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DYEING COTTON FABRIC WITH INDIGO DYE BY USING SELECTED NATURAL REDUCING AGENTS

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Abstract-natural indigo dye is basically used for denim fabric dyeing. Indigo is a vat dye, which means that it needs to be reduced to its water soluble leuco form before dyeing. For most industrial vat and indigo, dyeing processes, all of them are reduced mainly using chemically. However, this is considered environmentally unfavourable because of waste waters contaminating degradation products. 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 unfavourable chemical by ecologically by more attractive alternatives. In this study, a natural reducing agent which is prepared from a fruits like banana, banana peel, mango, mango peel, dates, grapes, green apple, green apple peel and figs have been studied on the cellulosic fiber (cotton fabric). This study also confronts a set of physical properties of dyed sample k/s value, light fastness, fastness to rubbing, fastness to wash, fastness to perspiration. The analysis depended on three factor are temperature, sodium calcium hydroxide and natural reducing agent concentrations. Natural reducing agent has best reducing activity at the temperature of 80°c for 1 hr. The 25ml of reducing agent and 1g of calcium hydroxide gave best result in vatting process at the temperature 50 ºc for 10 minutes duration whereas 10ml of reducing agent and 0.5g of calcium hydroxide gave best result in dyeing process at the temperature of 50ºc for 30 minutes duration. The reducing agent extracted from banana gave best as compare to other fruits in term of k/s value which was 0.930 and the colourfastness properties was also good as compared to other reducing agents.

Introduction

Indigo dye is an important dyestuff with a unique shade of blue color. The natural dye comes from several species of plant. The dye gives a brilliant and eye-catching blue color to the fabric. Though this colour partially penetrates into the fabric, it imparts surface blue colour. The word indigo is derived from the latin indicum and the greek indikon meaning 'blue dye from india' or being more specific-'indian substance'. [1] indigo is basically a dark blue crystalline powder that melts at 390-392c. It is soluble in chloroform, nitrobenzene, or concentrated sulphuric acid, but not soluble in water, alcohol and ether. The chemical structure/formula of indigo is c16h10n2o2. [2]

Natural indigo is the oldest known dye to mankind. When the synthetic substitute of the dye was not invented, all blue textiles were used to be dyed with indigo. These included the blue serge uniforms worn by the british police force and hospital staff, as well as military personnel and workman's clothes worn by millions of people, inspiring the term 'blue-collar worker'. [3] Asian countries like india, china and japan had used indigo as a dye for centuries.

The ancient civilizations in mesopotamia, egypt, greece, rome, britain, peru, iran, and africa had the knowledge about the dye. Since neolithic times in europe this dye is being used it was highly prized for its colour and light fastness .until the end of the 19th century; the sole source was from plants, woad (isatis tinctoria) and dyer's knotweed (polygonum tinctorum) in temperate climates and indigofera species in the tropics. Woad was widely grown in Europe, making some regions, especially toulouse (france) and erfurt (germany) making them economically very prosperous. It was used to make a woad vat for dyeing with indigo from india after that period it belongs to the legume family and its three hundred plus species have been identified. In ancient times, indigo was considered a precious commodity because plant leaves contain only about small amount of the dye (about 2-4%). Indigo remained a rare commodity in Europe throughout the middle ages; woad, a dye derived from a related plant species, was used instead. Indigofera tinctoria was originally domesticated in India; it is mentioned in manuscripts dating from the 4th century bc about its origin in india. [4]

Since synthetic indigo dye is being used, use of natural indigo dye has almost became extinct, but in recent years, the demand for natural dyes has been increasing in many countries again, because of health and pollution effects and a revival of interest in the relationship between dyes and culture. In present time, indigo is still cultivated for dye on a small scale basis in India (particularly in the northern part of Karnataka) and in some parts of Africa and central America. It is frequently grown as a secondary crop. Dissemination is usually by seeds which are sown at first in seed beds or directly into the field. Germination takes about 4 to 5 days. When the plants are about 4 to 5 months old and starts flowering then the branches are harvested. At 3 to 4 months intervals three times a year the plants could be continued to be harvested. The total life span of the plants could range from time period 2 to 3 years. [5]

