Middle-East Journal of Scientific Research 22 (7): 1090-1095, 2014
ISSN 1990-9233

DOI: 10.5829/idosi.mejsr.2014.22.07.21975

Eco-Sustainable Process Development for Indigo Dye by Using Natural Reducing Agents

[1]Mashiur Rahman Khan, [2]Zulhash Uddin and [3]Delwar Hossain

[1]Department of Apparel Manufacturing Engineering,
Bangladesh University of Textiles, Bangladesh
[2]Faculty of Textile Chemical Processing Engineering and Applied Science,
Bangladesh University of Textiles, Bangladesh
[3]Department of Textile Engineering, Dhaka University of Engineering and Technology, Bangladesh

Abstract: Natural indigo dye is basically use for denim fabric dyeing. For most industrial vat and indigo, dyeing processes, all of them are reduced mainly using sodium dithionite. This process produces large amounts of hazardous by-products which increase the costs for waste water treatment. Hence, many attempts are being made to replace the environmentally unfavorable sodium dithionite by ecologically more attractive alternatives. In this research, a natural reducing agent is ushered in that comes from a fruits (date, apple and banana) and have been studied on the cellulosic fiber for comparison it with sodium dithionite for dyeing on denim garments. This paper also confronts a set of comparison between natural indigo dyeing process with synthetic indigo dyeing, using natural and synthetic reducing agent considering physical properties (K/S value, colour difference,light fastness,fastness to rubbing, fastness to wash) of the treated samples. Structural changes of fiber were studied by using SEM (Scanning Electron Microscope) after using different reducing agents.

Key words: Natural indigo dye · Natural reducing agents · Indigo dye · SEM · Cellulosic fiber

INTRODUCTION

In indigo dyeing process have two major limitations: the selection of the reducing agent and the method of application. Indeed, using sodium dithionite is the cause of certain engineering problems, such as instability, storage, corrosivity, etc, [1] and ecological problems because of the production of large amounts of sodium sulphate and sulphite [2], known to increase the cost of wastewater treatment [3]. On the other hand, dyeing with indigo in a continuous process presents a well known limit: the inability to satisfy all consumers' expectations and to follow the evolution in fashion tendencies, which are easier to accomplish by an exhausting process. These problems were the main motivation to conduct this work. Firstly, it is required in the case of indigo to develop a new process and achieve a better dyeing quality than the one obtained in a conventional process. Secondly, the environmentally unfavorable sodium dithionite has to be substituted by ecologically more attractive alternatives. Investigations focused on the replacement of sodium dithionite by natural reducing agents. The use of sodium dithionite is known to offer an effective reduction of indigo, as well as other vat dyes and it enables very short fixing times in various dyeing methods [4]. Nevertheless, sodium dithionite has many issues such as low stability, it is easily oxidized by atmospheric oxygen [1] and risks of fire and health hazards during storage. Besides, the stability of its alkaline solutions decreases with the increase of temperature, even in the absence of oxygen [5]. Hence, large amounts of dithionite and sodium hydroxide are needed over the stoichiometric requirements of the reduction process. Furthermore, the oxidation of byproducts causes various problems with wastewater disposal [6]. In fact, sulphate, sulphite and thiosulphate ions resulting from the oxidation of sodium dithionite during the reducing process have harmful effect on the environment due to their toxicity, as well as their corrosive effect on the waste lines [7]. In addition, sodium dithionite affects the aerobic processes in

Corresponding Author: Delwar Hossain, Department of Textile Engineering,
Dhaka University of Engineering and Technology, Bangladesh. E-mail: delwar@duet.ac.bd.

Table 1: Fabric specification

Characteristics		Cotton
Yarn count (Tex)	Warp	12
	Weft	08
Ends per inch		120
Picks per inch		62
Weave		3/1 (Twill)

wastewater treatments and toxic hydrogen sulphide can be generated an aerobically from the sulphate present in wastewaters [4]. Possible alternatives for the application of dithionite as the reducing agent are bacteria induced reduction and electrochemical reduction. Glucose and other reducing sugars have recently been suggested as possible environmentally friendly alternatives as reducing agents for sulphur dyes [5] and there has also been interest in using glucose to educe indigo [6]. By considering large amounts of ingestion of indigo dye for blue jean and denim, it is important to develop eco-sustainable reduction system by providing a greener technology. Moreover, the use of reducing sugars extracted from food by-product may result in a significant saving of production costs and waste water treatment costs over current technology that employ sodium dithionite reduction [8]. This study aims at investigating the reduction and analysis methods of indigo and give insight on the reduction mechanism of indigo.

