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Hydrolysis of Sugar for Dyeing of Cotton Fabric with Sulphur Black

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Abstract

The importance of sulfur dyeing of cellulosic fibers, particularly cotton, is realized economically throughout the dyeing industry. At the present time, dyeing with sulfur dyes requires the use of various auxiliaries, many of which have adverse effects on the environment. The most damaging of these is the reducing agent sodium sulphide, required to reduce the dye molecules to a water-soluble leuco form to enable adsorption and diffusion into the fiber. In this study, attempts have been made to replace the sodium sulfide used within the sulfur dyeing process with a variety of environmentally friendly reducing sugars. The redox potential of various hexose and pentose monosaccharide's and reducing disaccharides was recorded and compared. Subsequently, cotton was dyed with the world's most important sulfur dye, C. I. Sulfur Black 1, using the reducing sugars under alkaline conditions, and compared to dyeing secured by employing commercial sulfide reducing agents. It was observed that reducing sugars gave comparable, and in many cases superior, color strength and wash fastness results, with respect to the commercial sulfide-based reducing agents, which still account for the vast majority of sulfur dyeing processes and that pose significant environmental concern. Employment of reducing sugars in sulfur dyeing could provide a sustainable, nontoxic, biodegradable, cost-effective alternative to sodium polysulfide and sodium hydrogen sulfide.

Keywords: Hydrolysis; Dyeing; Sugar and sodium hydrogen sulphide

Introduction

Textile dyeing is the process of coloration which adds color to the textile materials by using different chemicals, dyestuffs and dyes by forming fiber-dye bond depending on the fabric and process used. The art of coloring textiles is very old. Its history can be traced for at least 4000 years starting in India and gradually spread throughout the world. All coloring materials used at the time were naturally occurring products such as the roots, stems, leaves, fruits of plants and dried extracts of certain insects and shellfish.

There are different kinds of dyes such as direct dyes, reactive dyes, vat dyes, disperse dyes, sulphur dyes etc. these all dyes uses different chemicals for their application. Vat dyes have excellent wash and light fastness and uses for the dyeing of cotton. But its cost is high when we compare with others. For black colors sulphur dye is possible.

In the present scenario of Ethiopia dyeing is mostly carried out on cotton fabrics. And as far as dyeing process of cotton fabrics with black color is concerned most of the products are carried out in vat dyeing. This leads for the study to concentrate on the replacement of vat with sulphur black.

Currently environmental issues are serious issues throughout the world. Every textile business sector to become internationally competent in the market the chemicals used for each specific operation must not hazardous to the others health and environment. Sulphur black uses sodium sulphide as a reducing agent. But it is toxic and hazardous for the environment. This study is concerned for the replacement of sodium sulphide with hydrolysed sugar commercially available.

Materials and Experimental Methods

Materials

Commercially available sugar, Dyestuff –sulphur black, bleached fabric,

Chemicals used: The following laboratory grade chemicals were used:- Hydrochloric Acid (35%), Sodium hydroxide, sodium sulphide, Sodium chloride, Sodium carbonate and Phenolphthalein indicator as Indicators.

Equipment's

Laundro meter, Stove, Burettes, Weighing balance, Different beakers, Spoon, Thermometer, pH meter and Mini dryer

Methodology

Hydrolysis of sugar: There are following process through which the Hydrolysis of sugar will take place:-

Dissolution of sugar: 50 gm of sugar sample was taken in a clean and previously weighed beaker. About 15 mL of water was added in it and the mixture was heated to boil, till complete dissolution of sugar takes place. After that solution was kept for cooling.

Acid concentration: Dilute solution of hydrochloric acid was used for hydrolysis of sugar. The required volume of acid and the rest portion of water were added to pre-dissolved sugar solution, so that the total addition will be equal to 40 mL, and the solid content of the solution will be 70%. Hydrolysis was carried out at different concentrations of acid. The amount of hydrochloric acid was taken as percentage on the basis of weight of sugar [1].

Laboratory grade hydrochloric acid is about 35% w/v, and for reaction its actual content was considered. For this purpose acid was diluted to get 1 N, 2 N and 5 N concentrations and standardized by titration against standard NaOH solution using phenolphthalein as indicator. The acid concentration in each prepared solution was as follows (Table 1):

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For the hydrolysis of sucrose the concentration of hydrochloric acid used was on the basis of the weight of sugar. E.g. 1 % hydrochloric acid means 1 g of pure HCl used for the hydrolysis of 100 g of sugar. This means 1 g of pure HCl equivalent to 27.39 mL of 1 N HCl, 13.7 mL of 2 N HCl and 5.48 mL of 5 N HCl.