For natural indigo dye the leaves of indigo go through a process of fermentation and then oxidized to yield the blue dye. Traditionally fermentation was carried out by bacteria. Firstly harvested plants are packed into tanks and covered with water. After a few hours of soaking the plants in water, the leaves become saturated and fermentation begins. A thick layer of foam bubbles are formed at the top surface of the tank. The process can be so vigorous that planks are placed on top of the tub to keep the plants in. This process can take up to a day and a half to complete, but the timing has to be perfect .the indigo makers will smell and taste the fluid to check whether the dye is ready or not. Even if an hour is taken extra it could ruin it. As soon as the liquid tastes sweet and is a dark blue colour, it is drained off into another vessel at a lower level, leaving the plants behind. The liquid now formed contains indoxyl. [6] to stimulate oxidation of the indoxyl the liquid is stirred continuously for many hours for it requires oxygen for oxidation. On an alternative basis people will get into the vats to tread up and down to stir it up. After sometime the liquid turns a yellow-brown colour with floating dark blue patches. The solution is left to rest and the insoluble indigo settles to the bottom of the tank as a bluish sludge. To remove impurities and to stop the enzyme reaction which made the indigo the water is drained and filtered. The sludge is then dried to produce indigo 'cake' which is cut into cubes or made into balls. [7] indigo is a so-called vat dye, which means that it needs to be reduced to its water soluble leuco-form before dyeing. The reduced form is absorbed into the fibres, and when oxidized back to its blue form it stays within the fibre. Earlier the reduction and dyeing was done with fermentation. Nowadays, the most of the reduction has been done chemically by sodium dithionite. It is considered environmentally unfavourable since it produces sulphite, sulphate, thiosulphate and toxic sulphides as degradation products, which then contaminate the waste waters from the dyeing plants. Therefore there has been interest to find new possibilities to reduce indigo. Possible alternatives for the application of dithionite as the reducing agent are bacteria induced reduction and electrochemical reduction. A gram-positive, aerobic moderate and thermophile bacteria (clostridium isatidis) capable of reducing indigo dye was isolated from woad vat at the university of reading. In the electrochemical approach the possibilities are direct or indirect electrochemical reduction with different redox mediators. Organic reducing agents have also been possible alternatives to the sodium dithionite. Glucose and other reducing sugars have recently been suggested as possible environmentally friendly alternatives as reducing agents for sulphur dyes and there has also been interest in using glucose to reduce indigo. Anthraquinones have been previously recognized to stimulate indigo reduction by pure cultures of bacteria and anthraquinone-rich madder powder is known to have been an invariable ingredient in the medieval indigo dye

vat. Anthraquinones are also known to act as mediators in the indirect electrochemical reduction of indigo, where they transfer electrons between electrode and dye molecule. Some of the reducing agent is urine. Bran, madder, yeast, sugar, molasses, sodium hydrosulphite, ferrous sulphate, thiourea dioxide (thiox).[8] earlier the reduction and dyeing was done with fermentation which was an eco friendly process. Nowadays, the most of the reduction has been done chemically. It is considered environmentally unfavourable since it produces sulphite, sulphate, thiosulphate and toxic sulphides as this process produces large amounts of hazardous by-products which increase the costs for waste water treatment. Hence, an attempt is being made to replace the environmentally unfavourable chemical by ecologically more attractive alternative such as naturally available fruits have been used as reducing agent in indigo to dye cotton fabric. [9]

Objectives of the study

- To identify eco sustainable reducing agent for indigo dye.
- To dye the cotton fabric with indigo dyes using natural reducing agents
- To evaluate the colour strength, washing fastness, perspiration fastness, rubbing fastness, light fastness of dyed fabrics.

Materials and methods

The following section describes the materials used and methods followed in this research work.