Experimental Section

Materials

Indigo Dyes and Reducing Agents: The commercially available natural indigo used in this study was extracted from Indigofera tinctoria, Brand-Living blue. The synthetic vat dye was collected from BASF. Natural reducing agents (Date, Apple, Banana) collected from local fruit market [9].

Fabric Specification:

Indigo Dyes and Reducing Agents: The commercially available natural indigo used in this study was extracted from Indigofera *tinctoria,* Brand- Living blue. The synthetic vat dye was collected from BASF. Natural reducing agents(Date,Apple,Banana) collected from local fruit market.

Methods

Extraction of Reducing Agents: At first natural reducing agents (Apple, Banana, Date) are collected from local fruit market and chopping into small pieces with knife. One liter natural reducing agent can produce by synthesis 200 gram apple. Then the fruits peels were boiled for 30 min to obtain extracts. The extract was filtered and preserves the extract as a liquid form in a suitable jar in a room temperature.

Dyeing: Different dyeing condition has been used (Table 2). Three reducing agents with different amount has been used with fixed amount of dye 10 gm/l. Dyed fabric was washed with cold water and dried in room temperature.

Conditions for Experimental Process: Table 2 Conditions for dyeing with natural indigo dye with natural reducing agents (dates, banana, apple).

Measurement of Colour Strength: The colour yield of dyed samples were evaluated by light reflectance measurement using Data Color machine. The colour strength (K/S value) was assessed using the following Kubelka-Munk equation: [9]

$$K/S=(1-R)^2/2R$$

Where R is the decimal fraction of the reflectance of dyed fabric.

Fastness Testing: The dyed samples were tested according to ISO standards. The specific test were: Color fastness to rubbing- Test Method: ISO 105x12, Color fastness to perspiration-Test Method: ISO 105-EO4,Colour fastness to wash- Test Method: ISO 105C06C2S,Colour fastness to light- Test Method: ISO B02.

RESULTS AND DISCUSSION

Colour Fastness Properties: The dyed samples were tested for the wash fastness. The grey scale rating was done on a scale of 1-5. Among three natural reducing agents date reflects better performance than the apple and respectively the banana. The result (Table 3) shows that dry fastness is much better then the wet fastness. The overall rating of experimental samples specially sample TD107 is latitude to the standard sample. The fastness to perspirations was also satisfactory but the light fastness poses grade 4-5 standard which reference moderate to good result.

Table 2: Conditions for dyeing with natural indigo dye with natural reducing agents (dates, banana, apple)

			Dyeing conditions							
Sl.no	Types of Fabric	Types of dyes	Time(min)	Temp.(°C)	PH	Dye con. (gm/l)	Reducing agent (gm/l)	Ca(OH)2 (gm/l)	Sample code	Types of Reducing agent
01	Twill	Natural Indigo	30	30	10.8	10	100	10	TA101	Apple
02							150		TA102	
03							200		TA103	
04							250		TA104	
05							100	15	TA105	
06							150		TA106	
07							200		TA107	
08							250		TA108	
09							100	20	TA109	
10							150		TA110	
11							200		TA111	
12							250		TA112	
13							100	10	TB101	Banana
14							150		TB102	
15							200		TB103	
16							250		TB104	
17							100	15	TB105	
18							150		TB106	
19							200		TB107	
20							250		TB108	
21							100	20	TB109	
22							150		TB110	
23							200		TB111	
24							250		TB112	
25							100	10	TD101	Date
26							150		TD102	
27							200		TD103	
28							250		TD104	
29							100	15	TD105	
30							150		TD106	
31							200		TD107	
32							250		TD108	
33							100	20	TD109	
34							150		TD110	
35							200		TD111	
36							250		TD112	

Optimization of Reducing Agents with K/S Value and Colour Difference: Different reducing agents were used in different amount with fixed amount of dye-10gm/l. Varied hues were obtained from the treated samples. As shown in Figure 2 the different reducing agents cause difference in the hue color and significant changes in K/S value which is correlated to strength difference(Figure 3). The sample (TD-107) treated with 200 gm/l reducing agent (date) with 15 gm/l of CaOH shows highest K/S value compare to standard sample. Which indicate the higher absorbency of dye molecule in the fiber tends to get deeper shed.

