Oriental Journal of Chemistry Vol. **25**(1), 77-84 (2009)

A systematic and scientific approach to the extraction and dyeing with a natural dye on silk-annatto seeds dye

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(Received: October 02, 2008; Accepted: November 08, 2008)

ABSTRACT

Due to environmental awareness created among masses worldwide by environmentalists and green peace activists and hazards of certain synthetic dyes, people have realized the potential of natural dyes and tried to use them as an alternative to synthetic dyes. In this discussion efforts are taken to dye silk fabric using Annatto seed extract. The extract was prepared in aqueous, alkaline and alcoholic media. The method of dyeing silk with the seeds was standardized determining the optimum dyeing conditions namely dye material concentration, dye material extraction time, mordant concentration and mordanting method. The dyed silk fabrics are subjected to tests such as tensile strength, abrasion, crocking and light fastness tests. The antimicrobial property of the extracted dye samples is studied by using Gram positive and Gram negative culture. Annatto seeds dye on silk fabrics provides favorable results.

Key words: Natural dye, silk-annatto seeds, bacteria.

INTRODUCTION

Awareness and concern for environmental issues has led to extensive research for use of natural dyes for silk. A great deal of emphasis on the screening of newer natural coloring materials for the industrial application has gained impetus. This work was taken up for easy availability of natural resources.

Natural dyes – A Boon to Humanity

Natural dyes are being considered as a possible substitute to synthetic azo dyes. As a result of increased environmental awareness, natural dyes are being preferred over the synthetic dyes because they exhibit better biodegradability and compatibility with the environment. In addition, the dyes obtained from natural sources do not possess the danger of allergic reactions and are non-toxic in nature.

According to the literature survey, the amount of research efforts devoted to natural dyes is negligible. Concern for environment and hunger for eco friendly products and processes have compelled scientists and technocrats to develop products and process, which are eco friendly. Naturally colored silk is therefore seen as a substitute raw material ensuring ornamentation of textiles without use of highly toxic dyes and chemicals and hence in this direction annatto seeds were selected as a natural dye source for silk fabric.

Methodology

Choice of the substrate

Silk has been considered as a prominently superior fabric among highly priced textile fabrics, since it combines strength and durability with beauty, cleanliness, lightness and lusture[1]. Sericulture is widely practiced in India. India has unique distinction of producing all the commercially known varieties of silk namely Mulberry, Tussar, Eri and Muga. The abundant use of silk in various forms of textiles despite the tough competition from synthetic fibers reflects its supremacy[2]. Today in the era of natural and environmental friendly products is just making its beginning the sericulture industry can hope for a good future. India ranks second in the world next to China in the production of silk[3].

Dye source

Annatto (*Bixa orellana*) seeds were selected as a natural dye source to color silk fabric. Annatto seeds were collected from different places in and around Coimbatore district. A brief profile about the plant is given below.

Plant profile

Annatto called Sindurapushpi in Tamil, Karchchandha in Sanskrit, Sinduri in Bengali, Kunkum in Kannada, and Annatto in English. Annatto grows to a height of 2-5 meters belongs to the family *Bixaceae*. In India it is well distributed in Karnataka, Andhra Pradesh, Assam, Tamil Nadu, Orissa and Chhattisgarh and also reported to be cultivated commercially since last 5 years.

Important uses of Annatto

In Indian system of medicines especially in Ayurved, the leaves of Sinduri are reported as blood purifier. It is very effective for gonorrhea. Annatto dye is used mostly in dairy industry such as in coloring butter, cheese, ghee, chocolate, ice cream, in medicines an in making boot polishes. The color is also used in making bindi and kunkum. The waste annatto seeds after extraction of color have been reported suitable for animal feed.

Annatto dye

Annatto dye is basically a red orange pigment known as Bixin, extracted from the seed coat of Sinduri, contains carotenoid of various types out of which cisbixin alone accounts for 82% chemistry and performance of annatto colors is essentially of the bixin. Bixin is highly unsaturated compound. The bixin dissolves in vegetable oil, undergoes isomerisation and degradation reactions when heated.

Composition of Annatto seeds (Dukes Photochemical Database)

S. No	Constituents	g/100 g seeds
1.	Protein	around 12
2	Pentosans	11.35-14.97
3.	Total carbohydrates	39.91-47.90
4.	Ash	5.44-6.92
5.	Pectin	0.23-0.55
6.	Tannins	0.33-0.91
7.	Total Caratenoids	1.21-2.30

Standardization of the medium for dye extraction

Annatto dye was extracted in alkaline, alcoholic and in aqueous medium. The flow chart for the extraction of the dye in these three medium is given below.