Materials

1. Fabric

The commercially available ready to dye cotton fabric was purchased from jharcraft, commercial street, Bangalore and used for dyeing with indigo. The geometric properties of the fabric are given in the table below:

Table 1- characteristics of fabric

Sl. No.	Fabric properties	Values
1	Type of weave	Plain weave
2	Ends/inch	89
3	Pick/inch	68
4	Warp count(tex)	22
5	Weft count(tex)	28
6	Warp cover factor	11.65
7	Weft cover factor	11.12
8	Cloth cover factor	17.92
9	Gsm	88
10	Yarn twist	S

2. Dye source

The commercially available natural indigo used in this study. The indigo was sourced from the commercial market.

common name: indigo blue

botanical name: indigofera tinctorie

main coloring: indigotin

3. Auxiliary used

The commercially available auxiliary was used in this study. The auxiliary used are mentioned below:

3.1 Reducing agent

The fruit extraction was used as a reducing agent for making the insoluble dye in soluble leuco indigo form. The reducing agent used in study is mentioned below

Table 2- fruits used

Fruits	Code
Banana	A
Banana peel	B
Mango	C
Mango peel	D
Dates	E
Grapes	F
Green apple	G
Green apple peel	H
Figs	I

3.2 Calcium hydroxide

The calcium hydroxide is also called as ordinary lime, hydrated lime. It is used as a base for natural indigo dye.

Methods

4. Optimization of the reducing agents for extraction

Firstly, natural reducing agents are collected from local fruit market and cut into small piece. The fruit peels were also used separately. The quantities of the fruits used were varied during optimization process.

Table 3 - optimization of fruit used

Duration (hr)	Temperature	Dye %	Reducing agent (g/l)	Type of fruits	Sample code
1	40ºc 50ºc 60ºc 70ºc 80ºc 90ºc	5	50, 100, 150 200, 250, 300 400, 450, 500 550, 600, 650	Banana	A
1	40ºc 50ºc 60ºc 70ºc 80ºc 90ºc	5	50, 100, 150 200, 250, 300 400, 450, 500 550, 600, 650	Banana Peel	B
1	40ºc 50ºc 60ºc 70ºc 80ºc 90ºc	5	50, 100, 150 200, 250, 300 400, 450, 500 550, 600, 650	Mango	C

1	40ºc 50ºc 60ºc 70ºc 80ºc 90ºc	5	50, 100, 150 200, 250, 300 400, 450, 500 550, 600, 650	Mango Peel	D
1	40ºc 50ºc 60ºc 70ºc 80ºc 90ºc	5	50, 100, 150 200, 250, 300 400, 450, 500 550, 600, 650	Dates	E

Duration (hr)	Temperature	Dye %	Reducing agent (g/l)	Type of fruits	Sample code
1	40ºc 50ºc 60ºc 70ºc 80ºc 90ºc	5	50, 100, 150 200, 250, 300 400, 450, 500 550, 600, 650	Grapes	F
1	40ºc 50ºc 60ºc 70ºc 80ºc 90ºc	5	50, 100, 150 200, 250, 300 400, 450, 500 550, 600, 650	Green Apple	G
1	40ºc 50ºc 60ºc 70ºc 80ºc 90ºc	5	50, 100, 150 200, 250, 300 400, 450, 500 550, 600, 650	Green Apple Peel	H
1	40ºc 50ºc 60ºc 70ºc 80ºc 90ºc	5	50, 100, 150 200, 250, 300 400, 450, 500 550, 600, 650	Figs	I

Natural reducing agents were optimized by varying volume of fruits and fruit peels at different temperatures. About 500 g of natural reducing agent was boiled in 1000ml of water at 80ºc for 1 hr to obtain right volume extracts. Then, the extract was filtered and then preserved for further use.

5. Optimization of auxiliary used for vatting and dyeing

5.1 Reducing agent

The quantities of extracted reducing agent used were varied during optimization process of indigo vatting and dye bath.

Table 4- optimization of reducing agent used

Vatting(ml)	Dye bath(ml)
0.5	0.5
1	1
2	2
3	3
4	4
5	5
6	6
7	7
8	8
9	9
10	10
1l	11
12	12
13	13

Based on optimization process 25ml of reducing agent gave better result in vatting whereas 10ml of reducing agent gave better result in dye bath as compare to other quantities.

5.2 Calcium hydroxide

The amounts of the calcium hydroxide used were varied during optimization process of indigo vatting and dye bath.

Table 5- optimization of calcium hydroxide used

Vatting (g)	Dye bath (g)
1	**0.5**
2	1
3	2
4	3
5	4
6	5
7	6
8	7

Based on optimization process 1g of calcium hydroxide gave better result in vatting whereas 0.5g of calcium hydroxide gave better result in dye bath.

