

# Pre-treatment and Salt-Free Reactive Dyeing of Cotton materials Using Chitosan Extracted from *Oreochromis Niloticus* Scales.

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## Abstract

The coloration of cotton textiles is an important process for ornamenting the textile to emphasize the appearances and marketing potential. Cotton dyeing requires more amounts of common salt as an exhausting agent so as to penetrate the dye molecules into the interior structure of cotton and therefore the used common salt is not fully consumed within the process and drained as waste and makes more load within the effluent treatment process. Hence an idea has been made to reduce the load of effluent treatment and the problem to the environment use of an enormous amount of sodium chloride is replaced with the Chitosan extracted from the scale waste of *Oreochromis Niloticus*. The Chitosan was extracted from the thrown away wastes of *Oreochromis Niloticus* using the deacetylation reaction process. The extracted Chitosan was used as for the modification of cellulose structure of cotton, dyed with cotton material, and compared with conventional reactive dyeing using common salt as an exhausting agent. The effect of the use of extracted Chitosan in reactive dyeing as the exhausting agent was studied by testing washing fastness, rubbing fastness, light fastness, and environment testes BOD, COD, TDS and TSS values. It was found that the dye uptake was excellent and therefore the fastness properties were also excellent. The impact of dye effluent to the environment also found marginally by using extracted Chitosan was less as compared to sodium chloride load to the environment. it had been determined that the utilization of extracted Chitosan in cotton reactive dyeing was more active.

**Keywords** — Cotton dyeing; Chitosan; *Oreochromis Niloticus* Scales; Extraction

## I. INTRODUCTION

Cotton remains the king of fibers, and most of the world's apparel is formed of cotton. The cotton fabric has excellent physical and chemical properties, such as, stability, high dyeability, being cheap, easily available, hygroscopicity, air permeability, biodegradability, no static electricity, fine cross-section, high strength, durability, good mechanical properties, good moisture absorbency, versatile in application and luxury to wear[1]. Reactive dyes are

a category of dyes, which may covalently react with cellulose under suitable conditions to make strong and stable bonds. These dyes are desirable for cotton fabrics with appreciations to the excellent wash fastness arising from chemical bond formation between dye and cellulose under alkaline conditions. The reactive dyes provide a full range of bright shades, good wet fastness, and good light fastness, which is very desirable in textile applications [2].

In conventional dyeing process of cotton with reactive dyes, generally, 20-100 grams per liter of an exhausting agent like sodium sulphate or common salt to overcome the static repulsion between cotton surface charge and anionic reactive dyes so as to reinforce the substantivity and to enhance the dye adsorption and dyeing process [3]. In the absence of adequate salt concentration, reactive dye bath exhaustion is poor. Therefore, the main a part of the dye remains within the bath and subsequently gets hydrolyzed within the dye bath within the presence of alkali. A part of these hydrolyzed dyes gets exhausted on the material but remains unfixed. To get rid of the hydrolyzed/unfixed dye, time-consuming, energy-intensive and expensive washing off procedures is required [4].

The application process of reactive dye for cotton writes from the disadvantages of poor exhaustion of dye into the fiber, and therefore the use of massive amounts of electrolytes that ultimately create effluent containing a high concentration of color and salt, which causes environmental pollution [5]. These classes of dyes are most unfavorable one from the ecological point of view, these effluents produced gives high values of BOD/COD (Biological oxygen demand / Chemical oxygen demand) and increases salinity of the rivers affects the fragile biochemistry of aquatic life.

Pretreatment of cotton before dyeing offers a modest and active method of enlightening dye-fibre affinity, evading the need for salt as an electrolyte in the dye bath. As an attempt to reduce the use of salts, a number of researchers have cationized cotton fibers through chemical alterations with the compounds comprehending cationic groups to enrich the uptake of dyes from dye bath to fibers [6]. Fish waste is considered to be one of the major bio-pollutants which are generally discarded in coastal regions

through local markets and fish processing industries. Nowadays, it is being eyed as a newer bio resource. Fish scales are good source of chitin and chitosan. Chitosan applied to textiles has been widely studied for effects such as shrink resistance, improved dye uptake, and as auxiliary or anti-static agents, etc., because of the low toxicity and good biocompatibility of this natural polymer [7].

