

# Asian Resonance

## Application of Chitosan to Impart Antibacterial Property to Annatto Dyed Fabric

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#### Abstract

The use of eco friendly, biodegradable, natural raw materials has been increased with increase in awareness concerned to sustainability. Annatto is well known plant for its medicinal and coloring properties. In present investigation natural dye annatto has been applied to cotton with and without harda and alum mordant. The effect of mordant and mordanting method (pre, meta and post) on colour co-ordinates and fastness properties has been evaluated. Further the effect of pre and post treatment of chitosan on annatto dyeing, fastness properties of dyed samples, CRA, bending length, tearing strength as well as antibacterial properties of fabric were evaluated. Finding of the results show that application of pre and post chitosan treatment improves dye fastness, CRA, bending length as well as antibacterial properties of annatto dyed fabric.

**Keywords:** Annatto, Chitosan, Harda, Alum, Antibacterial property, Colour co-ordinates.

#### Introduction

Usage of large amount of water and synthetic dyestuff in textile industry creates pollution by discharging huge effluent which is mostly toxic in nature<sup>1</sup>. Toxicity is the main cause of human health problems which creates more pressure on the textile processing and dyeing industries<sup>2</sup> to reduce toxicity in effluent. Although, the growing awareness about sustainability and demand for eco friendly products plays crucial role in promoting natural dyes<sup>3</sup> and natural finishing agents in textile wet processing industries.

#### Aim and Scope of the Study

The main aim of the present study was to dye cotton fabric in orange shade using annatto by applying biopolymer chitosan to impart antibacterial activity to the fabric. Findings show promising results and can be industrialized.

#### Review of Literature

Annatto (*Bixa orellana*) is a small tree whose seeds and pulp are source of a dye and has been used all over the world as body paint and food colourant. Its pulp is rich in tannin but contains a mixture of colourants of carotenoid group<sup>4</sup>. The colourants in annatto are a mixture of bixin, the monomethyl ester of a dicarboxylic carotenoid compound and norbixin<sup>5</sup>, the dicarboxylic derivative of the same carotenoid as in bixin<sup>6</sup>. Annatto is a potential source of dye<sup>7</sup> and can be used directly in the colouration of wool, silk and cotton to give an orange-red shade. In the light of less toxic properties annatto may emerge as a good textile dye<sup>8</sup>. Gulrajani et al<sup>9</sup> studied dyeing of annatto on synthetic fibre such as nylon and polyester. It is observed that both the fibres have good affinity for this dye. Prabhavathi et al<sup>10</sup> also reported that annatto is an orange dye source which can be used efficiently to dye cotton fabric with the help of mordants. Natural extract from annatto can be successfully applied on silk. The annatto dyed silk fabric showed a very good light as well as good wash and moderate rubbing fastness in the rating scale<sup>11</sup>. Dyeing of cotton with natural dyes needs mordant for fixation. Harda which is obtained from myrobalan dried fruit is rich in tannin content<sup>12</sup> and acts as mordant<sup>13, 14</sup>.

Natural fibres are prone to moth and mildew attack. Therefore many chemicals have been used as antimicrobial finishing agents on textiles. But most of them are not eco-friendly<sup>15</sup>. Chitosan is a natural product derived from chitin; Chitin is the second most abundant natural polysaccharide after cellulose on earth. Chitosan is the de-acetylated

product of chitin. Chitin and chitosan are biodegradable, biocompatible, non-toxic and their amino and two hydroxyl groups can be chemically modified to generate different novel derivatives<sup>16, 17</sup>. Growth of wide variety of bacteria, algae and fungi, showing broad spectra of antibacterial activity is inhibited by chitosan<sup>18</sup>. Chitosan interacts with the membrane to alter cell permeability, even to cause the leakage of the intracellular components<sup>19</sup>. The other mechanism involves the binding of chitosan with DNA to inhibit RNA synthesis<sup>20</sup>.