6. Optimizing the indigo dyeing process

The indigo dye is one of the classifications of the vat dye. The indigo dyes are insoluble in nature and consist of following stages

6.1 vatting

The process of converting insoluble dye into soluble form is called as vatting as an indigo need to be reduced to its water-soluble form before it can be used in dyeing. The auxiliary used are fruit extraction and calcium hydroxide.

Table 6- vatting recipe

Recipe	
Dye	5%
Reducing agent	25ml
Calcium hydroxide:	1g
Temperature:	50ºc
Duration	15 min

Based on optimization process 1g of calcium hydroxide in 25ml of reducing agent at the temperature of 50ºc for 15 minutes was considered to be suitable for the vatting process.

6.2 Preparation of dye bat for dyeing

The reducing agent and calcium hydroxide were added to the bath which remove the oxygen from water and left for 5 to 10 min than the leuco indigo was introduced to bath for dyeing process.

Table 7- dye bath recipe

Recipe	
Liquor ratio	1:30ml
Reducing agent	10ml
Calcium hydroxide	0.5g
Temperature:	50ºc
Duration	30 min

The bath was prepared at liquor ratio 1:30 ml. 0.5g of calcium hydroxide, 10ml of reducing agent was taken in a bath and left for 5 to 10 min. Than leuco indigo was added with the dye bath and the dyeing process was carried out at the temperature of 50ºc for 30 minutes. After dyeing the sample was rinsed in cold water.

6.3 Oxidization

The process of converting soluble dye in insoluble form where the material will turn into blue colour from yellowish green. The oxidization process was conducted by exposing the material to the air.

6.4 Soaping

For the removal of excessive dye from the material surface, the soaping process was conducted

Table 8-soaping recipe

Recipe	
Soap	1%
Temperature	60ºc
Duration	5min

The dyed material was subjected to soaping with 1% of soap at 60ºc for 5 minutes was used.

7. Testing of the samples

The dyed sample will be subjected to following tests.

7.1 colour measurement

Measuring spectral data provides a higher level of color accuracy than densitometry alone. Spectrophotometers measure reflected or transmitted light across a light spectrum. The resulting data creates a visual curve. Spectral measurements ensure that color is consistent across varying substrates and production processes. The relative colour yields for each dyed fabric sample with different natural reducing agent were assessed as k/s values using the kubelka-munk equation,

$k/s = (1-r)^2 / 2r$

Where,

R = spectral reflectance of the colored textile material,

k = dye absorption and

s = light scattering

7.2 colourfastness to washing - ISO 105 co1

Wash fastness of dye is influenced by the rate of diffusion of dye inside the fiber fastnesses to washing of the dyed sample were determined using launder-o-meter as per the ISO 105 co1 wash method. A specimen of textile in contact with specified adjacent fabric is mechanically agitated in soap solution, rinsed and dried. The change in colour of the specimen and the staining of the adjacent fabric area assessed using standard grey scales.

Table 9- colour fastness parameters for washing

Colour fastness parameters		
Grey scale reading	**Colour chane**	**Staining**
5	No colour change	No staining
4	Slight change	Slight staining
3	Moderate change	Moderate staining
2	Heavy change	Heavy staining
1	Severe change	Severe staining

7.3 Colourfastness to perspiration- ISO

Colour fastness to perspiration of the dyed sample was determined as per ISO method using perspire meter. Colourfastness of dye sample, against perspiration is determined by exposing the dye sample to the action of both acidic and alkaline reagents. A specimen of textile in contact with other textile adjacent material is wetted in simulated perspiration solution subjected to a fixed mechanical pressure and allowed to dry slowly at a slightly elevated temperature, the change in colour of the specimen of the dyed samples were assessed using ISO grey scale set

Table 10- colour fastness parameters for perspiration

Colour fastness parameters		
Grey scale reading	**Colour chane**	**Staining**
5	No colour change	No staining
4	Slight change	Slight staining
3	Moderate change	Moderate staining
2	Heavy change	Heavy staining
1	Severe change	Severe staining

7.4 Colourfastness to light - AATCC test method 16:3:2012

The purpose of colourfastness to light test is to determine how much the colour will fade when exposed to a known light source. It is an off line quality assurance system. Generally man wears the fabric and goes outside of the home for doing their job. In day sun light fall on the fabric surface. So it needs to know much protection ability of fabric toward sunlight. Sample was placed in a testing mask with part of the sample exposed and part covered as a control. The testing mask was loaded in a weather-o-meter holder and placed in a rack in the weather-o-meter for exposure. Sample was exposed to 10 hour & colour change of the sample is evaluated using grey scale.

