Ecofriendly processing of sulphur and vat dyes—An overview

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Various developments that have taken place in the ecofriendly dyeing of sulphur and vat dyes have been briefly discussed. A good degree of progress in this direction has been made by the successful use of products like hydro insulin sulphur dyeing and zinc sulphoxylate formaldehyde in vat dyeing. However, the efforts to improve the dyeing technology of sulphur and vat dyes on cotton are still required.

Keywords: Cotton, Dyeing, Ecofriendly processing, Sulphur dye, Vat dye

1 Introduction
Sulphur and vat dyes are traditionally used to dye cellulosic fibres in varying hues as the sulphur dyes are economical and the vat dyes show excellent colour fastness. Both the dyes are similar in application. The insoluble dyes are applied in the reduced soluble form and are then converted back to their original pigment form by oxidation. It is the reducing agents used in the application of these dyes which create major effluent problems. Hence, the need to develop and use ecofriendly reducing agents is becoming very important. The various developments that have taken place in the ecofriendly dyeing of sulphur and vat dyes have been discussed in this paper.

2 Sulphur Dyes
Sulphur dyes were first manufactured in 1873 by heating organic cellulose containing materials, such as wood, saw dust, cotton waste, waste paper, etc. with alkali sulphide and polysulphide. Earlier, the sulphur dyes were prepared by the thionation of various organic materials with either sulphur or sodium polysulphide and then applied on cotton from sodium sulphide solution.

2.1 Mechanism of Dye Application
Sulphur dyes essentially have disulphide linkage (R-S-S-R) in its structure where R is the chromophore which is responsible for imparting colour. The two chromophores linked together make the sulphur dye molecules generally large. As the sulphur dye lacks solubilizing groups, it is first converted into sodium salt of soluble leuco form by the reaction with alkaline reducing agent such as sodium sulphide and sodium carbonate. The reaction may be represented as follows:

\[
RSSR+2H+Na_2CO_3\rightarrow RSNa+NaSR+H_2CO_3
\]

This reaction consists of a step in which the disulphide link splits to form two leuco anions, each accompanied by a sodium cation (Dye-SNa"). Such ions, known as mercaptides or thiolates, are water soluble. The reduced form of sulphur dye has the characteristic similar to that of direct dye and promotes dye exhaustion from the solution into the material by the addition of electrolytes like common salt or Glauber's salt.

After the complete exhaustion of dyes into the material, the leuco anions can be restored to its original pigmentary form by either air oxidation or chemical oxidation. Potassium dichromate plus acetic acid, hydrogen peroxide, sodium perborate, sodium peroxide, etc. are the commonly used oxidizing agents for this purpose. Every method of oxidation has its own advantages and disadvantages. Sodium dichromate and acetic acid oxidation gives the highest degree of overall wash fastness but results in increased harshness and poor absorbency. The use of sodium perborate or hydrogen peroxide gives brighter shades but generally results in poorer wash fastness. Perborate oxidized yarn or fabric gives a much softer hand and possesses greater absorbency. Oxidation using ammonium persulphate and acetic acid produces yarn with a very high degree of softness and...
absorbency, and good fastness properties\(^1,2\). The overall mechanism can be represented as follows:

\[
\text{Dye} \xrightarrow{\text{Alkaline Reducing}} \text{Acid Oxidation} \xrightarrow{\text{Acid}} \text{Dye+Na}^+ \xrightarrow{\text{Alkaline}} \text{Dye+NaOH}
\]

The problems associated with the application of sulphur dyes include the precipitation of dye, particularly when calcium, magnesium and iron salts are present, tendering of the goods dyed on prolonged storage due to liberation of sulphuric acid; and the bronzing of the shades. Of late, the most alarming problem, which has very seriously become a matter of concern on grounds of environmental consciousness, is the huge effluent load generated out of sulphur dyeing discharges\(^3\).

Sulphur dyes are available in a variety of forms, which include powders, dispersed powders, dispersed pastes, pre-reduced liquids and the liquid and powder forms of solubilised sulphur dyes. The liquid form contains partially or completely reduced dye with dispersing / stabilising agent. Thiosulphuric acid derivatives have no affinity for the fibre but are activated by the addition of sodium sulphide or sodium sulphohydrate. In contrast to the traditional dyes, the Diresul EV dyes from Clariant do not contain alkali, solvent or free sulphides, thereby eliminating unpleasant odours and evolution of hydrogen sulphide fumes due to accidental acidification of the dyestuff\(^4\).

