Raw Silk Processing to Textile Fibre and Comparative Analysis between Acid and Reactive Dyed Samples of Silk Fabric by Laboratory Experiment

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Abstract Silk fibre is one of the important and demanded textile fibre in the sector of textile fabric and garments manufacturing. In the industrial sector the coloration of silk fabric is done by acid dyes. In this paper, laboratory experiments were done for coloration of silk fabric. For coloration two different dyes, acid dye (Nylosan Orange NRL) and reactive dye (Lanasol Orange RG) have been used with their Different recipe, separately applied on different samples but with same properties of silk fabrics. Different tests like wash fastness, rubbing fastness, perspiration test, tensile strength and elongation were conducted on the dyed samples and comparison was made among all the results from Acid dyed and Reactive dyed samples. Getting better wash fastness, better absorbency and other positive test results for reactive dyes leads to the conclusion, that dyeing of silk with reactive dye has a great prospect in textile coloration.

Keywords Acid dye, Lanasol Orange RG, Nylosan Orange NRL, Raw Silk, Reactive dye, Silk fibre.

1. Introduction

Silk is a natural protein fibre of animal origin. The dyeing mechanism of silk dependents on free amino groups, carboxyl groups and phenolic with accessible –OH group. Because of slightly cationic character of silk with isoelectric point at above pH 5.0, it can be dyed with anionic dye such as acid, metal complexes, reactive and selected direct dyes. But the main objective of coloration of a textile fibre is that the permanency of the color and should not allow damage of natural abstract of fibre. This implies that it should not destroy its color during processing following coloration and dyeing & subsequent useful life (i.e. washing, light, rubbing, perspiration, and saliva). Silk contain very small amount of sulphur. There are two main types of silkworm, mulberry silk also called 'cultivated silk' and wild silk of which Tussah silk is the most important representative [1]. The mechanical properties of silk fibres are shown below (Table 1)

TABLE 1: MECHANICAL PROPERTIES OF SILK FIBRE [2]

Types of Fibre	Material	Density (g/cm3)	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation at Failure (%)
Natural Fibres	Spider Silk	1.3	1300-2000	30	19-30
	Enhanced B.Mori Silk	1.3-1.38	600-700	12.2	30-35
	B. Mori Silk	1.3-1.38	500	8.5-8.6	15

1.1 Chemical composition of silk:

The strands of raw silk as they are unwound from the cocoon consist of the two silk filaments mixed with sericin and other materials. About 75 % of the strand is silk i.e. fibroin and 22.5 % is sericin; the remaining materials consist of fat and wax (1.5 %), ash of silk fibroin (0.5%) and mineral salts (0.5 %). As a consequence of the spinning process, the fibre has two main part sericin and fibroin. Sericin called silk gum a minor component of the fibre. Sericin is yellow, brittle, and inelastic substance. It acts a twin fibroin filament and conceals the unique lustre of the fibroin. Sericin is an amorphous structure and it is dissolved in a

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hot soap solution. The greatest sericin content is present in outer layer of cocoon whereas the least sericin is present in the innermost layer of the cocoon. Fibroin is the principal water insoluble protein (i.e. 75% of the weight of raw silk). Fibroin has highly oriented and crystalline structure and it is composed of 16 various amino acids. Raw silk is treated for removing the gummy materials and various types of impurities from the silk by scouring process. Then the silk fiber is collected and supplied for producing silk fabric by weaving process [1], [2], [3]

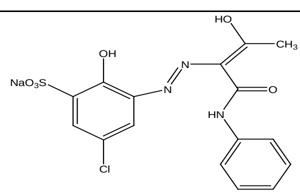
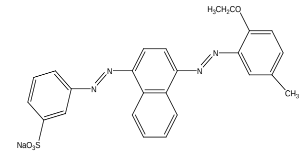
TABLE 2: EFFECT OF DIFFERENT TYPES OF CHEMICALS ON SILK FIBRE [3]

Particulars	Resultant Effect
Effect of Acids	The Fibroin of silk can be decomposed by strong acids into its constitute amino acids. In moderate concentration, acids cause a contraction in silk. Dilute acids do not attack silk under mild conditions.
Effects of Alkalis	Silk is less readily damaged by alkalis than wool. Weak alkalis such as soap, borax and ammonia cause little considerable damage. Silk dissolves in solutions of concentrated caustic alkalis.
Effect of Organic Solvent	Silk is insoluble in the dry-cleaning solvents in common use.
Effects of Insects	Insect does not affect silk.
Effect of Mildew	Silk is affected by mildew slightly.