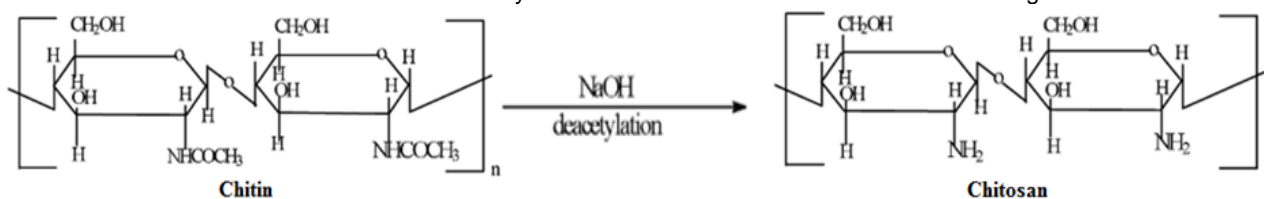
Chitosan is applied on cotton fabric with the help of various cross linking agents. Y. Chung *et al*<sup>21</sup> studied cross linking effect of various agents like DMDHEU, BTCA, citric acid and glutaraldehyde along with chitosan. In a study, it has been shown that cross linking by glutaraldehyde with cotton fabrics enhances the reduction of bacteria.

In present study cotton fabric was dyed using annatto dye. The natural dye annatto has been also applied to pre and post chitosan treated cotton fabric. Chitosan was applied to cotton fabric with the help of cross linking agent glutaraldehyde to improve the

**Methods**

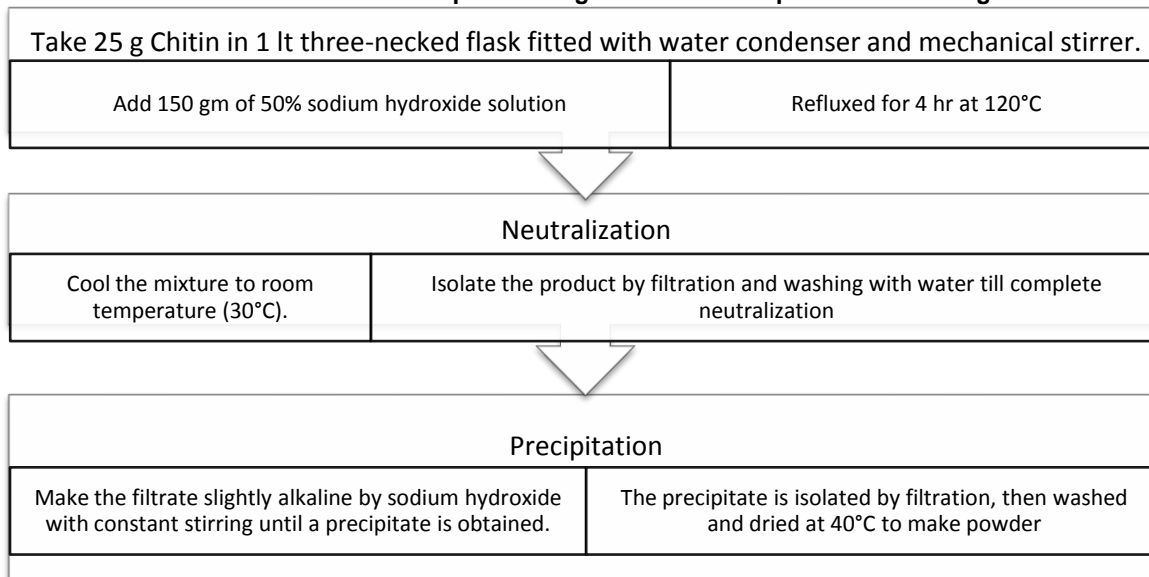
**Chitosan preparation**

The Chemical Reaction of Deacetylation of Chitin to Form Chitosan is Shown in Figure 1.



**Fig 1 Deacetylation Reaction of Chitin in Presence of NaOH to form Chitosan**

Chitosan from Chitin was Prepared using the Process Steps as shown in Figure 2



**Fig 2 Preparation of Chitosan from Chitin**

physical properties and to impart antibacterial property to cotton fabric. The colour co-ordinates, fastness properties of the dyed samples were evaluated thoroughly. The physical properties such as tearing strength, CRA and bending length of pre and post chitosan treated cotton samples were also determined.

