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## EFFECT OF OXIDIZING AGENTS ON THE FASTNESS PROPERTIES OF VAT DYES ON 100% COTTON FABRIC DYEING IN EXHAUST METHOD

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### ABSTRACT

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Vat dye is the popular choice for the coloration of cotton fabric when high color fastness is required. They are generally insoluble in water and are converted into water soluble form (leuco dyes) by reducing with a reducing agent. The reduced dyestuffs penetrate into the fibre, are then oxidized by air or different oxidizing agents & thus the dyestuffs get back to the original insoluble form which remains fixed in the fabric. The aim of the work was to compare among the oxidizing agents including air, the most commonly used oxidizing agent and other strong oxidizing agents like Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), Sodium perborate (NaBO<sub>3</sub>) and Potassium di-chromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>). Two hues Blue and Yellow, each of 4% shade owf were dyed on both 100% cotton knit and woven fabric in exhaust method. It was observed that oxidation at air and with H<sub>2</sub>O<sub>2</sub> show the best fastness performance among the four oxidizing agents. Considering the cost involved, air oxidation is the cheapest and also comparable in performance with other oxidizing agents.

**Key words:** vat dyes, reduction, leuco vat dyes, oxidation, fastness, cost, cotton

### INTRODUCTION

An estimate made in 1979 showed vat dyes to have a 15% share in the dyeing of cellulosic fibres, with indigo contributing a further 2% (Choudhury 2006). 'Indigo' is one of the oldest naturally occurring vat dyes and has been known to Indian people for more than 5000 years (Clark 2011). Vat dyes, so called because Indigo, the first member belonging to this class of dyes was dyed on textile materials in wooden vats in ancient days, are water – insoluble colored compounds. As such they can't be directly applied on cotton or other fibres as direct dyes are dyed. They have to be converted into a water – soluble form, having sufficient affinity for fibres. During the dyeing process, it is this soluble form of the dye that is applied on cotton, followed by reconversion of the soluble form into the original insoluble form. As a result, the insoluble dye is trapped in the fibre substance and can't come out during soaping or any other wet treatments, thereby ensuring excellent washing fastness (Shenai 1997). Vat dyes being insoluble in water and nonionic, are converted to leuco compounds on reduction followed by solubilisation with alkali in which state these show substantivity towards cellulose. After dyeing, parent dye structure is recovered by oxidising it within fibre, when the dye molecule get trapped in situ and establish linkage with fibre through H-bonds and van der Waals forces (Chakraborty 2010).

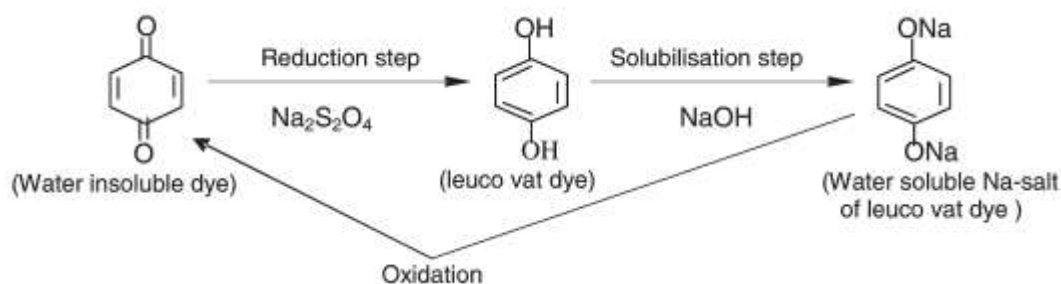


Fig. 1. Basic principle of dyeing cotton with vat dye (Chakraborty 2010)

In exhaust dyeing with vat dyes, the whole procedure takes place in the following four steps: (1) Solubilisation of the vat dye, i.e. conversion of the dye to sodium salt of leuco acid through the vatting process, (2) Application of the soluble dye to the textile material, i.e. dyeing, (3) Oxidation of the dye back to the parent insoluble form, (4) After-treatment or soaping (Clark 2011).