1.2 SILK DYEING WITH ACID DYES:

Acid dyes contains acid groups, such as –COOH and –SO3H which form attractions to the slightly basic –NH groups in the amide links of wool, silk and nylon [4]

TABLE 3: THE CHEMICAL CONSTITUTIONS OF SOME TYPICAL ACID DYES [5]

Name	Color Index (C.I.) & CAS Registry Number	Chemical Structure
C.I. Acid Orange 97	C.I. Number:13890 CAS Registry Number:6460-02-2	 (Single azo,1:2 Metal Complexes)
C.I. Acid Orange 116	CAS Registry Number:12220-10-9	 (Double azo class)

Although, in acid dyes the functional group like $-SO_3Na$ or $-SO_3^-$ are responsible for dye solubility in water (Solubilizing group) and also create ionic bond with protein fibre (e.g.: Silk). The reaction between silk fibre and acid dyes is given below:

Fibre-NH2(S) + H+ (aq) + HSO4- (aq) → Fibre-NH3+HSO4-

Fibre-NH3HSO4(S) + Dye-SO3-(aq) → Fibre-NH3+Dye-SO3-(S) + HSO4-(aq)

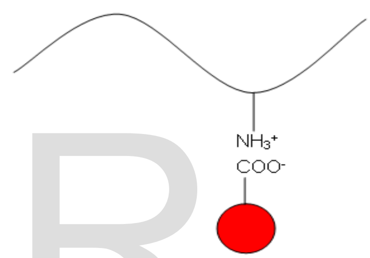


Figure 1: Silk fibre dyeing with acid dyes [4].

1.3 SILK DYEING WITH REACTIVE DYES:

Reactive dyes are anionic water soluble colored organic compounds that are capable of forming a covalent bond between reactive groups of the dye molecule and neucleophilic groups on the polymer chain within the fibre. Consequently, the dyes become chemically part of the fibre by producing dyepolymer linkages. In this regard, covalent dye-polymer bonds are formed, for instance, with the hydroxyl groups of cellulose, the amino, hydroxyl, mercapto groups of proteins and the amino groups of polyamides.

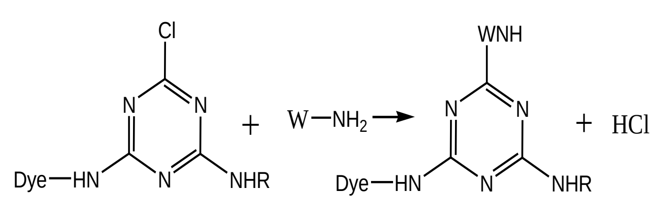


Figure 2: Reaction of a mono-chlorotriazine dye with amino group of protein

Forming a covalent bond between dyes and fibres had long been attractive to dye chemists, since attachment by physical adsorption and by mechanical retention had the disadvantage of either low wash fastness or high cost. It was anticipated that the covalent attachment of the dye

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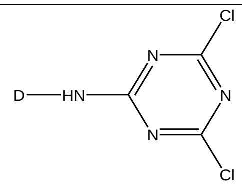
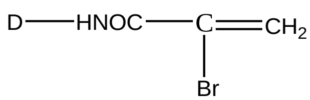
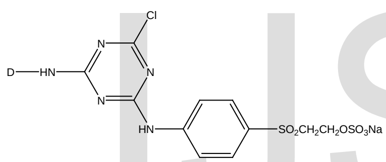
molecules to the fibre would produce very high wash fastness because covalent bonds were the strongest known binding forces between molecules. The energy required to break this bond would be of the same order as that required breaking covalent bonds in the fibre itself. Reactive dyes were initially introduced commercially for application to cellulosic fibres and this is still their most important use. Reactive dyes have also been developed commercially for application on protein and polyamide fibres [1]

TABLE 4: THE CHEMICAL CONSTITUTIONS OF SOME REACTIVE DYES APPLICABLE ON SILK [6]

Trade Name and Manufacturer	Reactive Group
Procion MX (Zeneca)	
Lanasol (CGY)	
Sumifix Supra (NSK)	

2. METHODS AND MATERIALS

2.1 RAW MATERIALS

Raw materials/Samples: Raw Silk, Silk Fabric (Weaved)

Dyeing Materials: Nylosan Orange NRL (Acid Dye), Lanasol Orange RG (Reactive Dye), Dyeing chemicals and auxiliaries, Laboratory dyeing machine, Woven fabric testing instruments.