SEM Analysis: The effect of dyeing on cellulosic fabric dyed with various reducing agents using different dye s like natural indigo and synthetic indigo dye and its combination with natural reducing agent and conventional reducing agent (Hydrose) are analyzed by SEM and the results are shown in Figure 4.

It is clear that the combination of natural indigo dye with natural reducing agent (date) produce a smooth and compact surface of fabric. On the other hand the combination of natural reducing agent with Sodium di thionite and the combination of synthetic indigo dye with Sodium di thionite shows typical hairiness on the fabric

Table 3: Colour fastness results of Natural indigo Dyed samples

					Colour fastness to Wash			
			Colour fastness to Rub			Staining on		
Sl No.	Sample code	Fabric type	Dry Condition	Wet Condition	Change of Shade	Cotton	Multi fiber	Colour fastness to Light
1.	Standard sample	Twill	4/5	4	4/5	4/5	4	6
2.	TA101		4	4	4	4	4	4
3.	TA102		4	3	3/4	4/5	3/4	5
4.	TA103		4	3/4	3/4	4/5	3/4	4
5.	TA104		4	3/4	3/4	4	3/4	4
6.	TA105		4	3/4	4	4/5	3/4	3
7.	TA106		4	3	3/4	4/5	3/4	2
8.	TA107		4	3	4/5	4/5	4	4
9.	TA108		4	3	4/5	4	3	4
10.	TA109		3/4	3	3/4	4	3/4	5
11.	TA110		4	3	3/4	4/5	3/4	5
12.	TA111		4	3/4	3/4	4	3/4	4
13.	TA112		4	3	4	4	4	3
14.	TB101		4	3	3/4	3/4	4	4
15.	TB102		4	3	3	3	3/4	4
16.	TB103		4	3/4	4	3	3	5
17.	TB104		4	3/4	4	3	3	5
18.	TB105		×	×	×	×	×	×
19.	TB106		×	×	×	×	×	×
20.	TB107		×	×	×	×	×	×
21.	TB108		×	×	×	×	×	×
22.	TB109		4	3/4	3	3	3	4
23.	TB110		4	3/4	4	3	3	4
24.	TB111		4	3/4	3/4	3	3	4
25.	TB112							4
26.	TD101		4	3/4	3/4	4	4	4
27.	TD102		3/4	4	4	3/4	4	5
28.	TD103		4	4/5	4/5	3/4	3/4	4
29.	TD104		4	4	4	3/4	3/4	4
30.	TD105		3/4	3/4	4	3/4	3/4	4
31.	TD106		4	3/4	4	3/4	3/4	4
32.	TD107		4/5	4	4/5	4/5	4	5
33.	TD108		4	4	4/5	4	4	4
34.	TD109		3/4	3/4	4	3/4	3/4	4
35.	TD110		4	4/5	4/5	3/4	3/4	5
36.	TD111		3/4	4	4	3/4	3/4	4
37.	TD112		4	3/4	3/4	3	4	4

*×=Formulated recipe did not work.