Chitosan was originally used as a dye deepening agent within the textile industry since it is cationic polymer, chitosan is taken into account to be a perfect fixer for anionic dyes and salt-free dyeing is feasible by using chitosan with the assistance of other additives [8]. Owing to the restrictions of cotton, some modifications are done to the chemical structure of cotton to enhance its dyeing properties, antimicrobial properties, shrinkage and other textile properties. Cotton fibers have a charge and since of this property, anionic dyes are repelled resulting in low exhaustion rates [9]. Chitin/chitosan is a naturally occurring biopolymer. It was found to be one of the world's most abundant biopolymer next to only cellulose. Despite the highly abundant of this biopolymer, is found only in a small group of living organisms, arthropods, some mollusk, and yeast and fungi. Chitin is a homopolymer of N-acetyl-D-glucosamine linked together by beta 1, 4 linkages. Structurally it is very similar to cellulose. Whereas chitosan is poly-β-(1-4)-D-glucosamine Chitosan is a natural carbohydrate biopolymer derived by deacetylation (DA) of chitin [10]

Chitosan, a polymer derived from chitin, is seen as a possible substitute for various hazardous chemicals used for the cationization of cotton. Chitosan is a linear Polysaccharide which consists of β (1, 4) linked D-glucosamine and N-acetyl-D-glucosamine which are allotted randomly in its structure. Chitosan is a biocompatible and the safest modifying agent that has been used in pretreatment of cotton to make it suitable for salt free dyeing with reactive dyes. Chitosan forms crosslinks with the cotton fibre and provides the amino groups for its cationisation, rendering it a positive charge. Chitosan treatment has proved to increase both the substantivity and reactivity of fibers towards reactive dyes under neutral conditions. Chitosan offers not only positively charged amine Group but also positive charged hydroxyl groups[11].

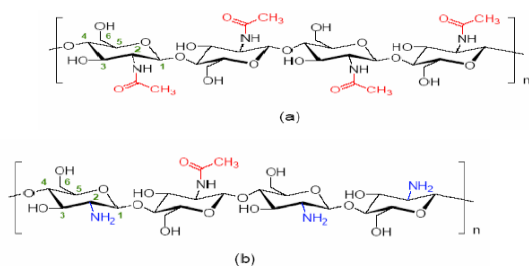


Fig 1: Comparison of the structures of chitin (a) and the chitosan (b)

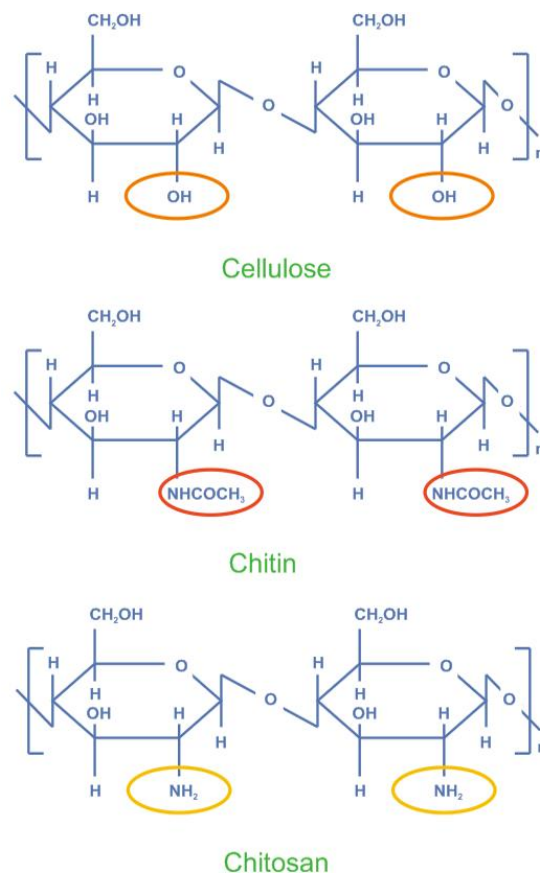


Fig 2: Structure of Cellulose, Chitin, and Chitosan [12]

