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EFFECT OF CROSS LINKING AGENTS ON THE IMPROVEMENT OF VISCOSE FABRIC STRENGTH

Salma Katun Sela[1] **and A. K. M. Nayab –Ul- Hossain**[2]

[1]*Department of Textile Engineering, Jashore University of Science & Technology, Jashore, Bangladesh*
[2]*Department of Textile Engineering, Khulna University of Engineering & Technology, Khulna, Bangladesh*

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ABSTRACT

Viscose is a regenerated cellulose fiber and in wet condition, its strength becomes less which can be ameliorated by using specific chemical treatment. Different cross-linking agents in different quantities were applied on viscose fabric after dyeing & the physical properties like GSM, absorbency and tear resistance were measured. Strength like tear resistance of the finished viscose fabric was ameliorated in comparison to the grey, pretreated and dyed viscose fabric. After the application of 10 g/l cross linking agent, tear resistance of dyed fabric was increased at maximum amount for the RucoPur SLR, Fornax W. For the cross-linking agent like Ruco-LubKmn400 & Ultratex SI, the application amount for the maximum strength improvement was 60g/l. When Turpex ACN was applied, the maximum strength improvement was at 30 g/l. The changes were also observed by understanding the morphological view using scanning electronic microscope (SEM). Safety issue of the applied chemicals carcinogenic arylamines was also tested. The strength of viscose was enhanced in this study though the fabric was treated in wet condition.

Keywords: *Viscose; strength; SEM; carcinogenic arylamines*

1. INTRODUCTION

There are three types of man-made regenerated cellulosic fibers such as: viscose, cuprammonium viscose (cupro) and sponified cellulose acetate. The international organization for standardization defines these terms in its standard ISO-2076-1977. But here "viscose" is the generic name. International organization for standardization does not use the name viscose. It states in ISO 2076-1977(E) "this name, used generically for regenerated cellulose fibers in some countries, doesn't have the same meaning everywhere. The organization suggests that the name "viscose" either be used or not. It can be used in one of its national standards. Here, the generic name "viscose" adopted by the U.S federal trade commission for fibers of the regenerated cellulose type. Viscose is regenerated cellulosic fiber composed of regenerated cellulose in which substituent have replaced not more than 15 percent of the hydrogen of the hydroxyl groups. Xu *et al. (*2006) studied an investigation to explain the characteristics of regenerated cellulosic fiber (viscose) and bamboo viscose fibre.

Viscose is regenerated cellulose fiber obtained from wood pulp or cotton linters, which are the source of pure natural cellulose, without any change in chemical constitution in polymer but only with certain variation in degree of polymerization and as well as with modified physical properties based on the necessity. Morehead *et al.* (1945) suggested a plausible mechanism of skin formation of viscose. Hermanst (1948) observed that the skin exhibiting properties towards dye are the same for lilienfeld –process viscose and ordinary viscose. The filament of viscose is very smooth and straight. But there are no convolutions as in cotton. Ordinary viscose has a tenacity of 18-23 cn/tex. But the filament of viscose or staple fiber are weaker because of it's very amorphous region and it's have a fair tenacity. Stepanik *et al.* (1998) experimented electron-treated pulp for viscose preparation which reduced production cost. When viscose fiber is wet, it is half strong than in dry condition. The reason for its very amorphous nature which readily permits the entry of water molecules. Buschle-Diller *et al.* (1994) studied the fall of strength for viscose was significant and also the weight loss.

Viscose has small elastic stretch than cotton and other natural cellulose fiber. When the viscose fiber is put under strain, the very amorphous region and fewer hydrogen bonds of viscose, enable the polymers to slide past each other. But after removing strain the polymers don't return to original position. Viscose is the most common man-made fiber which is not thermoplastic. It has a lower heat resistance and poorer heat conductivity than cotton. Vesel *et al.* (2009) observed a plasma treatment of viscose including chemical changes of the sample. For the shorter polymers & the very amorphous nature of the regenerated cellulose fibers are responsible for the much greater sensitivities of these fibers to acids, alkalis, bleaches, sunlight with regard to dyeing and printing. The regenerated cellulose fibers generally bright in color. Hindeleh *et al.* (1974) investigated a procedure to determine crystallinity, crystallinity size of cellulose II and cellulose IV in viscose. Lenz *et al.* (1993) explained fibrillability of viscose fibre which is suspended in water is positively correlated with the factor of crystallite orientation. In the man-made fibre field, viscose plays a role similar to that of cotton in the field of natural fibres. It is produced in greater quantity than any other man-made fibre; it is relatively cheap and has a wide

*Corresponding Author: shila_te@just.edu.bd

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range of applications. Huang *et al.* (2007) examined that viscose based activated carbon fibre was incised and also rougher.