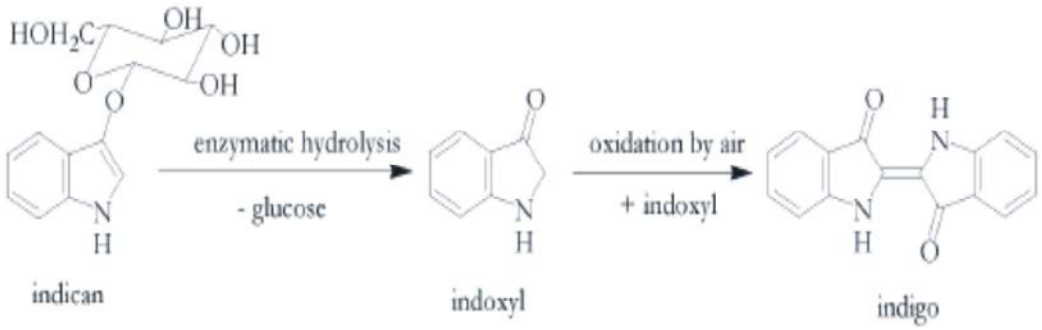

Fig 1: Production of Indigo from indigo plants

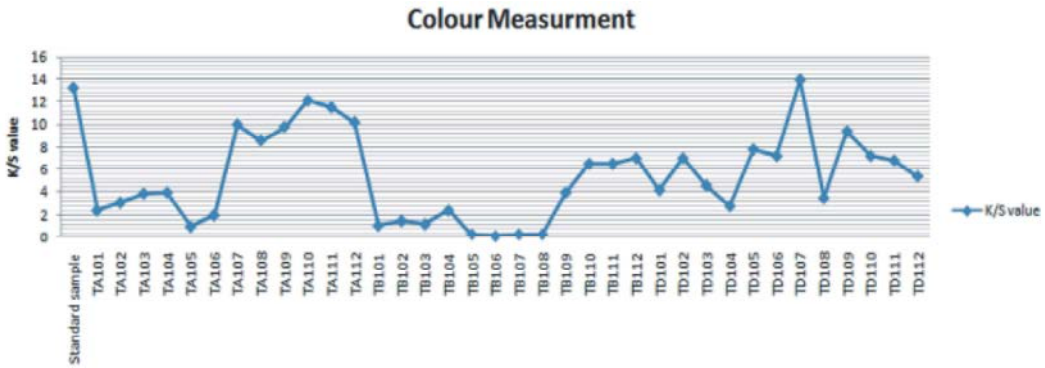

Fig 2: K/S value of samples with different reducing agents

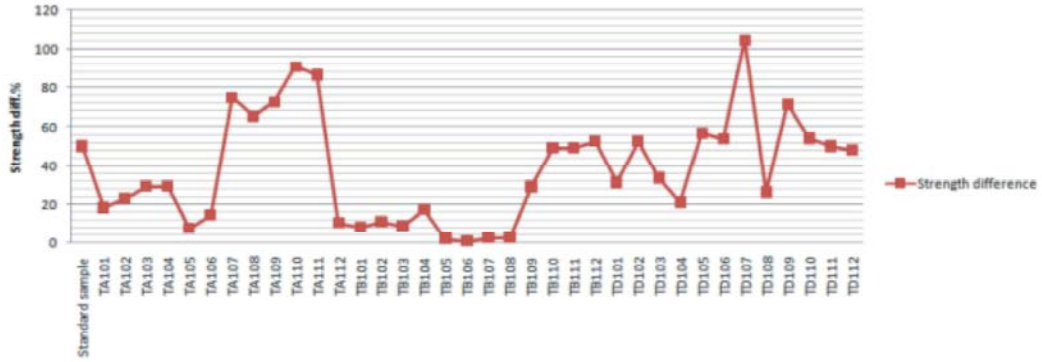

Fig 3: Strength difference % of samples with different reducing agents

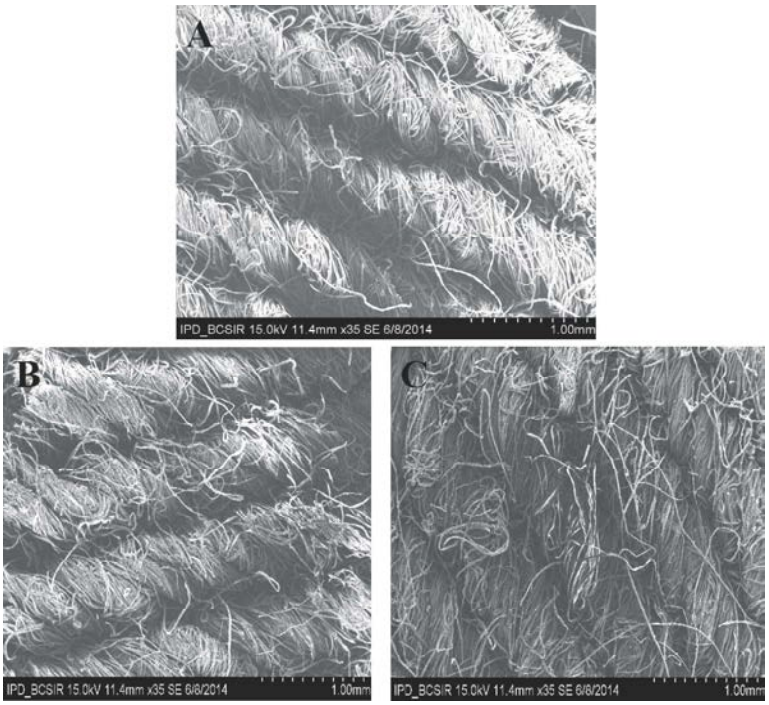

Fig 4: A) SEM photograph of cotton fabric dyed with Natural indigo dye using Natural reducing agent(Date) B) SEM photograph of cotton fabric dyed with Natural indigo dye using Sodium di thionite C) SEM photograph of cotton fabric dyed with synthetic indigo dye using Sodium di thionite.

surface which posses less smooth surface with uneven effect and less compactness of the fabric that can be conduce the fabric comfort ability as Drape, Stiffness, Air permeability properties.

CONCLUSION

From the various results obtained in this research work and the discussions carried out, the conclusions arrived are summarized below:

Indigo dye is used for the dyeing of cotton fabric in an environmentally suitable way. For this, different reducing agents such as date, banana, green apple etc. has been used in this experiment.

The objective measurements and evaluation affirmed that the physical properties of the sample treated by natural indigo dye with natural reducing agent is relatively better in comparison with natural dye with synthetic sodium di tionite and synthetic vat dyeing process The natural reducing agent (date, apple and banana) has the best reducing activity at high temperature and alkalinity, more specifically at a temperature higher than 80°C and a calcium hydroxide concentration of 15 g/L. The natural reducing agent date gives the best result compare to apple; banana.Thus, the dyeing bath was adjusted according to these results in order to determine the optimal physiochemical parameters of the dyeing process. As a result, it can be noted that for the reduction of indigo by natural