The level of dyeing effect of chitosan results in a consistent layer on the surface of the cotton fiber. It improves the surface properties of the fiber and reduces the Coulomb repulsion between the fiber and therefore the anionic dyes, therefore greatly improving the dye Uptake rate. The deepening effect is helping to protonation of the free amino on the chitosan molecule under acidic conditions. When the material is immersed within the chitosan solution, the charge of the fiber is increased, thus the repulsion force between the fiber and therefore the anionic dyes is reduced. The aim of this study is that the dyeing of cotton with reactive dyes using eco-friendly naturally synthesized chitosan which is extracted from Oreochromis Niloticus scales for modification of cotton structure to reinforce dye uptake in reactive dyeing and to device different fastness properties.

## II. MATERIALS AND METHODS

### A. Sample Collection and Sample Preparation

The fresh Oreochromis Niloticus scales were collected from local fish market and washed with clean water and the impurities were removed. Then the cleaned scales were dried at room temperature, subjected to size reduction and kept in open air tight container.

**B. Method of extraction of chitosan from Oreochromis Niloticus scale**

The powdered Oreochromis Niloticus scale was demineralized by adding 1000 ml of 1 M hydrogen chloride to 100 gram of scale. Then demineralization process was preceded at room temperature for agitation of 250 rpm for 24 hours. Then decalcified scales were filtered and washed with distilled water until eutral pH [13]. Demineralized scales were deprotienized with 100ml of 1M sodium hydroxide by constant stirring at 60oC for 5 hours. The residues were collected and washed with distilled water until getting neutral pH. The solid mass of processes were washed with ethanol for removing the color and dried for overnight at 60oC in an hot oven. In this process the chitin was obtained [14]. The derived chitin was deacetylaed using 100ml of 50% NaOH and boiled at 120oC for 120 minutes on a hot plate. The sample will be washed with distilled water and ethanol until the clear solution obtained. Then the clear solution was filtered and the solid material was oven dried at 120oC for one day [15].

**C. Proximate analysis of raw Oreochromis Niloticus scale**

Ash, fat and crude protein, moisture content were determined based on the standard procedure of AOAC (2000) official methods 934.05, 942.39, 920.01, and 984.13, respectively. Protein content was determined using conversion factor of 6.25 with % nitrogen for raw material. Accurately weighed 5g of the wet raw Oreochromis Niloticus scale were taken in concave glass and kept in a hot air oven at 110°C for 6 hour. Then samples were cooled into room temperature in desiccator and weighed again and the percentage of moisture content was calculated [16]. 2 gram (w1) of Oreochromis Niloticus scale sample was placed on previously weighed the clean crucible (w2). Then the crucible were placed in a furnace and it was heated at 550°C overnight, the heating was completed to prevent loss of fluffy ash and it was cool down to the room temperature kept desiccator. Then the crucible measured with ash (w3) and the weight was recorded [16]. The ash content of the tilapia skin was calculated using equation 1.

$$\%AC = \frac{W_3 - W_2}{W_1} \times 100 \text{ -----1}$$

Where: W1 =weight of the sample, W2 = weight of clean crucible, W3 = weight of crucible measured with ash.