The principle of sulphur dyeing involves (i) selection of a dye which is essentially insoluble in water, (ii) reduction of dye with an alkaline reducing agent to its leuco compound so that it develops substantivity towards the fibre, (iii) re-oxidation of the dye in situ in the fibre to the original insoluble compound after the dye exhaustion. The reducing agents required for converting the insoluble dye to sodium salt of the soluble leuco form are mainly sulphides and non-sulphides.

2.1.1 Sulphide Reducing Agents

Some of the reducing agents of sulphide type are sodium sulphide (Na\(_2\)S), sodium hydrosulphide (Na\(_2\)HS) and sodium polysulphide (Na\(_2\)S\(_x\)) where \(x\) varies from 1 to 6. The polysulphide variety is available as an aqueous solution while the sulphides and hydrosulphides are available in both solid and liquid form. In water, sodium sulphide undergoes hydrolysis as follows:

\[
\text{Na}_2\text{S} + \text{H}_2\text{O} \rightleftharpoons \text{NaHS} + \text{NaOH}
\]

When sodium sulphide and sodium tetrasulphide are present under identical concentrations, they have pH of 12.2 and 11.6 respectively. The polysulphides act not only as pH buffer but also as antioxidants.

Sodium hydrosulphite has little importance in the dyeing with sulphur dyes because the dyes of red, brown, green and olive types break down in presence of hydrosulphite and has the inherent drawback of poor colour yields and reproducibility. This can be overcome by exhaustion dyeing method using C.I. solubilized sulphur blacks. However, the use of solubilized sulphur dyes and hydrosulphite is more expensive compared to ready-to-use leuco sulphur dyes which already contain inbuilt sodium sulphide as the reducing agent and, is more compliant with continuous dyeing process. The simple water insoluble sulphur dyes can be vatted at about 70°C with sodium hydrosulphite and caustic soda / soda ash. The disadvantages of sulphide containing reducing agents are the discharge of thiosulphate formed by atmospheric oxidation of sulphides. This process is catalysed by large quantities of sulphur dye present in the effluent.

Rectifying methods include combination and neutralization in aerated tanks or reactors, precipitation in the form of iron sulphide, etc. These methods have already been followed by many users of sulphur dyes for a long time. Future development in sulphur dyes is likely to be with the aim of replacing sulphide type reducing agents in application. In this respect, the cost of the alternatives has to be balanced against the relatively simple oxidative method used to treat sulphide containing wastes\(^1,5\).

2.1.2 Non-Sulphide Reducing Agents

2.1.2.1 Glucose

Glucose has long been known as a reducing agent for sulphur dyes. Dye yield being highly dependent on temperature, glucose gives unsatisfactory results, especially in open dyeing machines like jiggers and winches. At temperatures above 90°C, glucose acts as a very good reducing agent. The subsidiary factors, which contribute to its function, are strongly alkaline conditions and polysulphide addition. There is a striking similarity in the results of exhaust dyeing with solubilized sulphur dyes in presence of glucose and with sodium sulphide at temperatures above
90°C. In continuous dyeing, promising results have been obtained with all solubilized sulphur dyes.

Water insoluble sulphur dyes can also be applied using glucose, caustic soda and polysulphides, only after their dissolution by boiling. Another product, which is being used for number of years by few companies, is Hydrol (a byproduct of sugar industry and molasses based material) which also offers sulphur dye reducing capacity. Since it is not based on sulphides, the process is ecofriendly.

2.1.2.2 Mercaptoethanol

β-mercaptoethanol, marketed by BASF and sold under the trade name Molleskal SF, can be used as a substitute for sodium sulphide. Solubilized sulphur dyes can be applied using mercaptoethanol and caustic soda using exhaust methods as well as one-bath pad-steam process. Advantages of the use of mercaptoethanol include sulphide-free effluent discharge and absence of obnoxious smell emission. Disadvantages include its restricted use excluding the ready-to-use liquid sulphur dyes and high expenses in overall dyeing.