Preparation of the fabric for dyeing

Bleaching

The silk fabric should be free from impurities before dyeing. Pretreatment methods such as degumming and bleaching were done to the silk fabric, the flow chart of which is given below.

Selection of the mordant

Dyes cannot combine directly with the substrate. A mordant is required to make the colors hold. The natural mordant, pre-mordant used for the present study was myrobalan.

The myrobalan fruits were crushed to powder, soaked in sufficient water and kept overnight. It is then filtered and the pretreated silk fabric was soaked in this solution for overnight.

Combinations of metal ions were used as post-mordants. Natural dyes require chemicals in the form of metal salts to produce an affinity between the fabric and the pigment and these chemicals are known as mordants[4]. The combination of metal salts used for the present investigations are $K_2Cr_2O_7$ + $CuSO_4$, $FeSO_4$ + $CuSO_4$, Alum + $FeSO_4$, $CuSO_4$ + Alum, $K_2Cr_2O_7$ + $FeSO_4$. The combination did not exceed 5g/100 ml[5].

The U.V and I.R spectrums for these solutions are recorded.

Optimizing the pH of the dye

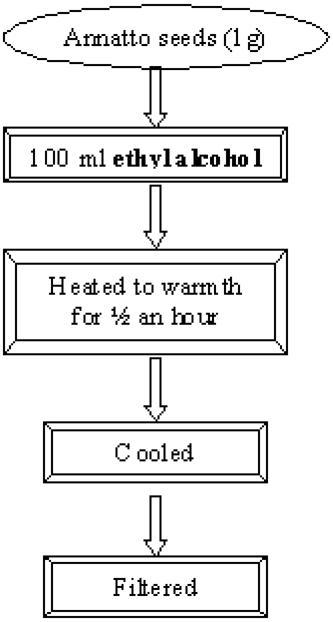
Annatto dye was extracted in alkaline, alcoholic and aqueous medium. The dyed samples were shown to a panel of judges for evaluation on the basis of brightness and darkness of the color. The sample obtaining rank one was selected as the best sample and the extraction pH used for that sample was selected as the optimum pH for extraction of dye. By changing the pH of this alkaline extract the color change was noted.

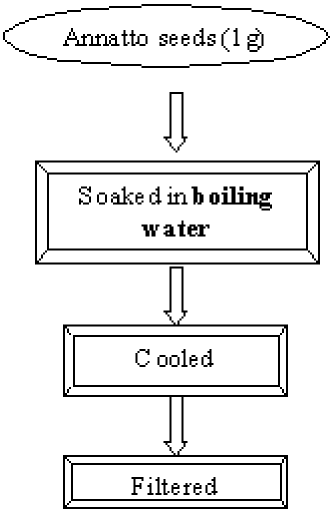
Optimizing the dye extraction time

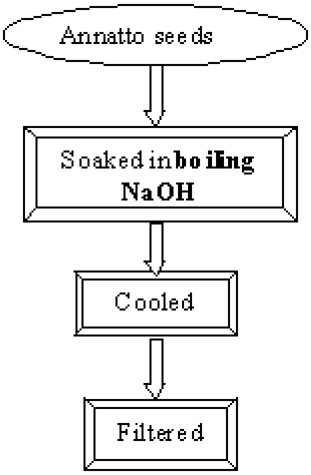
Extraction of dyes was carried out at optimum pH for 30 minutes and 60 minutes and silk samples were dyed in these extracts. The dyed samples were shown to a panel of judges for evaluation on the basis of brightness and darkness of the color. The sample obtaining rank one was selected as the best sample and based on that further studies were carried out.

Optimizing the temperature for dye extraction

The optimum temperature for the extraction of the dye was selected by extracting the dye at 30°







C and 60°C. The dyed samples were shown to a panel of judges for evaluation on the basis of brightness and darkness of the color. The sample obtaining rank one was selected as the best sample and based on that further studies were carried out[6].