The total fat content of the raw Oreochromis Niloticus scale was determined using A.O.A.C, 2000 920.39 method. About 10 g of the sample was placed in the Soxthlet extraction apparatus and 250 ml of n-hexane was added and the sample was subjected to carry out extraction for 4 hours at 80°C. Then it was allowed to cool and

the oil was separated from n-hexane by using rotary evaporator and the experiment was conducted for three times in order to reduce the error Then the % of total fat in the raw scale was calculated using equation 2 [16].

$$\%FT = \frac{\text{weight of the fat}}{\text{weight of the sample}} \times 100 \text{ -----2}$$

The protein content of the raw Oreochromis Niloticus scale was determined according to AOAC, 2000 method. Accurately weighed (1g) raw scale sample was taken and added into 20 ml of 98% Sulphuric acid, Kjeldahl digestion tube containing 5 boiling chips, anhydrous potassium Sulphate, 1g of copper Sulphate and 1g of selenium (IV) oxide, (5:1:1) catalysts and the mixture digested in digestion stove at 40<sup>0</sup>C until the clear pale green solution was obtained. The digests were cooled and filtered in 500 ml Kjeldahl digestion flasks containing 150 ml distilled water and neutralized with 70 ml (50%) of sodium hydroxide solution and attached to micro Kjeldahl distillation apparatus. About 50 ml of 30% boric acid solution and 3 drops of indicator (bromo cresol green plus methyl red) was added into 150 ml Erlenmeyer flask. The receiving flask was removed from the condenser after 150 ml of distillate was collected. The amount of protein (in terms of nitrogen) was determined by 0.1N standardized HCl used as treatment and the titration carried out on sample distillate until pink color was observed and the protein content was calculated using equation 3 [16].

$$\%Protein = \frac{(A-B) \times N \times 1.4007 \times 6.25}{W} \times 100 \text{ -----3}$$

Where A = Volume (ml) of 0.1 N HCl required for titration of sample.

B = Volume (ml) of 0.1 N HCl required for titration of blank.

W = weight of sample in gram.

N = Normality of HCl.

**D. Determination of degree of Deacetylation of tilapia fish scale extracted chitosan**

The degree of Deacetylation was measured by the acid-base titration. In brief, 0.1g-of chitosan was is solved in a conical flask containing 30 ml of 1M of hydrogen chloride at room temperature and 5 drop of methyl orange was added and the content of the flask was titrated with 0.1 M of sodium hydroxide until the color changes from red to orange color [17].Then the

degree of Deacetylation was calculated using equation 4

$$DD(\%) = \frac{C1V1 - C2V2}{M \times 0.0994} \times 0.016$$

Where C1 = concentration of hydrochloric acid, C2 = concentration of sodium hydroxide, V1 = volume of hydrochloric acid used to dissolve chitosan in ml, V2 = volume of sodium hydroxide consumed during titration and M = weight of chitosan in gram. The number 0.016 is the equivalent weight of NH<sub>3</sub>-group in 1 ml of hydrochloric acid solution. And 0.0994 is the proportion of NH<sub>3</sub>-group by weight in chitosan.

#### E. Fabric Finishing Process

The grey fabrics were prepared for dyeing and finishing process by removing natural and added contaminations by the standard procedure. The starches added during weaving process to enhance the strength of warp yarn were desized using Hydrochloric acid [18, 19].

The material was desized using 1% by material weight of hydrochloric acid at 45<sup>o</sup> C for 2 150 minutes using material is to liquor ratio of 1 is to 20 with 5.5 to 6 pH. The natural impurities of the cotton fabric such as oil, wax and mineral matters were removed by scouring process using sodium hydroxide in open bath method at boiling temperature to remove.

The desized material was scoured using 4% of Sodium hydroxide with 1% of Sodium carbonate at boiling temperature using material is to liquor ratio of 1 is to 20 for 150 minutes. The natural colouring matter present the scoured fabric was removed and the whiteness of the fabric was removed by hydrogen peroxide bleaching process to enhance the dye intake.

