contains tri-potassium phosphate, sodium hydroxide and sodium carbonate.

8. A method according to any of claims 5 to 7, in which the alkalizer comprises a water solution containing, by weight:

Tri-potassium phosphate 10-35%
Potassium hydroxide and/or sodium hydroxide 2-8%
Potassium carbonate and/or sodium carbonate 3-8%
Water Balance.