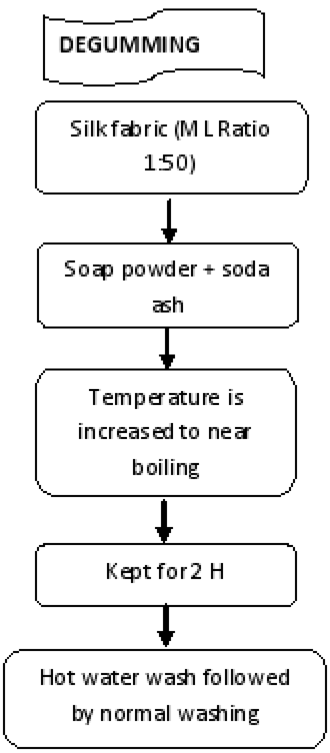
Evaluation of fastness properties for dyed and undyed fabric

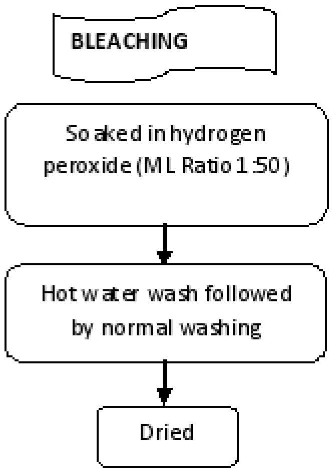
American Association of Textile Colorants and Chemists (AATCC,1995) has established standard terminology for rating color fastness properties of fabric through different test methods and also evaluating color staining and transfer in fabrics. The following tests were conducted to assess fastness to sunlight, washing, wet and dry crocking, wet and dry pressing. Laboratory tests such as abrasion resistance, breaking strength and elongation tests were also done[7].

Antimicrobial study

Microbes are very small organisms, which require a certain medium to grow, such as moisture, temperature, dirt and respective surfaces. Today in the era of eco friendly operation, it had become very important for human beings also to live in a world of hygiene and freshness. The major hindrance that comes in their way is microorganisms, which are the causative agents of deterioration, staining and odour. Apart from this effect, microbes cause harm to human being by transmitting diseases and infections. So it becomes very important to study the antimicrobial property of the dye source. So in the present investigation an attempt has been made to explore the antimicrobial property of the dye extract.

AATCC Test Method 100-1999 was used to determine the antimicrobial activity of the dye sample. *Escherichia coli* (*E. coli*), a gram-negative bacterium, were selected due to its popularity as a test organism and its resistance to common antimicrobial agents. *Staphylococcus aureus* (*S. aureus*), a pathogenic gram-positive bacterium, was used because it was the major cause of cross-infection in hospitals and it is the most frequently evaluated species. The gram negative *E. coli* and the gram-positive S. aureus cultures were left to grow in specific conditions. 20 ml of sterile nutrient agar media was added to each Petri dish and 2 ml of 24 h broth culture of bacteria was then added to the





respective plates. The extracted dye solution was dropped for about 5mm on the prepared base medium which is in a jell form. The Petri plates were incubated at 37°C and zones of inhibitions were measured excluding the diameter of the dye solution (5 mm)[8].

RESULTS AND DISCUSSIONS

Choice of the Medium for Dye Extraction

The effective extraction depends on the medium and several other parameters. In the present study efforts have been taken to identify a suitable solvent for extraction. The dye extract were prepared in alcohol, aqueous and alkaline media. Alcohol facilitates maximum extraction of dye in the given time. But it is not cost effective, eco friendly and due to its evaporating and bumping nature during heating aqueous extract was preferred over the other extract. But the color obtained in aqueous medium was not appreciable. The alkali extracts furnished better concentration of dye solution than aqueous medium. Therefore alkaline extract has been selected as the extraction medium.

Effect of mordant combination on color uptake

Natural dyes are commonly applied with mordant, as they are known to promote the binding of the dyes to fabric by forming a chemical bridge between dye and fiber, improving the staining ability of the dye along with increasing fastness property. Myrobalan pretreatment was applied to the silk fabric[9]. The silk fabrics were then dyed using an open dye bath. After dyeing, the samples were removed from the bath, rinsed to remove excess dye particles and subjected to post mordanting with various combinations namely $CuSO_4$ + Alum, $FeSO_4$ + $CuSO_4$, Alum + $FeSO_4$, $K_2Cr_2O_7$ + $CuSO_4$, $K_2Cr_2O_7$ + $FeSO_4$[10]. The color obtained were analyzed through U.V spectroscopy, the λ max and absorbance were given in Table 1.