2.2 SILK DEGUMMING PROCESS

Silk degumming process is applied on raw silk (Table 5 & Table 6).

TABLE 5: RECIPE FOR SILK DEGUMMING PROCESS

Chemicals and Parameters	Recipe
Soap	15 g/l
Wetting Agent	2 g/l
Material and Liquor Ratio (M:L)	1:50
Temperature	80°c
Time	90 min.
pH	10

TABLE 6: RECIPE CALCULATION FOR SILK DEGUMMING PROCESS

Particulars	Calculation
Fabric weight	49 g
Liquor (49×50)	2450 ml
Soap	(15×2450)/1000=36.75g
Wetting agent	(2×2450)/1000=4.9g
pH	10
Weight after degumming	47.7 g
Weight loss %	(49-47.7)/49% = 2.65%

Procedure for silk degumming:

At first the weight of the raw fabric was measured with electrical balance. Required amount of water was taken into the bath. Then required amount of wetting agent and soap was added into the bath and dissolved it properly. The pH of the bath was checked and the fabric was added into the bath. Then, the temperature of the bath raised to 90 degree Celsius and continued for 90 min. After 90 min, the fabric was taken out from the bath and washed with cold water then dried it using woven. Then the dried weight of the fabric was taken and the amount of weight loss was calculated.

2.3 SILK BLEACHING PROCESS

TABLE 7: RECIPE FOR SILK BLEACHING

Chemicals and Parameters	Recipe
Hydrogen per oxide (H2O2)	20 ml/l
Invatex AC (Organic acid)	0.2 g/l
Wetting agent	1 g/l
Tri-sodium-phosphate	to adjust pH 8-9
EDTA	1g/l
M: L	1:70
Time	90min.

Temperature	(65-70) ˚C

TABLE 8: RECIPE CALCULATION FOR SILK BLEACHING PROCESS

Particulars	Calculation
Fabric weight	47.7 g
Liquor	47.7×70=3339 ml
Hydrogen per oxide	(20×3339)/1000=66.6
Invatex AC	(0.2×3339)/1000=0.66 g
Wetting agent	(1×3339)/1000=3.33 g
EDTA	(1×3339)/1000=3.33 g
Tri-sodium-phosphate	(2.1×333.9)/1000=6.98 g
pH	8.5
Weight loss %	0.7%

Procedure for silk bleaching:

At first fabric weight was measured with electrical balance. The operation bath was filled with required amount of water. Then wetting agent, EDTA, Invatex AC added into the bath and the pH of the solution was measured and tri-sodium-phosphate was added to adjust the pH of the solution. Then the fabric was inserted into the bath and the temperature raised to 50oC. Then hydrogen per oxide was added into the solution. Then the temperature raised to 70oC. The temperature of the bath was kept around 65-70oC for 90 min. After 90 min. the fabric was taken out from the bath. Then washed it once in cold water and once in hot water, followed by rinsing in water. At last weight was taken to calculate the loss in weight due to bleaching.

2.4 SILK DYEING WITH ACID DYE

TABLE 9: APPLIED RECIPE FOR SILK DYEING WITH ACID DYE

Parameters	Recipe
Dye (Nylosan Orange NRL)	x %
Lyogen PAM Liq	0.50-2.00 g/l
Opticid (acid generator)	1.00 %(pH 4-5)
Wetting agent	1 g/l
EDTA	1 g/l
M: L	1:60
Temperature	(75-85) ˚C
Time	40-60 min
After Treatment	
Nylofixan P Liq	3.00-5.00 %
Acetic acid	2.00 %
Temperature	70oC
Time	30 min.
Rinse	Cold Rinse