2.1.2.3 Alkylation of Sulphur Dyeings

When the reduced form of a sulphur dye is treated with an alkylating agent, the thiolate ion loses its electron and becomes oxidized. As a result, conventional oxidation can be avoided. This results in colour stabilization of dyeings with excellent wet fastness properties resulting from the alkylation of the thiol (-SH) groups to -SR groups. In addition to alkylation, it is often needed to oxidize the chromophoric part of the reduced sulphur dyes to develop its true colour. But the alkylated dyeings result in poor light fastness.

Sulphur dyes containing quinoneimine groups show marked colour change on reduction. These agents are employed during the dyeing process and not at the time of oxidation. Final shade results from the chemical bond formation by alkylation followed by crosslinkage. The duration of reaction may vary from 5 to 10 s at 100-105°C (refs 7,8).

2.1.3 Oxidation of Sulphur Dyes

2.1.3.1 Dichromate

The most preferred oxidizing agent for sulphur dyes is the dichromate. Advantages include complete oxidation of all reduced sulphur dyes by chromium (IV) compounds, good colour yields, good fastness properties and the repeatability of the process. Disadvantages of such an application is that the fabric becomes harsher in feel and less hydrophilic, as a result of small amount of chromium compound getting fixed on the fibre, particularly with sulphur blacks. This restricts the use of dichromate in yarn dyeing. The general procedure involves application of 1-2 g/litre potassium chromate and 4-6 ml/litre acetic acid (60%) in the oxidizing bath with 0.3-0.6% dichromate and 2-3% acetic acid (owf) as the feeding addition at 70-80°C.

2.1.3.2 Iodates

Sodium or potassium iodate is used as a substitute for dichromate. The general procedure involves application of 1-5 g/litre of iodate in the oxidizing bath and 0.3–0.6% (owf) for the pending addition. The pH must be adjusted to 4-5 with acetic acid and the temperature of the bath should be maintained at 60°C.

Advantages include excellent colour and fastness reproducibility, softer handle and use of iodate as a reasonable alternative to dichromate. Disadvantages include high cost of the chemicals and iodine precipitation in washers when used in conjunction with formic acid instead of acetic acid.

2.1.3.3 Chlorite

The most effective chlorite oxidizing agent is Oxidwit SK, a product of Cassella AG and marketed by Hoechst. It is to be applied only in alkaline media and is a relatively weak oxidizing agent. The oxidation is carried out in presence of 2 g/litre soda ash (pH 10), 5-10 g/litre Oxidwit SK in the oxidizing bath and 2-3% (owf) for replenishing is used at 90-95°C and speed of about 50m/min in continuous oxidation boxes.

Advantages include very good and reproducible results, material cleaning due to the presence of dispersing agents, and removal of dispersed dyes and their breakdown products from polyester in case of polyester/cellulosic blend materials. The main disadvantage is the relatively slow reaction rate as a result of which chlorite is not suitable as a general substitute for dichromate.

2.1.3.4 Hydrogen Peroxide

Hydrogen peroxide in an alkaline medium is a useful alternative as it exercises a very high oxidizing action. So that in some cases, the sulphur dye is oxidized beyond the disulphide stage. This results in considerably high wet fastness of the dyed material. It also does not result in undesirable harshening of the yarn like that in case of dichromate.
The usual procedure involves the use of 2 g/litre hydrogen peroxide (35%) in the oxidation bath at a pH of 10 and temperature of 40°C. The feeding liquor should contain 1% hydrogen peroxide (35%) on weight of the running fabric.

2.1.4 Reduction in Chemicals

Oxygen in the air reacts with reducing agent present in the sulphur dye bath and destabilises the leuco form of the dyestuff which often leads to bronzy and unlevel dyeings. For this reason, excess of reducing agents is generally required to avoid this problem.

The breakthrough approach from Clariant entails the novel exhaust dyeing process (nitro process) which uses nitrogen, one of the nature’s most abundant and inert elements, for minimising the use of reducing agents required for sulphur dyeing. The process involves purging of inert nitrogen into the dyeing vessel, thus bringing about a substantial reduction in the amount of reducing agent. This results in cost savings as the purging process costs less.