Table 1: Effect of mordant combination on the color uptake

S. No.	Mordant	Percentage Combination	Absorbance wave length at (970 nm)
1.	Dye	-	0.246
2.	$CuSO_4$ + Alum	3 : 1	0.163
3.	$FeSO_4$ + $CuSO_4$	1 : 1	0.391
4.	Alum + $FeSO_4$	3 : 1	0.917
5.	$K_2Cr_2O_7$ + $CuSO_4$	1 : 3	0.591
6.	$K_2Cr_2O_7$ + $FeSO_4$	3 : 1	0.325

Table 2: Light fastness ratings for silk fabric

Days exposed	Grey scale values
7	3/4
6	3/4
5	4
4	4
3	4
2	4/5
1	4/5

Table 3: Color fastness ratings for silk fabric

	Rubbing	Washing	Crocking		Pressing	
			Dry	Wet	Dry	Wet
*CC	2/3	2/3	5	5	½	½
**CS	2	½	3	3	3	3/4

* - Colour Change ** - Colour Stain

From the table it is observed that the depth of color increased with all combinations of mordants except $CuSO_4$+ alum. $CuSO_4$+ alum combination has the lowest depth of absorbance. Similar results were substantiated with I.R spectrums.

Optimum pH of the dye extract

The pH of 1% alkaline extract was found to be 8. By changing the pH there was no observable color change.

Optimum time and temperature for dye extraction

1% alkaline dye solution has been used for dyeing silk. The temperature of the dye bath was maintained at 60° C for 60 minutes which furnished better results.

Fastness properties of dyed silk fabric

Color fastness to light

Table 2 gives the data for color fastness to light for silk fabric dyed with annatto extract. The dyed silk fabric shows a good fastness property. This may be due to the moderable fixation of this natural dye with the protein component of the silk polymer.

Wash and rub fastness

The dyed samples were tested for their wash fastness by washing with 0.5 % soap solution and also tested for rubbing fastness. The samples were resulted in loss of color in washing. It was rated 2/3 for wash and rub fastness in the grey scale in terms of CC and 1/2, 2 in terms of CS respectively.

Crocking fastness

For the wet and dry silk fabric crocking fastness were tested. The results of which is given in table 3. The crocking fastness of the silk fabric is measured both under wet and dry conditions. From the table it is clear that the silk fabric does not show any change in CC and CS values in wet and dry conditions. Thus the fabric showed a very good crocking fastness.

Color fastness to pressing

The dyed silk fabric was subjected to pressing under hot iron. There was no change in the grey scale values for both wet and dry pressing in terms of CC, but CS values for wet pressing was found to be 3/4 and 3 for dry pressing.

Table 4: Abrasion and Tensile strength on silk fabric

Name of the test			Silk fabric
Abrasion test	dyed	Initial wt(g)	0.077
		Final wt(g)	0.072
Tensile strength	dyed	Warp(inch)	1.94
		Weft(inch)	1.78
	undyed	Warp(inch)	2.0
		Weft(inch)	1.05

Table 5: Anti microbial assessment of the dye extract

S. No	Dye Extract	Number of Bacterial Counts	
		Gram Positive	Gram Negative
1	Aqueous	845	826
2	Alkaline	614	528
3	Alcoholic	686	644

Table 3 lists an overview of the fastness grades of annatto dye material on silk at optimum dyeing conditions with pre-mordanting methods.

The results of the dyed and undyed silk fabric on color fastness are pictorially represented in figure 1.

Abrasion test

The dyed silk fabric during abrasion test showed an average weight loss of 0.005 g which is negligible. Thus the fabric proved to have good tensile strength after dyeing.

Comparison of tensile strength of dyed and undyed fabric

The data in Table 4 clearly indicate that in warp wise there is not much difference, before and after dyeing. The dyed fabric showed better values weft wise than undyed fabric. It is also clear from the graph (Fig 2).

Anti microbial study

Anti microbial assessment for alcoholic, aqueous and alkaline extract were done. Table 5 clearly indicates the anti microbial study of the dye extract. From the recorded bacterial counts it is seen that the performance of alkaline dye extract was found to be good.

Chemistry behind Dyeing

Ion exchange

The non-specific force is very important process of acid dyeing of anionic dyes on fibers containing amino group i.e., wool, silk and nylon also with basic dyes on these fibers, on which they are not much used. The broad principles of the ion-exchange effects in the dyeing of silk have been described.