**Experimental**

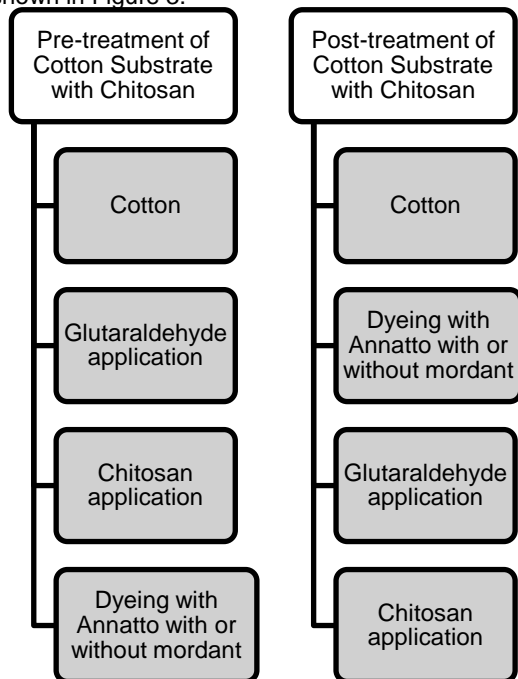
**Materials**

Plain woven bleached cotton fabric of 89 GSM, 90 EPI and 54 PPI was used for the present study. Before further dyeing and finishing operations fabric was definished and mild scoured. Natural dye annatto and natural mordant harda were procured from M/s Alps Industries Ltd. India. Chitin was purchased from Loba Chemie Pvt. Ltd and used for chitosan preparation. Glutaraldehyde (25%) was purchased from Central Drug House (Pvt.) Ltd, New Delhi and used as a cross linking agent for application of chitosan. Alum was also used as chemical mordant. The other chemicals used in the research work were of laboratory reagent grade.

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## Application of Chitosan on Cotton Fabric

In pre-treatment procedure, chitosan was first applied to fabric samples and then dyeing was carried out. While in post-treatment the samples were first dyed and after the completion of dyeing, chitosan was applied to cotton fabric. Flow chart of processes is shown in Figure 3.



**Fig 3 Flow Chart of Pre and Post Chitosan Treatment on Annatto Dyed Cotton**

Chitosan was applied to fabric samples with the help of glutaraldehyde as binding agent. Fabric samples were first treated with 10 gpl glutaraldehyde solution for 15 minutes and then dried at a temperature of 85°C for 5 minutes. After the drying operation, the fabric samples were treated with different chitosan solution (1, 5, 7.5 and 10 gpl) for 15 minutes. Again, samples were dried at 85° C for 5 minutes and followed by curing at 150° C for 3 minutes.

### Dyeing

Dyeing was performed by exhaustion method in open bath beaker dyeing machine using the following recipe.

Dye (Annatto)	5, 10, 15 % owf
Mordant (Alum/ Harda)	10% owf
Mordanting Procedure	Pre, Meta, Post
MLR	1:40
pH of the dye bath	Neutral
Temperature of dyeing	85°C
Time of dyeing	1 hr

After dyeing samples were washed with 2 gpl non-ionic soap solution at 70°C for 10 minutes and then rinsed thoroughly with cold water and dried.

### Colour Strength

The colour difference DE, colour value (L\*,

a\*, b\*) of the different dyed samples were determined using Datacolour spectrophotometer and Datacolour software interfaced with the computer<sup>22</sup>. Illuminant D65, observer 10<sup>0</sup> and CIE 1976 were used. The instrument was standardized with a white tile. Hunter co-ordinates had been measured on the instrument of various samples i.e. L\*, a\*, b\*. Where,

1. L signifies lightness (L+ -- more lighter, L- -- more darker)
2. a signifies redder or greener (a+ -- redder, a- -- greener)
3. b signifies bluer or yellower (b+ -- yellower, b- -- bluer)

Colour strength expressed as K/S was measured according to a previously reported method<sup>23</sup> by the light reflectance technique, and the relative colour strength was calculated by applying the Kubelka Munk equation:

$$\text{Colour strength (K/S)} = \frac{(1 - R)^2}{2R}$$

where, R is the decimal fraction of the reflectance of the coloured fabric,

K is the absorption coefficient and S is the scattering coefficient.