TABLE 10: SILK DYEING RECIPE CALCULATION FOR DIFFERENT SHADE % WITH ACID DYE

Parameters	For 1% Shade	For 1.5% Shade	For 2% Shade	For 3% Shade
Fabric weight	3.97 g	3.16 g	3.72 g	2.96g
Liquor	238.2 ml	189.6 ml	223.2 ml	177.6 ml
Lyogen PAM Liq	0.23 ml	0.19 ml	0.22ml	0.177 ml
Opticid	0.04 ml	0.03 ml	0.03 ml	0.29 ml
Wetting agent	0.23 ml	0.19 ml	0.223 ml	0.177 ml
EDTA	0.23ml	0.19 ml	0.223 ml	0.177 ml
Dye	3.97 ml	4.74 ml	7.44 ml	8.88 ml
After Treatment				
Nylofixan P Liq	0.14 ml	0.11 ml	0.13 ml	0.1036 ml
Acetic acid	0.08 ml	0.06 ml	0.07 ml (to adjust pH 4.5)	0.5 ml

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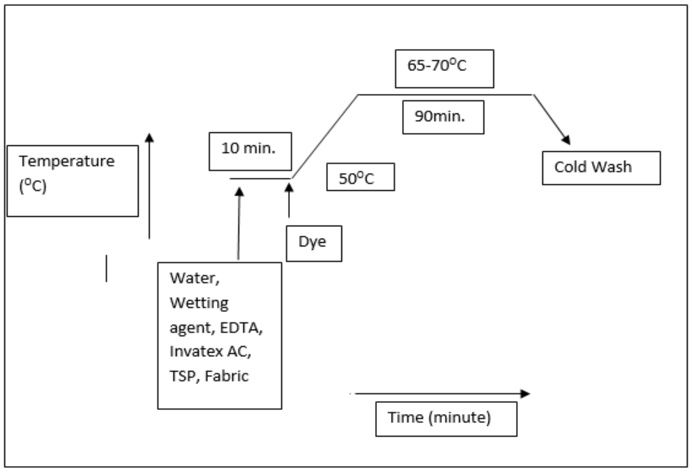

Figure 3: Dyeing curve of silk dyeing with acid dye (Nylosan Orange NRL).

2.5 Silk Dyeing with Reactive Dye

Table 11: General recipe for silk dyeing with reactive dye

Parameters	Recipe
Dye (Lanasol Orange RG)	X%
Glauber Salt (Na2SO4 . 10 H2O)	20-80 g/l
Soda ash	1-2 g/l (pH 8.5)
Sequestering agent	1 g/l
Wetting agent	1 g/l
After Treatment	
Cool bath and rinsing	
Soaping	1–2 g/l; ERIOPON® R liq. (anionic soaping agent); (15 min. at 80°c)
Cold rinsing	
Scooping (acetic acid)	1 ml/1 (10 min. at 40°C)

Table 12: Silk dyeing recipe calculation for different shade % with reactive dye

Parameters	For 1% Shade	For 1.5% Shade	For 2% Shade	For 3% Shade
Fabric weight	5.77 g	5.63 g	5.56 g	5.36 g
Required liquor	288.5 ml	281.5 ml	278 ml	268 ml
Dye	5.77 ml	8.44 ml	11.12 ml	16.08 ml
Glauber Salt	12.98g	12.66g	12.51g	12.06g
Soda ash	0.43 g	0.42 g	0.42 g	0.40 g
Sequestering agent.	0.288 g	0.281g	0 .278 g	0.268 g
Wetting agent	0.288 g	0.281g	0 .278 g	0.268 g

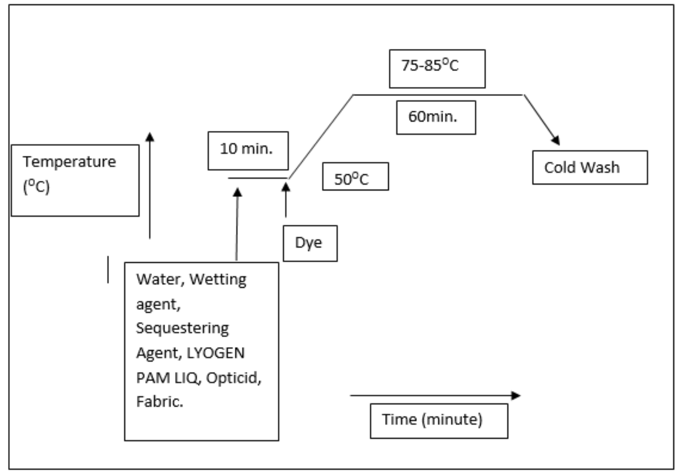

Figure 4: Dyeing curve of silk dyeing with reactive dye (Lanasol Orange RG).