In water the amine and carboxyl groups of the protein fiber (W) can be considered as Zwitterionic pairs and entering hydroxyl ions neutralize the amino groups.

$-OOC - W - NH_3^+ \rightarrow {}^-OOC - W - NH_2\}M^+$

M = Metal ions

In a similar way the acidic dyes neutralize the carboxyl group

$-OOC - W - NH_3^+ \rightarrow HOOC - W - {}^+NH_3\}X^-$

X = Halogens

Bixin is an aromatic compound with extended π electron system and a water solubilising group, -OH. The Bixin being acidic dye it attaches to the fiber by neutralizing the basic amino group and hence the fiber dye bond is strengthened.

Bixa orellana seeds contain Bixin as a major constituent.

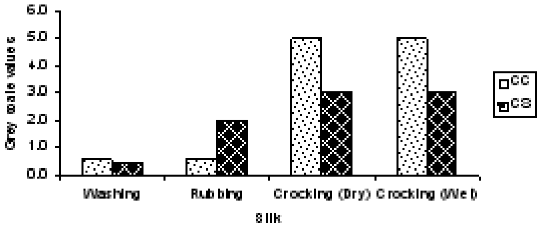


Fig. 1: Colour fastness ratings for silk fabric

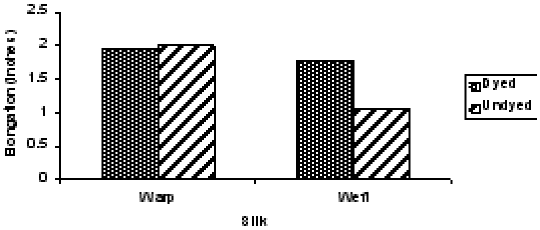
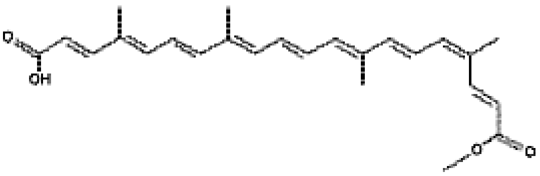


Fig. 2: Tensile strength on silk fabric

Bixin



Dye fiber bond

The dye molecules are in a state of constant motion in the dye bath. When, a fiber is introduced in water from the bath immediately passes into the pores of the fiber and an interface a very extensive internal surface is thus formed between water and the walls of the pores. Water molecules are small and travel rapidly in most cases filling the pores in a few seconds. Dye molecules are larger and take longer to push their way in, but their constant motion eventually carries them into all the larger pores. It carries them particularly to the walls of the pores, where at the fiber - water interface they remain closer together than in the bulk of the internal solution. The process is greatly assisted if the dye is ionic and fiber carries an opposite charge. It is also assisted by the Vander Waals forces, which are stronger with large than with small dye molecules and which tend to allign the dye molecules parallel to and in contact with molecular chains in the fiber walls. A further action then comes into force. Because of the constriction of the smaller pores, the amount of dye in the interface layer adds significantly to the total concentration of dye in the pores. The result is that most of the dye molecules then associate into larger units.

In hydrophobic fibers, placed in a bath containing disperse dye, the pores are in most cases extensive than in the hydrophobic fibers. Most of the dyeing occurs because the dye molecules possess strong affinity for the fiber polymer, arising from the formation of polar - mainly hydrogen bonds. Consequently they are able to push their way into the fiber substance, forcing the polymer chains apart. Some association of dye may also occur in this process.

CONCLUSION

Natural extract from annatto seeds in 1% NaOH can be successfully applied on silk. Silk samples post mordanted with different composition of copper sulphate, alum, potassium dichromate, ferrous sulphate gave better results. The fastness properties were found to be satisfactory. The annatto dyed silk fabric showed a very good light fastness. The dye stuff produced good wash fastness and moderate rubbing fastness in the rating scale. The samples showed a very good crocking fastness in dry condition scoring 4/5 in grey scale value and good crocking fastness in wet condition also. The fabric proved to withstand abrasion. On comparison with the undyed sample the dyed sample was found to retain its tensile strength. Thus annatto dye is a valuable natural colorant which is easily extractable in substantial amount. It possesses potential to be used as a colorant for silk for the development of orange to yellow orange color.

ACKNOWLEDGEMENTS

The authors thank the Department of Chemistry and Department of Textiles, of Avinashilingam University for Women, Coimbatore for providing the necessary facilities to carry out the work.

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