### Colour Fastness

The various fastness properties such as colour, wash, rubbing and light fastness were checked using IS: 3361- 1979, Test III, IS: 766- 1988 and IS: 2485-1985 test methods respectively.

### Testing Physical Properties

The change in physical properties of the fabric i.e. crease recovery angle, tearing strength and bending length were evaluated. Crease recovery angle of the fabrics was calculated using IS: 4681- 1968 test method on Shirley crease recovery tester. Tearing Strength of the fabrics was calculated using IS: 6489 – 1971 test method based on the Elmondrof tear tester. Bending length was calculated using IS: 6490-1971 test method on Shirley stiffness tester<sup>24</sup>.

### Antimicrobial Testing

Antimicrobial activity of the samples was evaluated qualitatively by AATCC 90 agar plate method. Staphylococcus aureus, a gram positive bacterium was used in the testing<sup>25-27</sup>.

### Results and Discussions

#### Annatto Dyed Samples

The fabric samples were dyed with annatto with and without harda and alum mordant. The L\*, a\*, b\*, K/S values of dyed cotton samples obtained are given in Table 1. As we increase the amount of dye the saturation increases. It is can be observed from table that 'a' and 'b' values are positive in all the pre, meta and post mordanted fabrics, that means they lie in the red-yellow co-ordinate. Samples without mordant gives poor to moderate fastness and it can be observed that samples dyed with the help of mordant improve the fastness property slightly. It can be analyzed that natural mordant harda as well as chemical mordant alum give satisfactory results.

**Table - 1**  
**Colour Strength and Colour Fastness of Annatto Dyed Samples in Combination with Harda and Alum as Mordant**

Mordant	Mordant %	Dyes %	K/S	L	a	b	Light Fastness	Rubbing Fastness	Washing Fastness
None	0	5	40.53	77.89	17.89	25.45	3	2/3	2
	0	10	39.39	74.77	20.96	29.89	3/4	3	2/3
	0	15	39.03	76.78	21.18	32.40	3/4	3	2/3
Harda Pre Mordanting	10	5	34.17	69.88	17.76	34.89	4	3/4	3
	10	10	33.97	68.76	19.40	35.32	4	3/4	3
	10	15	33.74	67.65	20.53	35.89	4	3/4	3
Harda Meta Mordanting	10	5	31.02	66.07	16.25	34.12	4	3	3
	10	10	30.42	63.99	19.08	38.25	4	3/4	3
	10	15	29.97	63.13	20.91	39.60	4	3/4	3
Harda Post Mordanting	10	5	32.38	68.04	15.93	30.79	4	3	3
	10	10	31.65	66.49	18.92	34.45	4	3/4	3
	10	15	31.13	66.40	20.41	34.92	4	3/4	3
Alum Pre Mordanting	10	5	40.89	78.63	18.61	26.76	3/4	3	3
	10	10	40.42	76.02	21.80	30.82	3/4	3/4	3
	10	15	40.36	75.25	21.95	34.81	3/4	3/4	3
Alum Meta Mordanting	10	5	40.37	79.74	16.32	23.78	3/4	3	3
	10	10	39.79	78.22	17.43	25.76	3/4	3/4	3
	10	15	39.61	75.98	18.77	27.56	3/4	3/4	3
Alum Post Mordanting	10	5	40.85	77.85	19.95	29.59	3/4	3	3
	10	10	40.66	74.85	23.92	31.81	3/4	3/4	3
	10	15	39.48	72.80	24.88	33.56	3/4	3/4	3

**Annatto dyed and chitosan finished samples**

The colour co-ordinates and colour fastness properties of pre and post chitosan treated annatto dyed cotton samples are shown in Table 2. The dyeing of samples was carried out by meta mordanting method taking 10% owf dye with and without mordant (10%). It can be analysed that even

after pre and post chitosan treatment the samples show positive 'a' and 'b' values so they lie in the red-yellow co-ordinate.