2.6 Test Procedures for Dyed Materials

2.6.1 Color Fastness to Washing

ISO 105 C03 was followed to carry out the wash fastness test of both acid and reactive dyed samples.

2.6.2 Color Fastness to Rubbing

ISO 105 X 12 was followed to carry out the rubbing fastness test of both acid and reactive dyed samples.

2.6.3 Color Fastness to Perspiration

ISO 105 E04 was followed to carry out the perspiration fastness test of both acid and reactive dyed samples.

2.6.4 Strength Testing

Strength test was carried out following the standard method of ASTM D5034.

Equipment: A computerized strength test machine namely "Universal Strength Tester (Titan)" was used to measure the strength of the samples.

The samples used in this test are:

Raw Silk

Bleached silk

Acid dyed silk

Reactive dyed silk

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3. Result and Discussion

3.1 Silk Fabric Dyed Samples with Acid Dye and Reactive Dye

Figure 5: Silk fabric dyed samples with acid dye and reactive dye

3.2 Grey Scale Evaluation for Color Fastness to Washing

Table 13: Grey scale assessment of change in color for colorfastness to washing of acid and reactive dyed silk woven fabrics

Acid dyed sample				Reactive dyed sample			
Nylosan Orange NRL	Nylosan Orange NRL	Nylosan Orange NRL	Nylosan Orange NRL	Lanasol Orange RG	Lanasol Orange RG	Lanasol Orange RG	Lanasol Orange RG
1%	1.5%	2%	3%	1%	1.5%	2%	3%
4-5	4-5	4-5	4-5	5	5	5	5

Comparing the grey scale grading for change in color for the test of color fastness to washing (Table 13), it can be seen that reactive dyed sample gives excellent (5) result in case of all kinds of tested shade % than acid dyed samples (4-5).

Table 14: Grey scale assessment of Staining for colorfastness to washing of acid and reactive dyed silk woven fabrics

Multifibre fabric	Acid dyed sample				Reactive dyed sample			
	Nylosan Orange NRL 1%	Nylosan Orange NRL 1.5%	Nylosan Orange NRL 2%	Nylosan Orange NRL 3%	Lanasol Orange RG 1%	Lanasol Orange RG 1.5%	Lanasol Orange RG 2%	Lanasol Orange RG 3%
Di-acetate	4	3-4	4	3	4	4-5	4	4-5
Bleached cotton	5	4-5	4-5	4-5	5	5	5	5
Polyamide	4-5	4	4	3-4	3	5	5	5
Polyester	5	4-5	5	4-5	5	3	2-3	2
Acrylic	5	5	5	5	5	5	4-5	4-5
Wool	5	5	5	4-5	4	4	4-5	3-4

From Table 14 it can be seen that incase of acid dye Bleached cotton, Polyester, Acrylic and Wool gives very good result and for these fabrics the staining result belongs to 4-5. For Polyamide the grading for staining is good up to 2% shade but in case of 3% shade it gives poor result (3-4). In case of Di-acetate no continuing result has been found but it gives the lowest grading among all of the samples for 3% Shade (Grey scale grading 3).

On the other hand, for reactive dye Di-acetate, bleached cotton, Acrylic gives very good result (Grey scale grading range 4-5), Wool also gives good result except high shade % (3-4 for 3% shade), polyamide also gives excellent result except 1% shade but in case of Polyester the staining result is very poor and lowest among other samples for higher shade % (Grey scale grading 2 for 3% shade).

From above circumstance, the conclusion is that more or less all fibres of multifibre fabric is stained by acid dyed silk and reactive dyed silk. But it is noticeable in assessment that Di-acetate, polyamide, polyester is more stained by both dyed samples than any other fibre of multifibre fabric.