The scoured material was bleached using a 2.5 % of universal beaching agent hydrogen peroxide at 95<sup>o</sup>C to boiling temperature 135 minutes using material is to liquor ratio of 1 is to 20

#### F. Pre-treatment of cotton fabric with Chitosan

Pre-treatment of cotton fabric with Chitosan was done using pad dry cure method. The Pre-treatment bath was setup with essential amount of water and chitosan solution with 10%, 15% and 20% concentration at room temperature. The well prepared material was introduced into the bath, after 5 minutes the required amount of sodium chloride was added and the finishing was carried out for 90 minutes. During this period Material liquor ratio (MLR) and temperature were maintained as constant. Then the samples were kept at hot air oven at 180<sup>o</sup>c

for 5 minutes [20]. The recipe used for finishing is mentioned in table I

#### G. Preparation of Dye Solution

Dyestuff stuff is weighed accurately based on the weight of cotton fabric and is pasted with minor amount of cold water and hot water is added and then it is prepared into solution based on M: L ratio. Then the dye solution is filtered by using nylon mesh.

#### H. Dyeing of Fabric with Reactive Dyes

The dye bath is setup with prerequisite amount of water and dye solution at room temperature. The well prepared material is introduced into the bath, after 5 minutes the required amount of sodium chloride is added for all the samples except sample 1. Then the temperature of the dyeing bath is gradually raised to 70<sup>o</sup>C and the dyeing is carried out for 90 minutes. During this period MLR and temperature are maintained as constant. The dyeing parameters are given in table II.

#### I. Dye Fixation

Calculated amount of soda ash solution is added and dyeing was carried out for further 25 minutes at 70<sup>o</sup>C, after 20 minutes the samples were taken out. The dyed material was rinsed by using cold water and then by hot water at 70<sup>o</sup>C. Then the rinsed material was neutralized by using 1 gpl of acetic acid solution at room temperature,—then by using cold water. Finally the material was soaped by using non-toxic soap oil at 95<sup>o</sup>C for 15 minutes and it is rinsed with cold water and dried in even.

#### J. Color Fastness Properties Test

The colour durability of the treated fabric during washing and caring is said to be the fastness of colour. The samples were tested for washing, rubbing and sunlight fastness test based on AATCC standards. AATCC- 107/2002, AATCC-008/2005, AATCC-16/2004 standards were followed for washing, rubbing and sunlight fastness tests [21].

### III. RESULT AND DISCUSSION

Ash, fat and crude protein, moisture content and degree of Deacetylation were determined based on the standard procedure of AOAC (2000) official methods 934.05, 942.39, 920.01, and 984.13, respectively and mentioned in the table III.

The laboratory test samples were prepared according to the description from relative percentage of all the dyed samples for the laboratory tests. The fabric was observed for wash fastness, rubbing fastness, light fastness, tensile strength, and environment testes BOD, COD, TDS and TSS values based on AATCC Methods [21].



TABLE I  
Recipe for Finishing

Description	Parameters		
Chitosan Extract	8% by material by weight	12% by material by weight	18% by material by weight
Temperature	Room temperature	Room temperature	Room temperature
Time	90 Minutes	90 Minutes	90 Minutes
Material liquor ratio (MLR)	1:20	1:20	1:20
Curing Temperature	180°C	180°C	180°C
Curing Time	5 Minutes	5 Minutes	5 Minutes

TABLE II  
Dyeing Parameters

Particulars	Sample1 8% Chitosan	Sample 2 12% Chitosan	Sample 3 18% Chitosan	Sample 4 No Chitosan
Dye	3%	3%	3%	3%
NaCl	0%	0%	0%	20%
Na <sub>2</sub> CO <sub>3</sub>	5%	5%	5%	5%
Soap Oil	1.5%	1.5%	1.5%	1.5%
M:L Ratio	1:20	1:20	1:20	1:20
Temperature	70°C	70°C	70°C	70°C
Dyeing Duration	90 Minutes	90 Minutes	90 Minutes	90 Minutes
Fixation Duration	25 Minutes	25 Minutes	25 Minutes	25 Minutes
% of Shade	2	2	2	2
pH for Dyeing	10-11	10-11	10-11	10-11