It can be also observed that with increase in chitosan concentration in pre as well as post treatment, there is slight improvement in fastness properties of dyed samples.

**Table - 2**  
**Colour Co Ordinates and Colour Fastness Rating of Annatto Dyed (10% Owf) Samples Treated with Chitosan in Combination with Harda and Alum as Mordant**

Mordant	Chitosan %	Mordant %	L	a	b	Light Fastness	Rubbing Fastness	Washing Fastness	
--	0	0	74.77	21.18	32.40	3/4	3	2/3	
Chitosan Pretreated	1	0	77.61	18.27	29.24	4	3/4	3	
	5	0	73.87	20.76	30.38	4	3/4	3	
	7.5	0	73.70	22.53	32.71	4	3/4	3	
	10	0	73.03	21.47	32.16	4	3/4	3/4	
Chitosan Post treated	1	0	68.02	19.53	37.46	4	3/4	3	
	5	0	73.17	22.34	37.43	4	3/4	3	
	7.5	0	64.66	15.74	36.85	4	3/4	3	
	10	0	73.88	20.84	35.37	4	3/4	3/4	
Chitosan Pre Treated	Harda Meta Mordanted	0	10	63.99	19.08	38.25	4	3/4	3
		1	10	70.94	16.13	28.88	4	3/4	3
		5	10	65.59	15.42	28.14	4/5	3/4	3
		7.5	10	62.12	15.41	29.06	4/5	4	3/4
		10	10	64.12	14.31	25.75	4/5	4	4
	Alum Meta Mordanted	0	10	78.22	17.43	25.76	3/4	3/4	3
		1	10	77.29	17.71	33.44	4	3/4	3
		5	10	76.10	15.47	32.77	4/5	3/4	3
		7.5	10	75.26	16.55	35.45	4/5	4	3/4
		10	10	75.82	15.43	33.87	4/5	4	4
Chitosan Post Treated	Harda Meta Mordanted	0	10	63.99	19.08	38.25	4	3	3
		1	10	67.93	19.22	37.82	4	3/4	3
		5	10	66.24	16.93	38.08	4/5	3/4	3
		7.5	10	65.71	17.87	38.55	4/5	4	3/4

Alum Meta Mordanted	10	10	70.28	17.52	26.02	4/5	4	4
	0	10	78.22	17.43	25.76	3/4	3/4	3
	1	10	75.46	18.32	43.18	4	3/4	3
	5	10	74.24	17.16	43.14	4/5	3/4	3
	7.5	10	72.08	18.16	49.92	4/5	4	3/4
10	10	72.27	16.89	48.25	4/5	4	4	

**Physical Properties**

Effect of chitosan and cross linking agent on pre and post chitosan treatment on various physical properties such as crease recovery angle (CRA), bending length and tearing strength of annatto dyed fabric are tabulated in Table 3. As it was expected, tearing strength of the samples decreases with the application of chitosan and glutaraldehyde. Cross linking agent makes the fabric stiffer and tearing becomes easier as the yarn movement gets restricted. Hence it can be analysed that residual tearing strength of pre and post chitosan treated dyed samples lies in between 69.45% to 76.8%.

Bending length of the fabric increases significantly with increase in chitosan concentration in

both pre and post treatment. It is a result of mechanical deposition of chitosan on fabric surface as well as in the open interstices of the fabric via cross linking with glutaraldehyde which made the structure more rigid. It can also be analyzed that with increase in chitosan concentration CRA and bending length both increases significantly. Overall the higher concentration of chitosan provides good anticrease finish to the fabric.

It is pertinent to mention that in post chitosan treatment; chitosan is applied on to the fabric which already consists mordant and hence have more affinity for the chitosan molecules and shows more improved results of CRA and bending length than pre treated chitosan samples.