Acid hydrolysis of sugar: Add 27.39 mL Hydrochloric acid with 1 Normality for 100 gm of dissolved sugar at room temperature. In this case add 13.695 mL of hydrochloric acid for 50 gm of dissolved sugar [2-4]. Hydrolysis of sugar with HCl was carried out at room temperature. The time required for glucose formation was checked at different time intervals. But in this case the minimum time was one day or 24 hours. And the maximum time was 40 days after adding of hydrochloric acid to the dissolved sugar (Table 2) [5-7].

Procedures of dyeing

Before dyeing is takes place the hydrolysed sugar must be neutralized its acidic media. This is done by adding of Na_2CO_3 and phenophitalin indicator to the solution of water and Hydrolysed sugar till it gives a pink color.

Solutions prepared by mixing X gram sulfur black dye, 2X gram Hydrolysed sugar and Y gram sodium hydro oxide in Q ml of water [8-11].

Reduction is carried out for 10 minutes at 80°C for the just prepared solution (Figures 1 and 2).

Dyeing: Started at about 40°C, heated to 80°C and hold for 1 h. Sodium chloride (45 gm/l) is added in two instalments (first half after 10 minute holding at 80°C, second half after another 10 minutes). The dyeing cycle is as follows:

Note: dyeing is carried out in beaker and it was stirred continuously manually.

Normality	1	2	5
g/L of HCl	36.5	73.0	182.5

Table 1: HCl acid concentration and its respective weight.

MLR 1:20, time 1 h. and temp 80°C	
Chemicals used	**Weight**
Sulphur black Dye stuff (8% o.w.f)	X gm
Sodium sulphide or (Hydrolyzed sugar)	X (2X)
sodium carbonate or (Sodium hydro oxide)	4 gm/lt
Sodium chloride	45 gm/lt

Table 2: Dyeing recipe for cotton fabric with sulphur black.

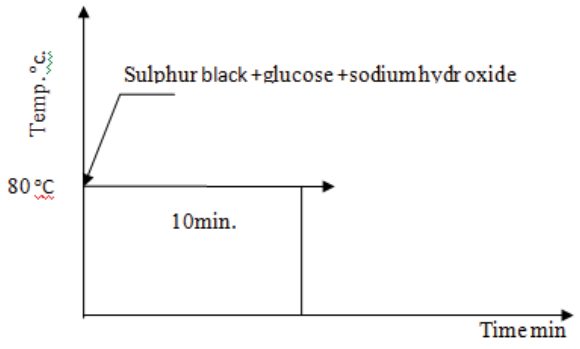

Figure 1: Reduction cycle of sulphur black.

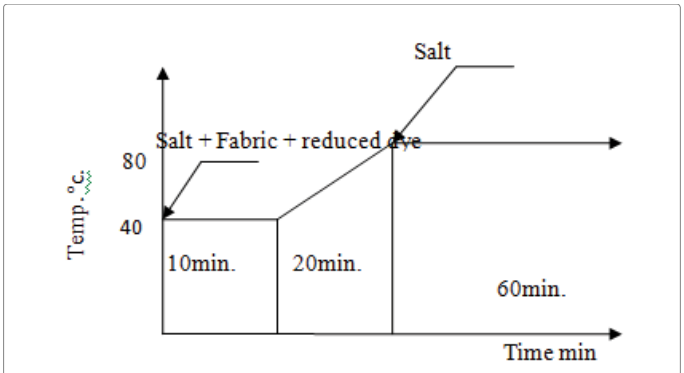

Figure 2: Dyeing cycle of sulphur black.

Then it was rinsed with tap water.

Oxidation: 60°C, 10 min, 3 g/l H_2O_2, 1 g/l CH_3COOH, 250 ml for each sample, then it was rinsed with tap water.

Soaping: (80°C, 5 min), then it was rinsed with tap water.

Drying: 100°C, 3 min.

Result and Discussion

Sulphur dyes are water insoluble dyes and have no affinity as like cellulose have but solubilised when treated with a weak alkaline solution of sodium sulphide or any other reducing agent to form a leuco compound. These leuco compounds are water soluble and have affinity for the cellulosic materials such as cotton, viscose, jute and flex etc. These dyes are absorbed by the cellulosic material in the leuco form from aqueous solution and when oxidized by suitable oxidizing agents, got converted into insoluble parent dye, which is fast to normal color fastness parameters. Due to the environmental problem of sodium sulphide we can replace with Hydrolyzed sugar.