TABLE III

**Ash, fat and crude protein, moisture content and degree of Deacetylation**

Sample	Ash Content (%)	Fat Content (%)	Crude Protein (%)	Moisture Content (%)	degree of Deacetylation
Sample 1	2.65	3.6	8.25	3.5	76.38
Sample 2	2.75	3.5	7.95	3.48	75.85
Sample 3	2.8	3.48	8.02	3.6	74.25
Sample 4	2.68	3.58	8.65	3.58	76.52
Sample 5	2.72	3.49	7.98	3.49	75.01
Mean Value	2.72	3.53	8.17	3.53	75.602

Note: The given values have tolerance of ± 0.5 % for Ash, Fat, Crude Protein and Moisture. And ± 1 Degree of deacetylation

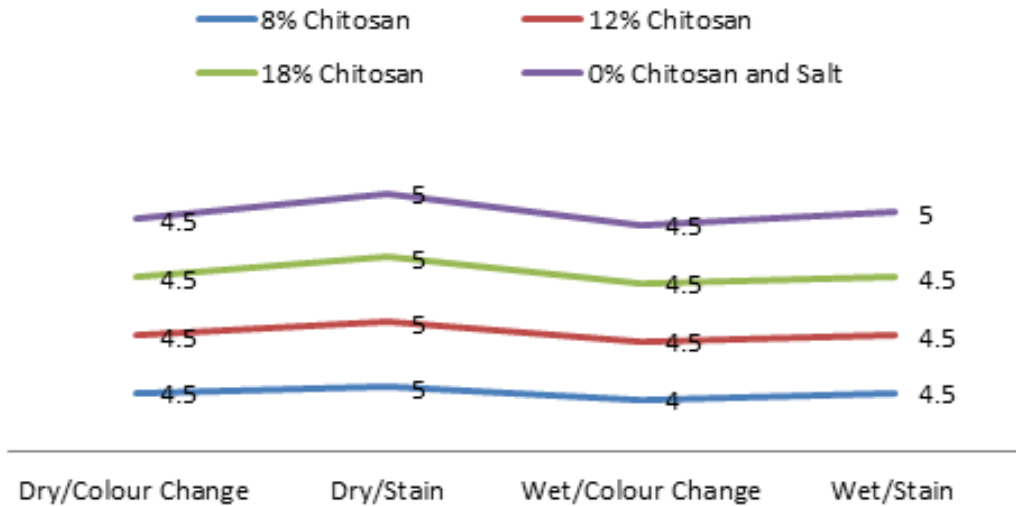


Fig 3. Rubbing fastness of dyed sample

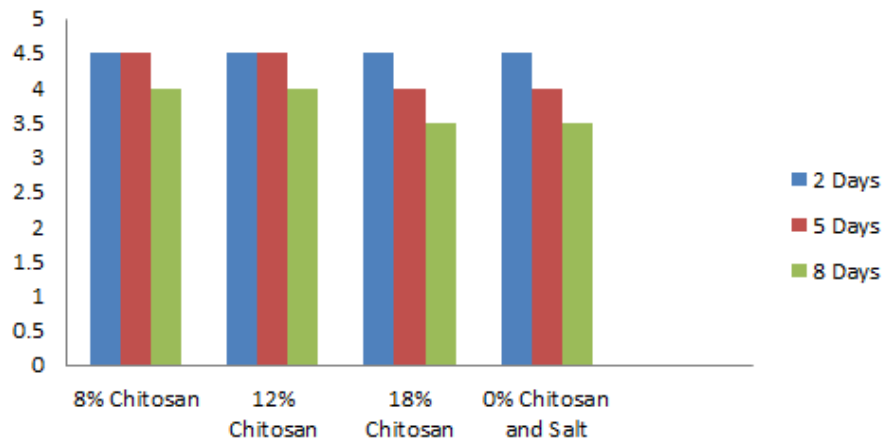


Fig 4. Light Fastness of Dyed Samples

### Washing Fastness

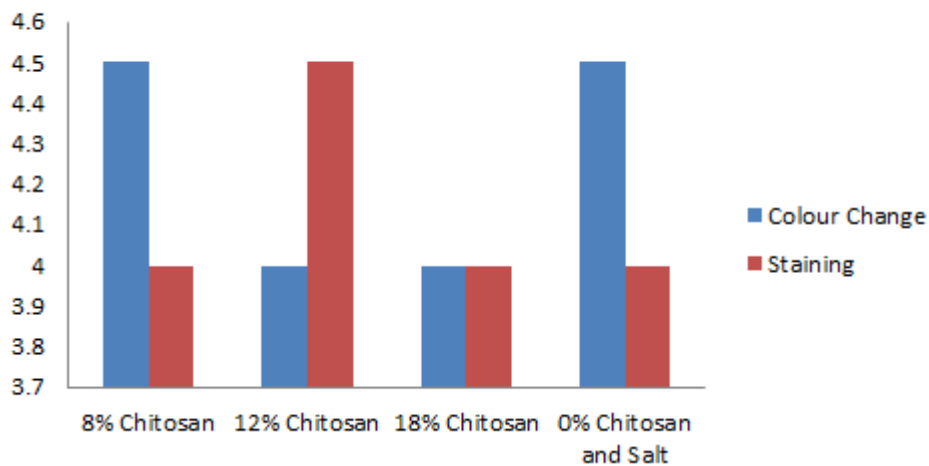


Fig 5. Washing fastness of Dyed Samples.

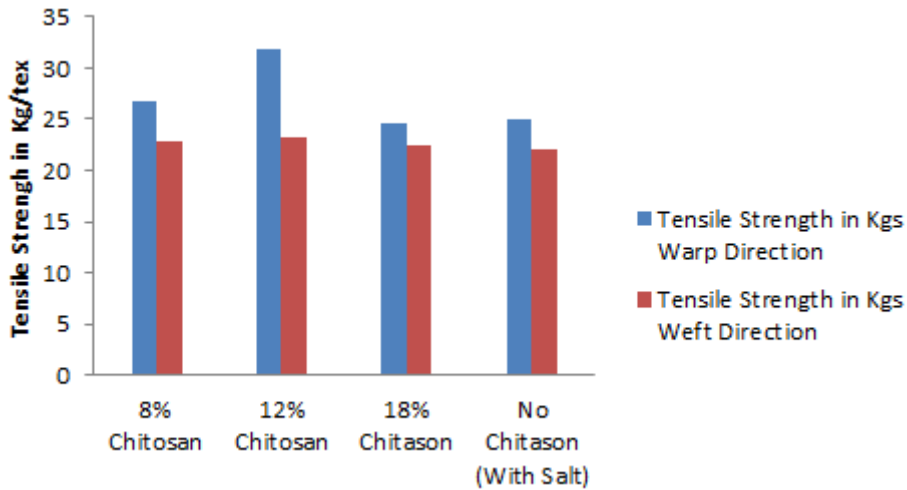


Fig 6. Tensile Strength of Dyed Samples.