reducing agent, a concentration of 15 g/L of calcium hydroxide, 200 ml/L of natural reducing agent, 10 g/L of natural indigo, a reduction time of 10 minutes, a dyeing time of 30 minutes and a reduction temperature of 80°C, are the optimal conditions to obtain the best dyeing quality of denim fabrics with natural indigo reduced by natural reducing agent. The analysis depended on three factors: temperature, sodium calcium hydroxide and natural reducing agent concentrations. Moreover, the interactions between different factors as well as their main effects on both responses studied were determined.

REFERENCES

1. Camacho, F., M.P. Páez, M.C. Jiménez and M. Fernández, 1997. Application of the sodium dithionite oxidationto measure oxygen transfer parameters, Chem. Eng. Sci., 52: 1387-1391.
2. Božič, M. and V. Kokol, 2008. Ecological alternatives to the reduction and oxidation processes in dyeing with vat and sulphur dyes, Dyes and Pigments, 76: 299-309.
3. Roessler, A. and X. Jin, 2003. State of the art technologies and new electrochemical methods for the reduction of vat dyes, Dyes and Pigments, 59: 223-235.
4. Božič, M. and V. Kokol, 2008. DYES PIGMENTS, 76: 299-309.
5. Blackburn, R.S. and A. Harvey, 2004. Green Chemistry Methods in Sulfur Dyeing: Application of Various Reducing D-Sugars and Analysis of the Importance of Optimum Redox Potential Environ. Sci. Technol., 38: 4034-4039.
6. Ushida, S. and M. Matsuo, 1991. Reduction of indigo by glucose, Nippon Kasei Gakkaishi., 42: 61-65.
7. Bechtold, T. and A. Turcanu, 2009. J. Cleaner Production, 17: 1669.
8. Shin, Younsook, Min Choi and Dong Il Yoo, 2013. Utilization of fruit by-products for organic reducing agent in indigo dyeing, Fibers and Polymers, 14(12): 2027-2031.
9. Mongkholrattanasit, Rattanaphol, Jiří Kryštùfek and Jakub Wiener, 2010. Dyeing and fastness properties of natural dyes extracted from eucalyptus leaves using padding techniques, Fibers and Polymer, 11(3): 346-350.