Table 11- colour fastness parameters for light

Colour fastness parameters	
Grey scale reading	**Colour change**
5	Excellent
4	Good
3	Fair
2	Poor
1	Very poor

7.5 colourfastness to crocking

A colored sample is rubbed with white crock cloth under controlled condition. Coloured transfer to the white test cloth is assessed by compassion with grey scale for staining and grade is assigned.

Table 12- colour fastness parameters for crocking

Colour fastness parameters	
Grey scale reading	**Staining**
5	No staining
4	Slight staining
3	Moderate staining
2	Heavy staining
1	Severe staining

Results and discussions

Table 13-image and sample code of the dyed samples with natural reducing agents

Sl. No.	**Sample code**	**Reducing agent**	**Pantone colour**	**Pantone Shade Code**
1	A	Banana		283 c
2	B	Banana peel		291 c
3	C	Mango		290 c
4	D	Mango peel		2905 c

Sl. No.	Sample code	Reducing agent	Pantone colour	Pantone Shade Code
5	E	Dates		284 c
6	F	Grapes		2975 c
7	G	Green apple		291 c
8	H	Green apple peel		292 c
9	I	Figs		2915 c

The necessary components in indigo dyeing are:

- Conversion of indigotin to leuco compound called indigo white the indigo releases its oxygen to bond with the free hydrogen released by the reducing agent. This process is known as the reduction of the indigo or vatting process. For this process 25ml of natural extracted reducing agent were used.
- Addition of an alkaline substance permits the indigo to become soluble in water. For this process 1g of calcium hydroxide was used.
- The vatting process was conducted at the temperature of 50ºc for 15 minutes with 5% of dye 25ml of reducing agent and 1g of calcium hydroxide
- The dye bath was prepared by 1:30 ml of liquor ratio, 10ml of reducing agent and 0.5g of calcium hydroxide. And the leuco indigo was added to the bath.
- The dyeing process was conducted at 50ºc for 30 minutes.
- The oxidization process was conducted by exposing the material to the air.
- The dyed material was subjected to soaping with 1% of soap at 60ºc for 5 minutes for the removal of excessive dye from the material surface

- **Colour measurement**

Results for the sample dyed with indigo dye by using natural reducing agents are summarized below:

Table 14- k/s value of dyed sample

Sl. No.	Sample Code	K/s	L*	A*	B*	C	H
1	A	0.93	64.633	-3.811	-12.726	13.286	253.331
2	B	0.606	72.222	-3.672	-6.915	7.83	242.03
3	C	0.431	75.146	-3.671	-10.914	11.515	251.407
4	D	0.689	69.321	-4.222	-12.787	13.466	251.729
5	E	0.768	69.479	-4.543	-5.133	6.845	228.488
6	F	0.42	77.537	-3.381	-6.69	7.495	243.19
7	G	0.586	67.166	-4.932	-12.074	13.042	247.782
8	H	0.732	69.166	-4.226	-6.219	7.581	235.803
9	I	0.698	64.694	-4.122	-12.075	12.759	251.15

The results obtained from the research work are tabulated in the table 14, in which

Sample A which was dyed using banana as reducing agent exhibited high colour absorption (k/s) values as compared to other sample which was dyed with other fruits.

Fastness properties of dyed samples

A fabric ability to retain its original colour is one of the most important properties of the textile product. Colour fastness is a resisting property of the textile material. To resist the colour loosing or reducing from the textile material surface during different mechanical, physical and chemical treatment is called colour fastness. Colour fastness properties of the textile material especially fabric is very important for processing and usage. It indicates that the fabric was not dyed satisfactorily or the chemical and dyes selected for dyeing are not appropriate as compared to fabric criteria.