Hydrolysis of sugar with hydrochloric acid can perform at different temperatures. But here we do only at room temperature and it will be done two times to check the reproducibility of the hydrolysed sugar.

Dyeing of cotton with sulphur black was done in using of different reducing chemicals. These chemicals have not the same reduction potentials and due to this we got different dyeing results.

Blank sample

Some textile dyestuffs are water insoluble and this makes difficult for dyeing. So for dyeing is carried out those dyes must change in to reduced form. From these sulphur dyes are grouped under this. Sulphur dyes have not a dyeing ability if they are not reduced to leuco or reduced form.

To see the difference in between samples dyed with reduced sulphur and before reduction this sample was done. The sample was under go for dyeing without any reducing chemical using of sulphur black dyeing cycle. And the result has no any dyeing effect rather it is staining effect on the surface of the fabric because Sulphur dyes are water insoluble dyes and have no affinity as like cellulose.

Sodium sulphide as a reducing chemical

The sample dyed in this condition visually has a lighter shade than the samples dyed with hydrolysed sugar using as a reducing chemical but deeper in shade than sample dyed without reduction.

Hydrolysed sugar as reducing chemical

In this condition different samples are dyed in the same dyeing condition of sulphur black. Their difference is the waiting time of hydrolysed sugar and batch of hydrolysis. The hydrolysis of sugar was done in two batches in the same condition at room temperature and from each batch dyeing is carried out at different waiting time intervals of the hydrolysed sugar in the same dyeing condition.

First batch samples: These are the samples which are dyed by reducing of the dyestuff from the first hydrolysed sugar and dyeing is takes place after a wait of three days of hydrolysis and then for 40 days. Waiting time means the time of glucose formation. Here visually the samples are identical in their shade to each other.

Second batch samples: In this case hydrolysis was done next to that of the first hydrolysis. And Dyeing is takes place after one, two and three days of sugar hydrolysis consecutively in the same dyeing conditions. After dyeing is carried out for checking of the efficiency and effectiveness of the dyeing chemicals we do some analysis. The analysis was visual detection and experimental methods. In visual observation samples dyed with hydrolysed sugar as reducing chemical have deeper shade than the sample which was dyed with sodium sulphide. The samples dyed with hydrolysed sugar as a reducing chemical in between batches and within batches seem similar results in visual detection.

In experimental analysis two experimental tests were done. These are

- k/s value and
- Wash fastness tests

Wash fastness tests: It includes following methods:

- ***Color Fastness:*** The resistance of a material to change in any of its color characteristics, when subjected to washing is called color fastness to washing.
- ***General principle:*** A specimen of the textile to be tested, with the adjacent fabric attached is subjected to washing under specified conditions. Te extent of any change in color and that of the staining of the adjacent fabric are assessed and the rating is expressed in fastness numbers. There are two types of adjacent fabrics; (1) single fiber fabric and multiple fiber fabric. In the case of multitier fabric only one specimen is required and in the of single fiber fabric two adjacent fabric are required.

Wash fastness is done to compare the fixation rate of the dyestuff to the fabric surface between the original and reuse samples. This is carried out on Laundrometer based on ASTM standard for wash fastness of dyed fabrics. The parameters are MLR of 1:50 and 5 gpl detergent and treatment at 600°C for 30 min. The fabric specimen needed for this test is prepared by cutting on 10 cm x 4 cm size from the dyed fabric, bleached fabric and polyester fabrics. The three samples will be stitched in the sandwich system i.e. the dyed fabric is inside the two Undyed fabrics. During test there is also adding of 10 stainless steel balls to the bath which contains sample fabric and detergent. After 30 min. the stitched sample was unstitched the two long sides' and one short side and when it dries comparison is made with original sample. This comparison also compare with the standard grey scale to get the reading by visual observation [7].

