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Specification No. 124383, Application No. 124383, Dated 10th December, 1969. Complete Specification left on 12th October, 1970. Application Accepted 26th August, 1971.

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"A PROCESS FOR THE REDUCTION OF A VAT DYE OR SIMILAR TYPE OF DYES"

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH RAFI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI OF 1860)

Provisional Specification: Section-4.

The following Specification describes the nature of this invention.

This is an invention by Handady Venkatakrishna Udupa, Director, Mysore Seshaier Venkatachalapathy, Scientist and Sankaranarayanier Chidambaram, Scientist, all of the Central Electrochemical Research Institute, Karaikudi, Madras State, all Indians.

This invention relates to the discovery of a new reducing agent, Dialdehyde Starch (DAS) for dyeing textile materials with vat dyes and similar type of dyes which require the use of reducing agents like sodium hydrosulphite.

Hitherto it has been proposed to reduce vat dyes by employing generally sodium hydrosulphite and zinc hydrosulphite in alkaline medium. It had also been found earlier that oxycellulose behaves in a similar manner to hydrosulphite in the reduction of vat dyes from hot alkaline suspensions.

This is open to objection that the reducing agents reported in literature suffer from the following drawbacks:—

(i) Unstable, (ii) costly, (iii) non-availability in the market, (iv) to be imported and (v) insoluble material (oxycellulose) remaining in the bath.

The object of this invention is to obviate these disadvantages by the use of the new reducing agent, dialdehyde starch now suggested. According to the present invention, the vat dye is reduced to a leuco compound by using alkaline solution of dialdehyde starch and the textile material is impregnated with the leuco dye in the bath. Afterwards, the textile material is removed from the vats, washed with water and wrinkled and kept in air for oxidation and drying.

To these ends, the invention broadly consists first in the reduction of the vat dye by the use of an alkaline solution of dialdehyde starch to prepare the dye bath. Then the textile materials are impregnated and later exposed for the air oxidation. In this connection, it may be mentioned that an alkaline solution of sodium hydrosulphite has only a copper number of 13 as against a copper number of 6 to 11 only for oxycellulose and 68 for dialdehyde starch, the new reducing agent.

The following typical examples are given to illustrate the invention:

#### EXAMPLE I-(Vat dyeing with Vat Brown-2 G)

2 g of dialdehyde starch was added to 35 cc of water containing 2 g of sodium hydroxide in a flask. 0.5 g of vat

Brown-2 G was added to it and the whole contents was heated to boiling and kept for 2 minutes. The textile material (5 g in the form of a hank) which had undergone pretreatment, with auxiliaries like Turky red oil and wetting agents, was immersed in the vat bath for 15 minutes. Then the textile material was removed, wrinked, washed with water and exposed to air for drying and oxidation. A brilliant brown coloured textile material was obtained, with a shade superior to that obtained from a bath using 2 g of sodium hydrosulphite instead of dialdehyde starch.

# EXAMPLE II-[Dyeing with Vat Yellow-G (N)]

The procedure was the same as given under Example I except that 0.5 g of vat yellow G (N) was taken instead of Vat Brown-2 G. A brilliant yellow coloured material was obtained which was superior to the sample dyed using 2 g. of sodium hydrosulphite.

## EXAMPLE III-(Dyeing with Vat Violet R. R.)

The procedure was the same as given under the Example I except that 0.5 g of Vat Violet R. R. was used instead of Vat Brown 2 G. A brilliant violet coloured textile material was obtained with the same advantage as indicated for the other two dyes.

The following are the main advantages of the invention:

- (1) The reducing agent employed in this process is handled in a quite stable form as DAS and is generated as needed by heating with alkali.
- (2) The reducing agent employed in this process is cheaper when compared to sodium hydrosulphite and does not leave a residue as in the case of oxycellulose.
- (3) It has copper number nearly 5 times more than sodium hydrosulphite which makes it a superior reducing agent weight for weight. The reducing power of the reagent employed is therefore very high and hence imparts brilliancy to the textile material.
- (4) This reducing agent can be produced indigenously from starchs (Indian Patent No. 79,075) and is substitute for sodium hydrosulphite and in certain cases is also superior to it.
- (5) The use of this reducing agent, DAS, in textile dyeing should lead to considerable economy.
- (6) The use of this reducing agent will avoid any sulphur residues on the yarn leading to degradation of cellulose.

#### Complete Specification Section-4.

The following Specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed:—

This invention relates to a process for the reduction of a vat dye and has particular reference to the discovery of a new reducing agent, dialdehyde starch (DAS) for dyeing textile materials with vat dyes which require the use of reducing agents like sodium hydrosulphite.

Hitherto it has been proposed to reduce vat dyes by employing generally sodium hydrosulphite and zinc hydrosulphite in alkaline medium. It had also been found earlier that oxycellulose behaves in a similar manner to hydrosulphite in the reduction of vat dyes from hot alkaline suspensions.

PRICE: TWO RUPEES.

This is open to objection that the reducing agents reported in literature suffer from the following drawbacks:

(i) Unstable, (ii) costly, (iii) non-availability in the market, (iv) to be imported and (v) insoluble material (oxycellulose) remaining in the bath.

The object of this invention is to obviate these disadvantages by the use of the new reducing agent, dialdehyde starch now suggested.