- **Colourfastness to washing**

Results for the sample dyed with indigo dye by using natural reducing agents are summarized below

Table 15- colourfastness to washing

Sl. No.	Sample Code	Change in the colour	Stain on the adjacent fabric
1	A	4	4
2	B	3.5	4
3	C	4	4
4	D	4	4
5	E	3	4
6	F	3	4
7	G	3.5	4
8	H	3	4
9	I	3	4

The results obtained from the research work are tabulated in the table 15, in which

Sample a which was dyed using banana as reducing agent, sample c which dyed using mango as reducing agent and sample d which was dyed using mango peel as reducing agent exhibited slight change in colour fastness to washing as compared to other sample which was dyed with other fruits. Whereas all the sample shows stain on the adjacent fabric grade 4 that can be graded as slight staining result.

- **Colourfastness to perspiration**

Results for the sample dyed with indigo dye by using natural reducing agents are summarized below

Table 16- colourfastness to perspiration (acidic)

Sl. No.	Sample Code	Change in the colour	Stain on the adjacent fabric
1	A	5	4
2	B	3.5	4
3	C	4	4
4	D	4	4
5	E	3.5	4
6	F	3	4
7	G	3.5	4
8	H	3	4
9	I	3	4

Table 17- colourfastness to perspiration (alkaline)

Sl. No.	Sample Code	Change in the colour	Stain on the adjacent fabric
1	A	5	4
2	B	3.5	4
3	C	3	4
4	D	4	4
5	E	3.5	4
6	F	4	4
7	G	3.5	4
8	H	3	4
9	I	3	4

The results obtained from the research work are tabulated in the table 16 and 17, in which sample a which was dyed using banana as reducing agent exhibited no change in colour fastness to perspiration (acidic and alkaline) as compared to other sample which was dyed with other fruits. Whereas all the sample shows stain on the adjacent fabric grade 4 that can be graded as slight staining result.

- **Colourfastness to light**

Results for the sample dyed with indigo dye by using natural reducing agents are summarized below

Table 18- colourfastness to light

Sl. No.	Sample Code	Change in the colour
1	A	4
2	B	3.5
3	C	3.5
4	D	4
5	E	3.5
6	F	4
7	G	2.5
8	H	2.5
9	I	2

The results obtained from the research work are tabulated in the table 18, in which sample a which was dyed using banana as reducing agent and sample d which was dyed using mango peel exhibited good colour fastness to light as compared to other sample which was dyed with other fruits.

- **Colourfastness to crocking**

Results for the sample dyed with indigo dye by using natural reducing agents are summarized below

Table 19- colourfastness to crocking

Sl. No.	Sample Code	Stain on the adjacent fabric	
		Wet	Dry
1	A	4	3.5
2	B	4	3
3	C	4.5	4
4	D	4	3.5
5	E	4.5	4
6	F	4.5	4
7	G	3.5	3.5
8	H	3.5	3.5
9	I	3	3

The results obtained from the research work are tabulated in the table 19, in which sample c which was dyed using mango as reducing agent, sample e which was dyed using dates as reducing agent sample f which was dyed using grapes as reducing agent exhibited slight staining to crocking as compared to other sample which was dyed with other fruits.

Summary

- **Colour measurement (k/s)**

Sample a dyed using reducing agent of banana were 0.930, sample b dyed using reducing agent of banana peel were 0.606, sample c dyed using reducing agent of mango were 0.431, sample d dyed using reducing agent of mango peel were 0.689, sample e dyed using reducing agent of dates were 0.768, sample f dyed using reducing agent of grapes were 0.420, sample g dyed using reducing agent of green apple were 0.586 , sample h dyed using reducing agent of green apple were 0.732 and sample I dyed using reducing agent of green apple peel were 0.998.

- **Colourfastness to washing**

Sample a dyed using reducing agent of banana, sample c dyed using reducing agent of mango, sample d dyed using reducing agent of mango peel, showed change in colour grade 4 that can be graded as slight change. Whereas sample b dyed using reducing agent of banana peel, sample g dyed using reducing agent of green apple, showed change in colour grade 3.5, and sample e dyed using reducing agent of dates, sample f dyed using reducing agent of grapes, sample h dyed using reducing agent of green apple, sample I dyed using reducing agent of green apple peel showed change in colour grade 3 that can be graded as moderate change.