Vat dyes, available in highly fine powder form, added to water to form a dispersion of very fine particles. This is then added to a dye-bath for reduction and brought into solution, thus eliminating the necessity for vatting as a separate step. In the second step, the sodium salt of leuco vat dye is absorbed on the surface of fibre, from where it diffuses into the interior of the fibre – is the actual dyeing process. After the completion of the exhaustion process the dyed material is rinsed to remove superficially held dye particles and any residual reducing agent and alkali. The leuco dye is then reconverted into its original form by oxidation. This is carried out with hydrogen peroxide or sodium perborate, or more recently sodium metanitrobenzene sulphonate. Other methods of oxidation are also available such as treatment with hypochlorite, potassium dichromate and acetic acid, or treatment with sodium chlorite, but these are not recommended due to their environmental effects (Clark 2011).

Generally large projects do air oxidation to make soluble dyes into insoluble form and to generate final color on goods. As air is available in environment, no cost is involved to get & using it. The present work highlights a comparative study among air oxidation with other three oxidizing agents namely Hydrogen peroxide ( $H_2O_2$ ), Sodium perborate ( $NaBO_3$ ) and Potassium di-chromate ( $K_2Cr_2O_7$ ) in terms of fastness performance.

## MATERIALS AND METHODS

### Materials

#### *Fabric Sample*

The specifications of both the woven and knit samples are given below:

Table 1. Specification of the fabric sample

Fabric Type	Specifications				
	Ends/inch EPI	Picks/inch PPI	Areal Density (GSM)	Warp Count (Ne)	Weft Count (Ne)
100% Cotton Woven (1/1 plain weave)	120	80	130	40	30
100% Cotton Knit (Plain Single Jersey)	Areal Density (GSM) = 130, Yarn Count (Ne) = 36'S				

#### *Dyes and Chemicals*

The dyes and chemicals used in different stages of processing are listed below with their functions.

Table 2. List of dyes and chemicals used with their functions

Dyes and Chemicals Used	Functions
Dye 1: NOVASOL BLUE RS md, Swiss Color, Switzerland	Used for coloration of blue shade
Dye 2: YELLOW 5G md, Swiss Color, Switzerland	Used for coloration of yellow shades
Kieralon OL	Used as wetting agent
EDTA disodium salt (99%)	Used as sequestering agent
Common Salt (NaCl)	Used as electrolyte of the dye bath
Caustic Soda (NaOH) 34 Be'	Used to saponify oil & wax in scouring
Sodium Silicate ( $Na_2SiO_3$ )	Used as stabilizer during bleaching
Hydrosol (Sodium hydrosulphite)	Used as reducing agent
Sodium Carbonate ( $Na_2CO_3$ )	Used to control pH
Hydrogen peroxide ( $H_2O_2$ ) (35%)	Used as bleaching agent and for oxidation
Na-perborate ( $NaBO_3$ )	Used for oxidation
Potassium dichromate ( $K_2Cr_2O_7$ )	Used for oxidation
Acetic acid	Used for neutralization
ECE reference detergent (without optical brightener)	Used for soaping and fastness check

### Methods

Combined scouring bleaching was performed for the preparation of fabric to be dyed with vat dyes. Drop test was carried out to ensure the pretreatment is perfect or not. After the preparation the fabric was dyed with vat dyes. The vat dyed fabric was then exposed for oxidation with various agencies. Air oxidation was performed by hanging it on open air till drying. Oxidation with  $H_2O_2$ ,  $NaBO_3$  and  $K_2Cr_2O_7$  were also carried out in separate bath. After that, soaping and neutralization (with acetic acid) of the dyed samples were performed according recommendation.

Table 3. Recipe for fabric preparation, dyeing and oxidation

Recipe for Fabric Preparation	Recipe for Dyeing with Vat dyes	Recipe for Oxidation
$H_2O_2$ (35%) = 5% owf	NOVASOL BLUE RS (for blue shade)= 4% owf	$H_2O_2$ = 2 g/l
NaOH (34 Be') = 4% owf	YELLOW 5G (for yellow shade)= 4% owf	Or $NaBO_3$ = 2 g/l
$Na_2CO_3$ = 3% owf	NaOH (34 Be') = 7% owf	Or $K_2Cr_2O_7$ = 2 g/l
Kieralon OL = 1% owf	Hydrosol = 6% owf	M:L = 1:30
$Na_2SiO_3$ (Stabilizer) = 2% owf	Salt (NaCl) = 10% owf	Temperature = 60°C
pH = 10.5 – 11	Kieralon OL = 1 g/l	Time = 20 min
M:L = 1:30 (for open bath)	EDTA disodium salt (99%) = 1 g/l	
Temperature = 100°C	pH = 12	
Time = 1hr	M:L = 1:30	
	Temperature = 70°C	
	Time = 45 min	

## Testing

The fastness testing method of the dyed fabric samples and the instruments used are given in Table 4.