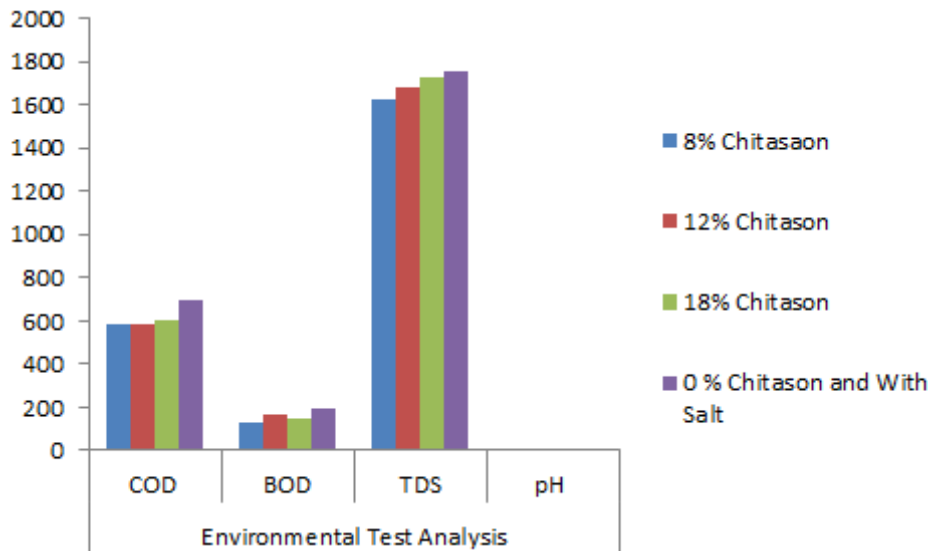


Fig 7. Results of COD, BOD, TDS and pH Value

The rubbing fastness for wet and dry conditions of chitosan pre-treated and without chitosan were tested results shows that there the rubbing fastness are in the very good to fare range and there is no significant changes between chitosan treated and salt treated and hence its understood that the quality of rubbing fastness not changed to non-use of salt in dyeing process.

The results obtained for washing fastness of fabrics which are dyed using reactive dyes with different proportions of chitosan as a structural modifier by pre-treatment process and cotton fabrics dyed with g salt as exhausting agent is not having significant changes and hence the use of chitosan extracted from *Oreochromis Niloticus* scales for the pre-treatment in reactive dyeing may considered by replacing the use of salt as an exhausting agent.

**K. Tensile Strength Test**

Tensile strength of dyed samples using chitosana and sodium chloride were tested using tensile strength tester using ASTM D5034-95 standards [22].

The graph shows that the samples dyed without salt and with chitosan have high strength considerably as compared with the samples dyed with sodium choloride as an exhausting agent.

**L. Environmental Test Analysis**

Chemical Oxygen Demand (COD): The COD is used as a measure of oxygen equivalent of organic matter content of sample that is susceptible to oxidation by strong chemical oxidant for sample from a specific source. It is the measure of both biologically oxidizable and biologically inert organic matter [23]. The effluent from dyed samples with chitosan and

with salt are collected distinctly and evaluated for COD and the results are given in fig 7.

Biological Oxygen Demand (BOD): The BOD is a measure of the quantity of oxygen used by microorganisms in the oxidation of organic matter [24]. The effluent from dyed samples with chitosan and with salt was collected distinctly and evaluated BOD and the results are given in fig 7.

Total Dissolved Solids (TDS) : The TDS (mg/l) and TSS (mg/l) values of effluents for dyed samples with chitosan and with salt were premeditated and given in fig 7.

pH Value: The pH Value of effluents for dyed samples with chitosan and with salt were evaluated and given in fig 7 .

The result shows that that the COD, BOD, TDS and pH of the samples dyed with different chitosan as pre-treatment component are better as compared to the samples dyed with sodium chloride as an exhausting agent.

#### IV. CONCLUSION

Cotton fabrics pre-treated with the chitosan extracted from *Oreochromis niloticus* Scales were dyed with reactive dyes with sodium chloride as an exhausting agent. The dyed samples were analyzed for the colour fastness strength properties and environment tests. The obtained results show that the colour fastness properties within the dyes were not having any significant changes and hence it's proved that there are no changes by using chitosan as structural modifier. The tensile strength of chitosan treated fabric is improved considerably as compared to the fabric dyed with sodium chloride as an exhausting agent. The effluent load of dyeing process were reduced by using chitosan as a pre-treatment agent for dyeing based on the COD, BOD, TDS and pH analysis. Hence based on the study we can conclude that the use of salt can be eliminated by using chitosan as Cotton structure modifying pre-treatment agent in reactive dyeing.

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