3.3 Color Fastness to Rubbing

Table 15: Assessment result of rubbing fastness at warp ways for acid and reactive dyed woven fabrics

Acid dyed sample								Reactive dyed sample							
Dry				Wet				Dry				Wet			
Nylosan Orange NRL				Nylosan Orange NRL				Lanasol Orange RG				Lanasol Orange RG			
1%	1.5%	2%	3%	1%	1.5%	2%	3%	1%	1.5%	2%	3%	1%	1.5%	2%	3%
5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5

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TABLE 16: ASSESSMENT RESULT OF RUBBING FASTNESS AT WEFT WAYS FOR ACID AND REACTIVE DYED WOVEN FABRICS

Acid dyed sample								Reactive dyed sample							
Dry				Wet				Dry				Wet			
Nylosan Orange NRL				Nylosan Orange NRL				Lanasol Orange RG				Lanasol Orange RG			
1%	1.5%	2%	3%	1%	1.5%	2%	3%	1%	1.5%	2%	3%	1%	1.5%	2%	3%
5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5

From Table 15 and Table 16 it can be seen that, in case of both acid and reactive dyed silk fabric samples the grey scale grading is 5. Which is to say, no color transferred to undyed cotton cloth. So it can be said that color fastness to rubbing for both acid and reactive dyed silk is totally satisfactory.

3.4 COLOR FASTNESS TO PERSPIRATION

TABLE 17: ASSESSMENT RESULT OF COLOR CHANGE TO PERSPIRATION ON ACID AND REACTIVE DYED WOVEN FABRICS

Acid dyed sample				Reactive dyed sample			
Acidic perspiration solution		Alkaline perspiration solution		Acidic perspiration solution		Alkaline perspiration solution	
Nylosan Orange NRL 1%	Nylosan Orange NRL 3%	Nylosan Orange NRL 1%	Nylosan Orange NRL 3%	Lanasol Orange RG 1%	Lanasol Orange RG 3%	Lanasol Orange RG 1%	Lanasol Orange RG 3%
5	5	5	5	5	5	5	5

TABLE 18: ASSESSMENT RESULT OF STAINING TO PERSPIRATION ON ACID AND REACTIVE DYED WOVEN FABRICS

Multifibre fabric	Acid dyed sample				Reactive dyed sample			
	Acidic perspiration solution		Alkaline perspiration solution		Acidic perspiration solution		Alkaline perspiration solution	
	Nylosan Orange NRL 1%	Nylosan Orange NRL 3%	Nylosan Orange NRL 1%	Nylosan Orange NRL 3%	Lanasol Orange RG 1%	Lanasol Orange RG 3%	Lanasol Orange RG 1%	Lanasol Orange RG 3%
Di-acetate	5	5	5	5	5	5	5	5
Bleached cotton	5	5	5	5	5	5	5	5
Polyamide	4-5	4	4-5	4-5	5	5	5	5
Polyester	5	5	5	5	5	5	5	5
Acrylic	5	5	5	5	5	5	5	5
Wool	5	5	5	5	5	5	5	5

From Table 17 it can be seen that, for both acid and reactive dyed silk fabric, there was not any change in color due to the perspiration test.

From Table 18 it can be seen that, for staining to perspiration test, only polyamide fibre among different fibres of multifibre fabric is stained by acid dyed silk, none of fibre from multifibre fabrics is stained by reactive dyed silk.

3.5 TENSILE STRENGTH TEST REPORT

The result of tensile strength test of raw silk, degummed and bleached silk, acid and reactive dyed silk are shown as below:

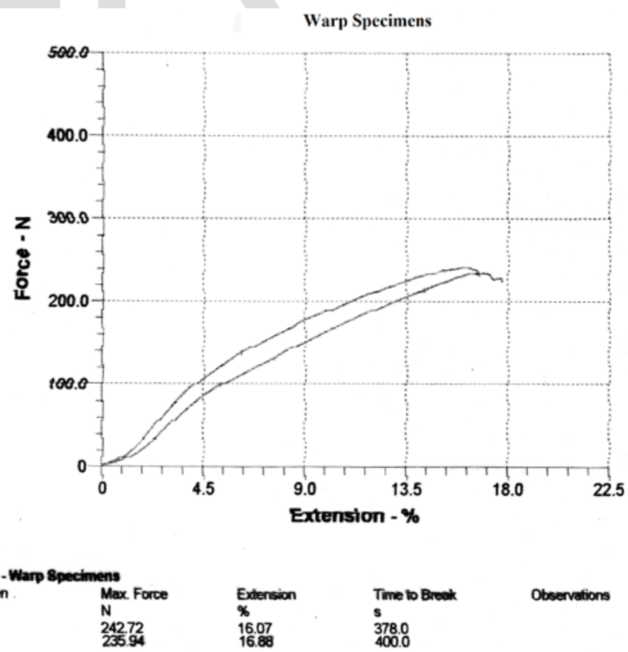

Figure 6: Graphical Representation of Tensile Strength of Raw Silk

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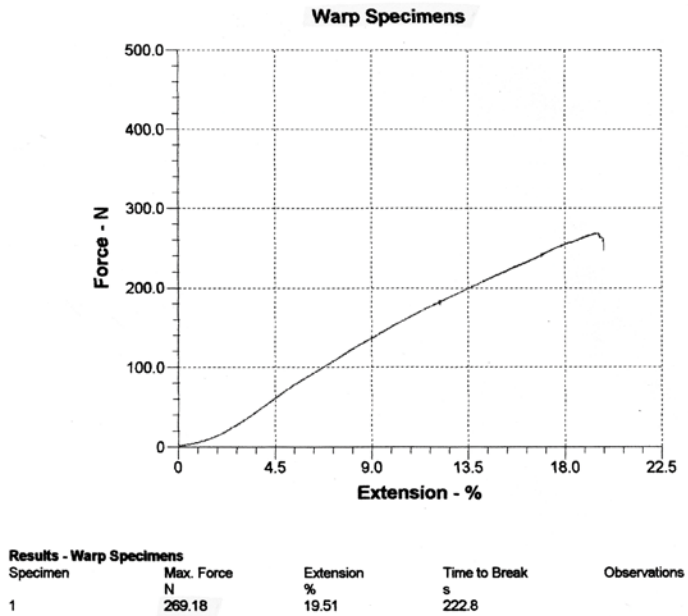

Figure 7: Graphical Representation Tensile Strength of Degummed and Bleached Silk

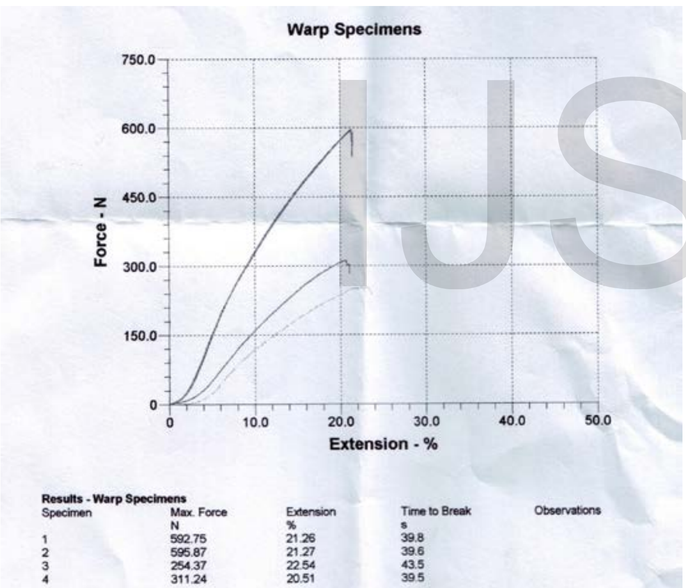

Figure 8: Graphical Representation of Tensile Strength of Acid dyed silk (1&2) and Reactive Dyed Silk (3&4) fabric

TABLE 19: DATA ANALYSIS OF TENSILE STRENGTH (N) AT DIFFERENT STATES OF THE SILK FABRICS

Different States	Data 1 (Max. Force N)	Data 2 (Max. Force N)
Raw Silk	242.72	235.94
Degummed and Bleached Silk	269.18	-
Acid Dyed Silk Fabric	592.75	595.87
Reactive Dyed Silk Fabric	254.37	311.24

From TABLE *19* it can be seen that, the tensile strength is increased in every step of processing but in case of reactive dyed sample (254.37 N, 311.24 N) the tensile strength is comparatively very poor than acid dyed sample (592.75 N, 595.87 N).