Table 4. Physical properties tested of the samples

No	Property	Standards	Instrument used
1	Color fastness to wash	ISO 105 C03	Gyrowash
2	Color fastness to water	ISO 105 E01	Perspirometer
4	Color fastness to rubbing	ISO 105 X12	Crockmeter
5	Color fastness to light	ISO 105 B02	Microsol

## RESULTS AND DISCUSSION

### Color fastness to wash

The change in color of the specimen and the staining of the adjacent fabric are assessed by color changing & color staining Grey Scales. The data are shown in the Table 5.

Table 5. Color fastness to wash data

Shade	Fabric Type	Oxidation with	Color Fastness to Wash							
			Color Changing	Color Staining						Average Staining
				Acetate	Cotton	Nylon	Polyester	Acrylic	Wool	
4% Blue	Knit	Air	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Hydrogen peroxide	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Sodium perborate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Potassium di-chromate	3	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	Woven	Air	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Hydrogen peroxide	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Sodium Perborate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Potassium di-chromate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
4% Yellow	Knit	Air	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Hydrogen peroxide	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Sodium perborate	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Potassium di-chromate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	Woven	Air	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Hydrogen peroxide	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Sodium perborate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Potassium di-chromate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5

It is seen that color fastness to wash in color changing is found 3 to 4-5 i.e. average to very good rating. And for staining in washing we have found 4-5 i.e. very good rating. Air and hydrogen peroxide show better result among the four oxidizing agents. Sodium perborate and potassium dichromate shows similar results and in case of staining all the oxidizing agents exhibit same rating.

### Color fastness to water

The change in color of the specimen and the staining of the adjacent fabric are assessed by color changing & color staining Grey Scales. The data are shown in Table 6.

Table 6. Color fastness to water data

Shade	Fabric Type	Oxidation with	Color Fastness to Water								
			Color Changing	Color Staining						Average Staining	
				Acetate	Cotton	Nylon	Polyester	Acrylic	Wool		
4% Blue	Knit	Air	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Hydrogen peroxide	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Sodium perborate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Potassium di-chromate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	Woven	Air	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Hydrogen peroxide	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Sodium perborate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Potassium di-chromate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
4% Yellow	Knit	Air	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Hydrogen peroxide	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Sodium perborate	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Potassium di-chromate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	Woven	Air	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Hydrogen peroxide	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Sodium perborate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
		Potassium di-chromate	3-4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5

It is seen that color fastness to water in color changing is found 3-4 to 4-5 for all samples i.e. average to very good rating. And for staining in wet condition we have found 4-5 i.e. very good rating. As like before, air and hydrogen peroxide shows the best results whereas sodium perborate and potassium dichromate shows similar results. For staining all the oxidizing agents exhibit the same rating.

### Color fastness to rubbing

The change in color of the specimen for dry rubbing and wet rubbing are shown in Table 7.

Table 7. Color fastness to rubbing

Shade	Oxidation with	Color fastness to Rubbing			
		Knit		Woven	
		Dry rubbing	Wet rubbing	Dry rubbing	Wet rubbing
4% Blue	Air	4-5	3	4-5	3
	Hydrogen peroxide	4-5	3-4	4-5	3-4
	Sodium perborate	4-5	2-3	4-5	2-3
	Potassium dichromate	4	2	4	2
4% Yellow	Air	4-5	3	4-5	3
	Hydrogen peroxide	4-5	3-4	4-5	3-4
	Sodium perborate	4-5	2-3	4-5	2-3
	Potassium dichromate	4	2	4	2

Vat dye is physically trapped into the fibre system. Therefore, It is weak in rubbing, specially in wet rubbing. Mechanical friction tends to draw out dyestuffs from fibre by staining. And if rubbing object is wet, it creates more friction with fabric. Also in wet condition, cotton rubbing cloth do swelling and it's surface becomes more rough than dry state as in dry state fabric is more flat that creates comparatively less friction than wet condition. It is seen that color fastness to dry rubbing is 4 to 4-5 i.e. very good. And color fastness to wet rubbing remains 2 to 3-4 i.e. poor to average. As like before, here, too, air and hydroegen peroxide show better result among the four.