According to the present invention, there is provided a process for the reduction of a vat dye by the use of a dye bath consisting of a reducing agent in an alkaline medium characterised in that dialdehyde starch is used as the reducing agent to prepare the dye bath.

An alkaline solution of dialdehyde starch is used to prepare the dye bath.

The reducing agent is generated from dialdehyde starch as needed by heating with alkali.

Starch oxidised to dialdehyde starch using electrolytically regenerated periodic acid in accordance with the process of Indian Patent No. 79075 may be used.

Sodium hydrosulphite may be mixed with dialdehyde starch.

Thus, sodium hydrosulphite may be added to dialdehyde starch.

The reducing agent for carrying out the process may comprise dialdehyde starch and sodium hydrosulphite.

Thus the vat dye is reduced to a leuco compound by using alkaline solution of dialdehyde starch and the textile material is impregnated with the leuco dye in the bath. Afterwards, the textile is removed from the vats, washed with water and wrinkled and kept in air for oxidation and drying.

Thus, first the vat dye is reduced by the use of an alkaline solution of dialdehyde starch to prepare the dye bath. (By "dye bath" we mean a solution containing the dye, alkali and the reducing agent.) Then the textile materials are impregnated and exposed for the air oxidation.

The following are the main advantages of the invention:

- (1) The reducing agent employed in this is handled in a quite stable form as DAS and is generated as needed by heating with alkali.
- (2) The reducing agent employed in this process is cheaper when compared to sodium hydrosulphite and does not leave a residue as in the case of oxycellulose.
- (3) The use of this reducing agent, DAS, in textile dyeing should lead to considerable economy.
- (4) The use of this reducing agent will avoid any sulphur residues on the yarn leading to degration of cellulose.

The following typical examples are given to illustrate the invention:

### EXAMPLE I-(Vat dyeing with Vat Brown-2G)

2 g. of dialdehyde starch was added to 35 cc of water containing 2 g. of sodium hydroxide in a flask. 0.5 g. of vat Brown-2G was added to it and the whole contents was heated to boiling and kept for 2 minutes. The textile material (5 g. in the form of a hank) which had undergone pretreatment, with auxiliaries like Turkey red oil and wetting agents, was immersed in the vat bath for 15 minutes. Then the textile material was removed, wrinkled, washed with water and exposed to air for drying and oxidation. A brilliant brown coloured textile material was obtained, with a shade superior to that obtained from a bath using 2 g. of sodium hydrosulphite instead of dialdehyde starch.

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EXAMPLE II-[Dyeing with Vat Yellow-G (N)]

The procedure was the same as given under Example I except that 0.5 g of vat yellow G (N) was taken instead of Vat Brown-2 G. A brilliant yellow coloured material was obtained which was superior to the sample dyed using 2 g. of sodium hydrosulphite.

#### EXAMPLE III—(Dyeing with Vat Violet R. R.)

The procedure was the same as given under the Example I except that 0.5 g. of vat violet R. R. was used instead of Vat Brown-2 G. A brilliant violet coloured textile material was obtained with the same advantage as indicated for the other two dyes.

#### **EXAMPLE IV**

2 drops of Turkey red oil was added to 0.1 g. of Magenta in a beaker and a paste was made. 1.5 g. of DAS was added to 2 g. of NaOH in 100 c.c. of water. It was heated to 80°C and 0.1 g. of sodium hydrosulphite was added. To this solution, the paste of the dye was added and vatting was continued till the leuco colour was obtained. Then 10 g. of yarn was immersed in this vat for 15 minutes. Then the yarn was removed and wrinkled.

A brilliant magenta coloured material was obtained.

# EXAMPLE V

0'I kg. of dye consisting of Magenta and Blue was added to a hot solution 1 kg. of dialdehyde starch in 2 kg. of caustic soda in water at 90°C. Then this solution was added to a stock vat which was previously dyed using zinc and sodium sulphite. When the leuco colour was obtained after 25 minutes, 10 kg. of scoured yarn was immersed for about 20 minutes. Then removed, wrinkled and air oxidised. A brilliant bluish violet shade was obtained.

#### We Claim

- A process for the reduction of a vat dye by the use of a dye bath consisting of a reducing agent in an alkaline medium characterised in that dialdehyde starch is used as the reducing agent to prepare the dye bath.
- A process as claimed in claim (1) wherein an alkaline solution of dialdehyde starch is used to prepare the dye bath.
- A process as claimed in claims (1) or (2) wherein the reducing agent as needed is generated from dialdehyde starch by reacting with alkali.
- A process as claimed in any of the preceding claims wherein sodium hydrosulphite is mixed with dialdehyde starch.
- A process as claimed in any of the preceding claims, wherein the vat dye is reduced to a leuco compound by using the alkaline solution of dialdehyde starch and textile material is impregnated with the leucodye in the bath,
- A process as claimed in claim (5) wherein afterwards, the textile material is removed from the vats, washed with water and wrinkled and kept in air for oxidation and drying.
- 7. A process as claimed in any of the preceding claims which broadly consists first in the reduction of the vat dye by the use of an alkaline solution of dialdehyde starch to prepare the dye bath, then the textile materials are impregnated and later exposed for the air oxidation.
- 8. A process for the reduction of a vat dye substantially as hereinbefore described.

Dated this 5th day of October, 1970

(Sd. PATENTS OFFICER).

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