The new application process makes use of Diresul EV dyes, reducing the dye cycle time to <4 h for 100% cotton fabrics. As compared to traditional sulphur dyeing, the new process saves >1 h of the dyeing time. The use of Diresul EV dyes offers the following benefits:

- Contain no alkali or reducing agent, thus neither corrosive nor toxic.
- Free of sulphides, no unpleasant odour during dyeing.
- Low pollution load in effluent streams.
- Suitable for using sugar as reducing agent.
- No soiling of the dye vessel.
- Good combining properties.
- Specially suitable for the nitro process.
- Good overall fastness properties.

2.2 Sulphur Dye Effluent

Dye effluent from traditional dyeing system contains unfixed dyestuff, sulphides and inorganic salts. Discharge of inorganic salts is normally not permissible because of the danger to life from liberated hydrogen sulphides, high pH and unpleasant odours. Concentration of dye depends upon the dyeing method, applied depth and the particular dye used. The legislation has imposed strong restrictions on the amount of chromium compounds in the effluents. This has limited the use of dichromate, which has been very popular with the dyers.

The reduction in chemical usage can lower BOD and COD loads, leading to almost complete elimination of sulphides in effluent streams. In addition to savings in chemical costs and less environmental pollution, the chemical reduction results in fast chemical and dye addition in short heating-up time. Techniques to reduce the sulphur dyeing effluent load involve oxidation or precipitation. Oxidation enables the colour in the effluent to be precipitated, which can be removed by separation or filtration. This is because the dyestuff is converted to an insoluble oxidized form.

During treatment, the sulphide containing streams must not be allowed to come into contact with acidic effluents because hydrogen sulphide will be evolved. However, mixing with bleaching liquors is beneficial since both sodium hypochlorite and hydrogen peroxide are efficient in the oxidation of sulphides.

2.2.1 Oxidation Treatment

Air has been the traditional oxidizing agent used to treat sulphide and is very cost effective and widely used. The oxygen in air converts sulphide to thiosulphate, the rate of oxidation being always linear. The rate determining step is the dissolution of oxygen in water. The factors which affect the process are fine bubble generation, intimate mixing of air and effluent, operation depth and addition of agents such as manganese salts, anthraquinone disulphonic acids and hydroquinones. During the reaction, the pH generally decreases from 12 to 10. Aeration should be undertaken below pH 9 to avoid the risk of hydrogen sulphide evolution.

2.2.2 Bacterial Treatment

Bacterial treatment plants are in use in both dye manufacturing and dyeing industries. The treatment plants require an uniform effluent flow of fairly constant composition. The treatment removes sulphides, thiosulphates and organics, but it does not produce a solid waste that has to be disposed off to landfill sites.

2.2.3 Chemical Oxidation

Hydrogen peroxide or sodium hypochlorite in small quantities is used for this purpose. Sodium hypochlorite can sometimes be obtained as a byproduct from the chlorinating colour removal process. The unreacted chlorine is absorbed in sodium hydroxide solution to give hypochlorite.
2.2.4 Precipitation

Sulphides can be precipitated, using lime, alum and ferrous sulphate / aluminium sulphate. The technique involves some form of settlement / filtration of the precipitate after treatment and the safe disposal of the residue which might contain sulphides. Organic matter and colours are usually removed by this treatment5,12.

3 Vat Dyes

Vat dyes, in general, are water insoluble pigments but can be dissolved to give solutions containing individual molecules or molecular ions. The dyes can be transported to the surface of the fibre, adsorbed on and diffused into suitable fibres. Vat pigments can be dissolved by alkaline reduction into their leuco form followed by regeneration of the original insoluble pigments by the process of oxidation after diffusion of the soluble ions into the fibre.

In the insoluble pigmentary form, all the vat dyes have a common chemical feature consisting of one or more pairs of carbon atoms, doubled to an oxygen atom, i.e. all the vat dyes contain keto groups. In general, vat dyes can be classified into following three different classes

Indigoid

![Indigoid structure](image)

Anthraquinonid

![Anthraquinonid structure](image)

Conjugated dicarbonyl

![Conjugated dicarbonyl structure](image)

The following chemical reaction depicts the general transformation, which takes place at the time of dyeing13:

2Na₂S₂O₄ + O₂ + 4 NaOH → 4Na₂SO₃ + 2H₂O

This is another reason why sodium hydroxide has to be added in excess to avoid the dye bath to become acidic due to the precipitation of leuco acid compound15.