**Table - 3**

**Change in Various Physical Properties of Sample after Chitosan Application**

Mordant	Chitosan (%)	Alum Mordant (%)	Harda Meta Mordanted (%)	CRA (°)	Bending Length (cm)	Tearing Strength (Kg)	Residual Tearing Strength (%)
Undyed Fabric	0	0	0	192	1.81	2	--
Chitosan Pre treated	1	0	0	193	1.88	1.472	73.6
	5	0	0	195	2.31	1.536	76.8
	7.5	0	0	195	2.40	1.5	75
	10	0	0	195	2.40	1.408	70.4
	1	0	10	199	2.02	1.536	76.8
	5	0	10	199	2.16	1.408	70.4
	7.5	0	10	200	2.36	1.52	76
	10	0	10	203	2.52	1.536	76.8
	1	10	0	195	2.05	1.408	70.4
	5	10	0	198	2.25	1.42	71
Chitosan Post treated	7.5	10	0	198	2.38	1.428	71.4
	10	10	0	200	2.52	1.416	70.8
	1	0	0	201	2.7	1.468	73.4
	5	0	0	201	3	1.39	69.5
	7.5	0	0	206	3.1	1.532	76.6
	10	0	0	206	2.9	1.408	70.4
	1	0	10	194	2.8	1.389	69.45
	5	0	10	197	3	1.52	76
	7.5	0	10	204	3	1.53	76.5
	10	0	10	206	3.2	1.448	72.4
	1	10	0	194	2.8	1.518	75.9
	5	10	0	194	2.9	1.452	72.6
	7.5	10	0	197	2.7	1.406	70.3
	10	10	0	202	3.1	1.418	70.9

**Antibacterial Testing**

Antibacterial activities of the different natural dyed samples with and without natural and chemical mordant in combination with pre and post chitosan treated are given Table 4. It is observed that annatto dyed samples give large halo size and few isolated bacteria having leaching type activity. Annatto dyed samples with natural and chemical mordant show

hallo size but no bacterial growth hence the activity is satisfactory. Antibacterial activity of the all the samples treated with chitosan show no zone of inhibition as well as no growth of bacteria is found under the sample. It can be concluded that due to glutaraldehyde, chitosan gives permanent antibacterial finish to the dyed samples and making the finish non-leaching type.

Table - 4  
Antibacterial Activity of Samples Dyed with Annatto (10% owf)

	Size of halo (mm)	Bacterial Growth on Agar surface below the sample	Bacterial Growth Inhibition %	Grading of Activity of Treated Sample
Undyed definished fabric	-	Completely covered by bacterial growth	No inhibition	Not satisfactory
10 % Annatto Dyed Fabric	7	Few isolated bacteria	90-95%	Satisfactory, leaching type
10% Annatto Dyed + 10% Harda meta mordanted	2.55	no growth	95-98%	Satisfactory
10% Annatto Dyed + 10% Alum meta mordanted	2.5	no growth	95-98%	Satisfactory
1 %Chitosan Pre- Treated + 10% Annatto Dyed Fabric	-	No growth	98-100%	Satisfactory, non-leaching type
1 %Chitosan pre- Treated + 10% Annatto Dyed + 10% Harda meta mordanted	-	No growth	98-100%	Satisfactory, non-leaching type
1 %Chitosan pre- Treated + 10% Annatto Dyed + 10% Alum meta mordanted	-	No growth	98-100%	Satisfactory, non-leaching type
1 % Chitosan Post- Treated + 10% Annatto Dyed Fabric	-	No growth	98-100%	Satisfactory, non-leaching type
1 %Chitosan post- Treated + 10% Annatto Dyed + 10% Harda meta mordanted	-	No growth	98-100%	Satisfactory, non-leaching type
1 %Chitosan post- Treated + 10% Annatto Dyed + 10% Alum meta mordanted	-	No growth	98-100%	Satisfactory, non-leaching type

### Conclusion

The results show that cotton can be dyed naturally with annatto in orange shade. Combination with mordants changes the tone of the colour. The findings of the study show that chitosan treatment increases the depth and fastness rating of dyed samples. Chitosan imparts antibacterial property to samples and presence of cross linker glutaraldehyde makes it non-leaching and permanent type of finish. Bending length and crease recovery angle of chitosan treated samples improves to some extent however, tearing strength decreases. The results show that natural dye annatto in combination with biopolymer chitosan has great potential in producing sustainably dyed textile materials having antibacterial property.

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