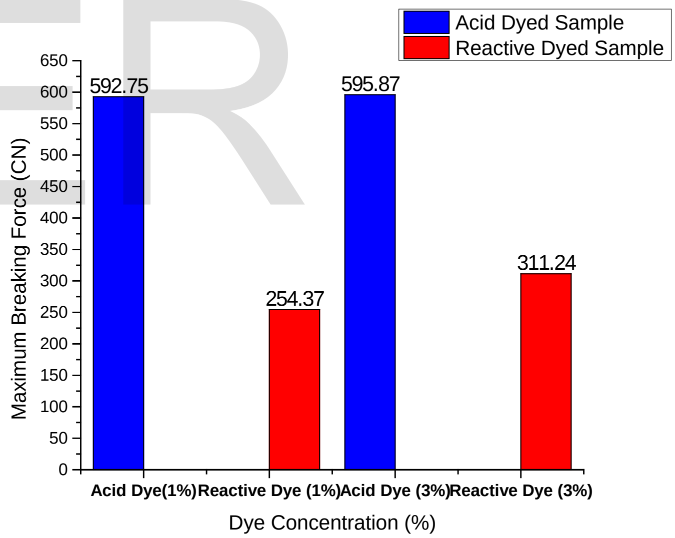

Figure 9: Maximum Breaking Force vs. Dye Concentration

From Figure 9 it can be seen that, with the increase of Dye Concentration % the Maximum Breaking Force has been increased for both acid and reactive dyed samples but in case of reactive dyed sample it is comparatively poor than acid dyed sample.

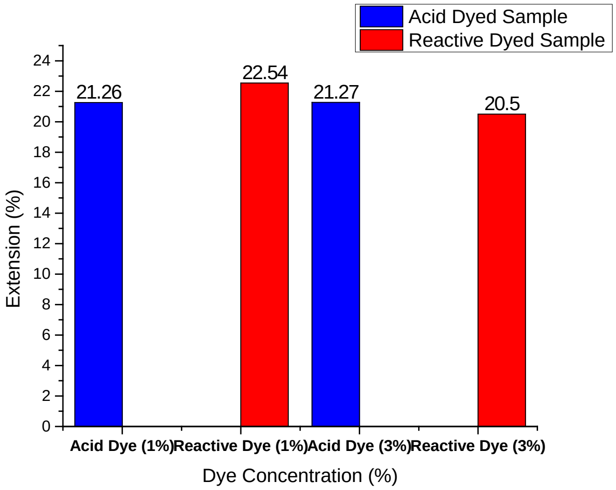

Figure 10: Extension vs. Dye Concentration

From Figure 10 it can be seen that, with the increase of Dye Concentration % the Extension % does not show any significant changes for both acid and reactive dyed samples.

4. Conclusion

This paper contains two parts, one part contains the processing of raw silk and another part contains the comparative consequences from Acid dyed and Reactive dyed Silk woven fabrics.

Comparing the consequential data of different tests for acid dyed and reactive dyed silk fabrics, it has come that, application of reactive dye on silk fabric gives satisfactory result comparing to acid dyed samples. For light shade reactive dyed samples were relatively dull than acid dyed samples of same shade % but with the increase of shade % the depth of color increased for reactive dyed samples comparing with acid dyed samples which indicates well fixation of reactive dyes on silk fabric. In case of wash fastness test, reactive dyed samples showed even better result than acid dyed samples. In case of other fastness tests like rubbing and perspiration tests reactive dyed samples gave almost similar and somewhere way better results than acid dyed samples.

In case of tensile strength the comparative analysis showed that reactive dyed samples tears at low force (N) than acid dyed samples, in case of acid dyed samples the tensile strength is increased like twice that raw silk, but in case of reactive dyed sample the increase percentage of tensile strength is not that much. In case of elongation % there is not much difference between acid dyed and reactive dyed samples. Treating silk in alkali medium during reactive dyeing process is the possible cause for loosing or not gaining that much strength like acid dyed samples.

Although with few obstacles, this experiment came out with great prospect for silk dyeing with reactive dyes and lead to a way for reactive dyes to be a well substitute of acid dyes.

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