The other reducing systems for vat dyes include thiourea dioxide which is relatively expensive and can over reduce some of the dyes. Several hydroxy alkyl sulphonates are used selectively for printing and high temperature applications. The stability of the alkaline solutions of reducing agent decreases with the increase in temperature, surface area exposed to the air and agitation of the bath, and decrease in concentration of alkali13.

To avoid oxidation of the leuco vat, a large excess of sodium hydroxysulphite is used in conventional vat dyeing. The excess amount is at least five times the theoretical amount, which might lead to problems like over reduction, hydrolysis and crystallization of vat dyes, especially at higher temperatures16.

3.1 Reducing Agents / Methods

3.1.1 Sodium Hydroxysulphite

The most commonly employed reducing agent in case of vat dyeing is sodium hydroxysulphite (Na₂S₂O₄), commonly known as hydros. This compound is not stable in strong alkaline conditions in the absence of air. Alkaline solution of hydros has a certain degree of reduction potential and thus, can reduce all commercial vat dyes to their water-soluble forms, economically and quickly, without any chance of over reduction. As the leuco sodium salt of the vat dye is a salt of strong alkali and weak acid, the sodium salt has the natural tendency to exist in enol form. To ensure complete conversion, sodium hydroxide is added prior to hydros addition14. Since hydros reacts with aqueous as well as atmospheric oxygen, it leads to the formation of a number of acidic compounds which have to be neutralized by sodium hydroxide.

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3.1.1.1 Oxidation of Hydroxysulphite

The factors which affect hydroxysulphite oxidation during dyeing process are temperature, concentration of alkali and rate of oxidation in chemical padding. The unwanted oxidative decomposition of hydros can be checked by pre-exposure of the dye bath to oxygen, as is found in many modern dyeing machines, and by carrying out the reduction process in an enclosed environment14,15.
3.1.1.2 Influence of Polyacrylamide

High molecular weight polymeric additives can be added to promote increased dye uptake or adhesion in discontinuous padding dyeing and continuous pad-steam dyeing of cotton fabric with indigoid and anthraquinonoid vat dyes. The inclusion of the polymeric additives in the solution of reducing agent decreases dyeing imperfections, which otherwise would have resulted from premature oxidation of the reduced water soluble vat dye on the fabric. When the high molecular weight polymeric additives are included in the chemical pad bath, an increase in colour yield results.

3.1.1.3 Control of Sodium Hydrosulphite

The application of vat dyes requires not only a large number of processes but also greater skill and control than are necessary for other dyes. Thus, apart from the cost of the dye itself, the process of dyeing is more expensive. The soluble alkaline leuco form is normally used, but it is often unstable and only a careful control and use of the correct amount of reducing agent can result in successful dyeings.

Addition of too little of sodium hydrosulphite results in oxidation and the dye comes out of the solution. This results in poor colour value, incorrect heavy colour selvedges in jigger dyeing and poor fastness to washing. Too much of hydros addition can result in destruction of the dye chromophore, leading to loss of colour yield.

The control of hydrosulphite concentration is not so simple and can be taken care of by time to time checking of the appearance of the dye bath and by the use of Caledon yellow paper. Complete reduction of the dye is indicated by the change of the litmus paper from yellow to blue.

3.1.2 Stable Reducing Agents

Of late, economical attempts to stabilise hydrosulphite and maintain its high reactivity necessary for pad-steam work have failed. Formaldehyde has been found to decrease the reactivity of hydros to a great extent. However, the sulphoxylate product resulting from the reaction of zinc with a molecule of bisulphite (obtained by reaction between formaldehyde and sodium hydrosulphite) has been found to be much more stable than hydros, particularly at ordinary temperatures.

\[
\text{Na}_2\text{S}_2\text{O}_4 + 2\text{HCHO} + \text{H}_2\text{O} \rightarrow \text{OHCH}_2\text{SO}_2\text{Na} + \text{OHCH}_2\text{SO}_3\text{Na}
\]

Zinc Sulphoxylate Formaldehyde

The addition of sodium borohydride (NaBH₄) as a stabiliser, apart from economic point of view, has shown disputable results. Borohydrides have the ability to reduce sodium sulphite to hydrosulphite. In other words, these can replenish hydrosulphite solutions. The reaction, however, is most active only at a pH value close to 7. For a pad-steam process at pH values close to 13, regeneration of hydrosulphite, therefore, becomes questionable.