- **Colourfastness to perspiration (acidic)**

Sample a dyed using reducing agent of banana, showed change in colour grade 5 that can be graded as no colour change, and sample d dyed using reducing agent of mango peel, sample c dyed using reducing agent of mango, showed change in colour grade 4 that can be graded as slight change. Whereas sample b dyed using reducing agent of banana peel, sample e dyed using reducing agent of dates, sample g dyed using reducing agent of green apple showed change in colour grade 3.5, and sample f dyed using reducing agent of grapes, sample h dyed using reducing agent of green apple, sample I dyed using reducing agent of green apple peel showed change in colour grade 3 that can be graded as moderate change.

- **Colourfastness to perspiration (alkaline)**

Sample a dyed using reducing agent of banana, showed change in colour grade 5 that can be graded as no colour change. Sample d dyed using reducing agent of mango peel, sample f dyed using reducing agent of grapes, showed change in colour grade 4 that can be graded as slight change. Whereas sample b dyed using reducing agent of banana peel, sample e dyed using reducing agent of dates, sample g dyed using reducing agent of green apple

showed change in colour grade 3.5, and sample c dyed using reducing agent of mango, sample h dyed using reducing agent of green apple, sample I dyed using reducing agent of green apple peel showed change in colour grade 3 that can be graded as moderate change.

- **Colourfastness to light**

Sample a dyed using reducing agent of banana, sample d dyed using reducing agent of mango peel, sample f dyed using reducing agent of grapes, showed change in colour grade 4 that can be graded as good, and sample b dyed using reducing agent of banana peel, sample c dyed using reducing agent of mango, sample e dyed using reducing agent of dates, showed change in colour grade 3.5, that can be graded as fair whereas sample g dyed using reducing agent of green apple sample h dyed using reducing agent of green apple, showed change in colour grade 2.5 and sample i dyed using reducing agent of green apple peel showed change in colour grade 2 that can be graded as poor.

- **Colourfastness to crocking(dry)**

Sample c dyed using reducing agent of mango, sample e dyed using reducing agent of dates, sample f dyed using reducing agent of grapes showed stain on the adjacent fabric grade 4.5 and sample a dyed using reducing agent of banana, sample b dyed using reducing agent of banana peel, sample d dyed using reducing agent of mango peel, showed stain on the adjacent fabric grade 4 that can be graded as good where as sample g dyed using reducing agent of green apple , sample h dyed using reducing agent of green apple showed change in colour grade 3.5 and sample i dyed using reducing agent of green apple peel showed stain on the adjacent fabric (dry) grade 3 that is graded as fair.

- **Colourfastness to crocking(wet)**

Sample c dyed using reducing agent of mango, sample e dyed using reducing agent of dates, and sample f dyed using reducing agent of grapes showed stain on the adjacent fabric grade 4 that can be graded as good. Whereas sample a dyed using reducing agent of banana, sample d dyed using reducing agent of mango peel, sample g dyed using reducing agent of green apple, sample h dyed using reducing agent of green apple showed stain on the adjacent fabric grade 3.5 and sample b dyed using reducing agent of banana peel, sample i dyed using reducing agent of green apple peel showed change in showed stain on the adjacent fabric grade 3 that can be graded as fair.

Conclusion

Indigo is a so-called vat dye, which means that it needs to be reduced to its water soluble leuco-form before dyeing. The reduced form is absorbed into the fibres and when oxidized back to its blue form it stays within the fibre. Earlier the reduction and dyeing was done with fermentation. Nowadays, the most of the reduction has been done chemically by sodium dithionite. It is considered environmentally unfavourable since it produces sulphite, sulphate, thiosulphate and toxic sulphides as degradation products, which then contaminate the waste waters from the dyeing plants.for this, natural reducing agents from fruits such as dates, banana, mango, grapes, green apple, figs and their peel were used for dyeing cotton using indigo dye. The analysis was based on three factor are temperature, sodium calcium hydroxide and natural reducing agent concentrations. Natural reducing agent has best reducing activity when extracted at the temperature of 80°c for 1 hr.25 ml of reducing agent with 1g of calcium hydroxide processed at 50^{o} c for 10 min gave best results in vatting process. Whereas 10 ml of reducing agent with 0.5 g of calcium hydroxide dyed at 50^{o} c for 30 min gave best results in dyeing process. the reducing agent extracted from banana gave best as compare to other fruits in term of k/s value which was 0.930 and the colourfastness properties was also good as compared to other reducing agents. Hence, this study will be helpful useful in dyeing cotton fabric using selected natural reducing agent.

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