**Color fastness to light**

Color fastness to light indicates the change of shade due to exposure under sunlight. The data for individual samples are shown in Table 8.

Table 8. Comparative study on Color Fastness to Light for different Oxidation process

Shade	Oxidation with	Color fastness to light (color changing)	
		Knit	Woven
4% Blue	Air	4	4
	Hydrogen peroxide	4	4
	Sodium perborate	4	4
	Potassium dichromate	4	4
4% Yellow	Air	4	4
	Hydrogen peroxide	4	4
	Sodium perborate	4	4
	Potassium dichromate	4	4

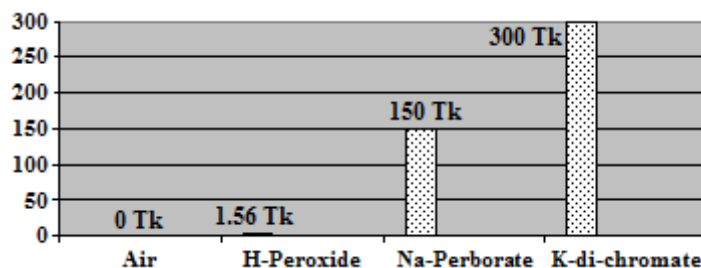
**Cost analysis:**

Cost according to consumption data are shown in Table 9.

Table 9. Cost involved for oxidation of processing per Kg fabric

Chemicals used in oxidation	Current Market Price (BDT) / per Kg	Cost of processing (Tk) of 1Kg fabric
Air	Free at environment	0
2 g/l Hydrogen peroxide	26	1.56
2g/l Sodium perborate	2500	150
2g/l Potassium dichromate	5000	300

The comparison can be presented more precisely in the following bar diagram:



It is seen that in air oxidation there is no cost involved where little bit more cost is involved in H<sub>2</sub>O<sub>2</sub> oxidation. NaBO<sub>3</sub> and K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> possess too much costs of processing.

From above analysis we can draw a decision from below point of view:

**In terms of color fastness**

All color fastness results show that fixation with air and H<sub>2</sub>O<sub>2</sub> give the best performance in case of both color changing & color staining. NaBO<sub>3</sub> shows average performance & K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> shows the worst performance for all tested color fastness.

**In terms of cost analysis:**

Air is fully free & available in environment. So air oxidation is the most economical way of oxidation. H<sub>2</sub>O<sub>2</sub> is comparatively cheap chemical but it includes additional cost of processing. NaBO<sub>3</sub> is high price chemical & K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> is very high price chemical that includes more cost.

**In terms of shade analysis:**

Air oxidation helps to generate good shade and shade generally doesn't go dull or pale tone. H<sub>2</sub>O<sub>2</sub> oxidation creates little bit blue tone generally and also we found little bit uneven shade by using it. K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> shows yellow tone on cotton. NaBO<sub>3</sub> shows medium to little bit dull tone.

**CONCLUSION**

In this comparative study different oxidation processes were justified in terms of cost, color fastness, color tone and production feasibility in exhaust vat dyeing on cotton fabric. Oxidation with air and hydrogen peroxide show best result among the four oxidizing agents in all aspects. Sodium perborate and potassium dichromate has relatively less efficiency than the other two, therefore, need fixing agent to upgrade the fastness rating; adding

extra costs of processing. However, when specific tone of shade is required, these can be used with special care. Oxidation with hydrogen peroxide shows very good performance if all process parameters are well controlled (i.e. time, temperature, pH etc.). If these parameters are not controlled properly or if one of them lacks control, there might have a chance of uneven shade generation. Also this chemical is health and environment hazardous since it creates skin irritation, allergic symptom, lung disease to human. Also reduces soil fertility and tissue damage to fish. So, proper care is must to deal with this agent successfully. Oxidation with air shows very good performance where there is only a risk of premature oxidation (air oxidation in dyeing). To control it, the fabric is always kept immersed under dye liquor. Otherwise overall processing is very smooth, no such mentionable risk. Obtained shade is also bright & even. Air is friendly to human & environment. Therefore, still it is the first choice of dyer for processing of vat dyes.

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