Glucose also acts as reducing agent, but it is highly dependent on temperature. The self-potential of glucose is very stable even at 350°C and can reduce the total potential of the vat. The glucose addition is a very effective means of preventing over-reduction of sensitive dyestuffs in continuous pad-steam process.

3.1.3 Reduction by Indirect Electrolysis

In contrast to direct electrolysis where the dye itself is reduced at the surface of the cathode, in indirect electrolysis the reducing power of the cathode is transferred to the solution by a soluble reversible redox system (mediator). This reversible redox system is continuously regenerated at the cathode so that the renewal of the reducing agent is achieved. This technique offers the possibility of full dye bath recycling, including the reuse of the reducing agents. The chief characteristics of a mediator are as follows:

- Sufficient negative reduction potential.
- Stable reducing conditions in the dye bath.
- Reproducible dyeing.
- Low concentrations of chemicals employed.
- Simple waste water treatment.
- Recycling of chemicals and waste water.

A great advantage of reduction with iron (II) amine complexes is the high rate of dye reduction which is much faster than that with sodium hydrosulphite. The iron (II) amine complexes thus allow a better control and measurement of reduction conditions in the dyeing liquor.

3.2 Dye Recycling

Reuse of the mediator system with vat dyes demands removal of the residual dye from the dye
liquor. This is possible because of the insolubility of the oxidized form of dyes in aqueous solution and their ability to form suspensions which can be removed by a filtration process from the oxidized dye liquor.

Iron amine complexes, e.g. iron (II/III) triethanolamine complexes, possess high negative redox potential in alkaline solutions and can, therefore, be used as suitable reducing agents for the indirect electrochemical reduction of vat dyes. Good reproducibility can be achieved and the dyeing levels are similar to those of conventional vatting techniques.

3.2.1 Closed Circuit Technique

The dye bath is circulated through an electrolyte cell at a rate that must follow the threshold conversion rate in the cell to be exceeded. The dye bath potential is governed by Fe (II)/Fe (III) ratio and is an important control variable in the dyeing process. The process demands complete filtration of the circulating liquor to avoid contamination of the cell by impurities released from the goods.

With this technique, the mediator and vat dyes like indigo can be recovered from wash water. The wash water is passed through ultrafiltration unit to remove the insoluble dye and the concentration of the mediator in the filtrate is increased by nanofiltration to a final value of 0.6 mol/litre of iron (III) complex. The iron (II) complex concentrate is metered on a regulated basis to maintain the Fe (II)/Fe (III) ratio at a constant level. This method of reduction offers the advantage of allowing almost any desired amount of reduction equivalents to be admitted into the dye bath of constant concentration ratios.

3.2.2 Mediator Concentrate Technique

The contents of the dye bath are neither circulated through the electrolytic cell nor does a dye solution flow through it. In this process, metering is similar to that in the closed circuit technique but the composition of the dye bath is connected to the consumption of the reducing agents in the installation, as in case of conventional reducing agents. Here, an upper limit to the volume of reducing agent that can be metered is imposed. If this limit is exceeded, the dye bath will overflow unless additional technical measures are adopted to prevent it.

A comparison of the two concepts for electrolytic reduction reveals that the closed circuit technique offers the greater advantages in the dyeing of vat dyes like indigo. This is because rapid process control is possible by measurement of the potential, and the control of the cell current can be exploited more effectively if the dye liquor is circulated through the electrolytic cell. Another advantage is that the composition of the dye bath is independent of dye consumption so that the failing risk is avoided.

4 Conclusion

Efforts are being made to reduce the use of environment-objectionable reducing agents in case of sulphur and vat dyes. Looking at the successful use of products like hydrol in sulphur dyeing and zinc sulphoxylate formaldehyde in vat dyeing, one could say that good degree of progress in this direction has been made. However, there is still room for improving the dyeing technology of sulphur and vat dyes on cotton if one is forced to use these dyes for their obvious advantages in cost and/or performance.

References