- ***Fastness to washing:*** In the test, change in color of the textile and also staining of color on the adjacent fabric are assessed. A 10 cm × 4 cm swatch of the colored fabric is taken and is sandwiched between two adjacent fabrics and stitched, the sample and the adjacent fabric are washed together. Five different types of washing are specified. Depending of the gray scale reading the wash fastness will range from 1 to 5. Which means 1 indicates poor fastness and 5 is good fastness. But this is done in visual comparison. The wash fastness result as per the Gray scale reading will as follows (Table 3).

k/s value test: The color strength (K/S) value was assessed using spectrophotometer. A spectrophotometer device for the measurement of spectral transmittance or spectral reflectance, an instrument that measures the characteristics of light reflected from or transmitted through an object, which is interpreted as spectral data.

K/s is the ratio of the absorption coefficient (K) versus the scattering coefficient (S) for a reflectance measurement. The ratio is derived mathematically from the reflectance measurement.

This is already done three times for each sample at different positions. And the result as per the reading is as follows (Table 4):

Sample name	Wash fastness result(as per the gray scale reading)
S1(first sample after one day of hydrolysis second batch)	5
S2(second sample after two days of hydrolysis second batch)	5
S3(third sample after three days of hydrolysis second batch)	5
S4(first sample after one day hydrolysis of first batch)	5
S5(second sample after 40 days of hydrolysis first)	5
S6(sample dyed using of sodium sulphide as reducing chemical)	5
S7(blank sample)	3/4

Note: From the above table results we can understand that all the samples except the blank one have a reasonable wash fastness.

Table 3: Wash fastness test results as per the grey scale Reading.

Sample name	k/s value of samples
S1(first sample after one day of second batch)	27.68 at 560 nm wave length
S2(second sample after two days of hydrolysis second batch)	28.7114 at 540 nm wave length
S3(third sample after three days of hydrolysis second batch)	29.3726 at 540 and 560 nm wave length
S4(first sample after three days of first batch)	29.683 at 540 and 560 nm wave length
S5(second sample after 40 days of hydrolysis first batch)	29.4582 at 540 and 560 nm wave length
S6(sample dyed using of sodium sulphide as reducing chemical)	25.6998 at 540 nm wave length

Table 4: k/s value of samples.

As you seen from the above table we can understand that, the time of hydrolysis has an impact on the formation of hydrolysed glucose from sugar. If it has higher waiting time it gives better glucose formation. This means as waiting time increases the chance of glucose formation increases. If it has higher than 3 days waiting time it gives better glucose formation. This was checked by the depth of shade of the dyed material.

From the above table samples which are dyed after one and two days of glucose formation time has lighter shade than samples dyed after three and above waiting time of glucose formation. But if it is below that its shade is comparable lighter means the formation of glucose is not completed.

The other thing here is that samples dyed using of hydrolysed sugar after three days and above have same depth of shade. This means the glucose formation waiting time needs 3 days. And glucose formation may continue but for dyeing we can get enough glucose after three days. Because the samples dyed with glucose which have 3 days waiting time and 40 days waiting time haves similar depth of shades. Using of hydrolysed as a reducing chemical gives better result in depth of shade than of sodium sulphide. This means hydrolysed sugar has better reduction potential than sodium sulphide. So, using of hydrolysed sugar as a reducing chemical has extra advantage in depth of shade of the dyed material.

Conclusion

Generally dyeing of cotton fabric with sulphur black is possible using of sodium sulphide and hydrolysed sugar as a reducing chemical. But due to the environmental problems of sodium sulphide like

- Bad smell
- Toxicity
- Contamination of sea/river and destroys marine life
- High COD

It is not recommended to use sodium sulphide as a reducing chemical during dyeing of cotton fabric with sulphur black. The right way to solve this environmental problem is using of hydrolysed sugar as a reducing chemical for sulphur black dyeing of cotton fabric.

Not only was that, using of hydrolysed rather than sodium sulphide as a reducing chemical also has some additional advantages. Fabrics dyed using hydrolysed sugar as a reducing chemical has also better depth of shade in color than samples dyed under sodium sulphide reduction.

The other thing is it forms comfortable working conditions for workers because the bad odours and inhalation of sulphur fumes have been removed and the substituted chemical is much safer to handle.

Quality of the dyed fabric is better in case of hydrolysed sugar reduction. Elimination of free sulfur from the fabric helps for the termination of formation Sulfuric Acid which forms by oxidizing of free sulfuric, which then attacks and may eventually destroy the fabric.

Generally using of hydrolysed sugar as a reducing chemical during sulphur dyeing of cotton fabric have following merits:-

- Environmental
- Financial
- In Quality wise and
- Formation of Comfortable working condition for workers and other advantages.

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