Although viscose is similar to cotton in its cellulosic structure, it provides a range of yarns and fabrics with their own characteristic properties. Guan *et al.* (1998) prepared a blend of chitosan & viscose and observed that the addition of chitosan can improve compatibility into the blend. Chen *et al.* (2006) made a study of incorporation fire –retardant properties to viscose. The cellulose of viscose has been modified to some degree during manufacture, and the alignment of the molecules is not identical with that of natural fibres such as cotton. Also, the fact that viscose is a manufactured material enables to control the physical characteristics of the final product. Viscose can be made coarse or fine, its strength and elasticity, luster and color can be controlled. Moreover, as a manufactured material, viscose is not subject to changing economic and climatic circumstances such as those that affect the properties and price of a natural fibre. Viscose conducts heat more readily than silk does, and the viscose has a cooler feel against the skin. Viscose is also highly absorbent, and this enhances its value as a clothing material. Engström *et al. (*2006) studied enzymatic pretreatment to enhance the accessibility of cellulosic material. The loss of strength which viscose undergoes when wet is probably its most serious shortcoming, but modern resin finishes have done much to overcome this problem. Properly finished viscose garments have high dimensional stability when wet. The introduction of viscose staple has enabled manufacturers to blend viscose with other natural and synthetic staple fibres, and viscose staple is used very largely in this way. Viscose contributes its moisture absorption and other cellulosic characteristics to blends of stronger and less absorbent fibres, including most of the synthetics.

Blends of viscose with other fibres may be processed by any of the familiar techniques. Staple lengths are provided to suit blends and systems; a 50 mm staple, for example, can be handled on cotton machinery and a 100 -150 mm staple may be used with wool. Babaarslan (2001) studied that central part positioning had an effect on the structure, properties and performance of polyester viscose core spun yarn along with spandex. Viscose is chemically almost identified with cotton. It can be dyed with all cotton dyestuffs, but techniques of dyeing are influenced by special factors. Viscose has a greater affinity for dyes than has cotton. Affinity may vary according to manufacturing conditions and will not necessarily be identical in viscose produced from the same plant. Viscose swells to a greater extent than cotton when it is immersed in water. The fibres are weakened, and yarns or fabrics must be handled with great care in dye bath. Viscose is dyed in the form of hanks, staple fibre, and piece goods. The high degree of swelling can cause difficulties when viscose is dyed in packaged form. Reactive dyestuffs which chemically bond to the cellulose molecules are often used. Viscose swells in the alkaline solutions used in dyed with dyestuff. And the leuco compounds are absorbed more rapidly than in case of cotton. Kumbasar *et al.* (2000) studied application of the mixture of three anionic thickeners in reactive dye printing on viscose. Viscose fibre will withstand the caustic soda solution used in process like mercerization but disintegrates during subsequent washing. Special techniques are used to overcome this problem, including the use of caustic soda, or mixtures of caustic potash and caustic soda, in place of caustic soda.

The finishing of viscose fabrics is concerned largely with minimizing the shortcomings inherent in viscose, notably its sensitivity to water. Resin finishes of many types are now used effectively for this purpose, providing increased dimensional stability during washing, improved wrinkle resistance and crease resistance. These finishes should be used with care, as they may cause loss of tear strength and produce a harsh, broadly handle. Emam *et al*. (2014) made a study where used nano particles for the production of anti-bacterial colored viscose fibres. In general, viscose fabrics wash like cotton; they are cellulosic fibres. But viscose goods are much less strong than cotton, especially when wet, and this must always be remembered when viscose garments are laundered. The use of resin finishes has done much to increase the dimensional stability of viscose fibres when wet, but it is always necessary to take great care when wet viscose fabrics are being handled. Viscose fibre itself does not shrink appreciably, but a woven fabric may undergo progressive shrinkage, even when it has been treated with a resin finish to provide dimensional stability. Much depends upon the way the cloth has been constructed. The strength of viscose is a vital issue especially in wet condition. When the viscose will be treated with chemicals in wet condition, this is more concern to degrade its strength. Tear resistance test on fabric or tear strength is measured to check how the material can withstand the effects of tearing or cuts when in tension. Force required propagating and existing tear is measured. Normally strength properties of viscose fabric are poor. In this study five cross linking agents were used at different proportion to improve the tear resistance of viscose fabric and to understand their performance. At the same time, the safety issue of all the applied cross-linking agents was measured. Reactive dyes are dyes utilized for dyeing protein, cellulose and polyamide fibre and they are anionic. It becomes associate degree integral a component of the fibre because of the formation of a chemical bond between the reactive cluster of this dye and additionally the fibre compound. Reactive dyes dissolves in di-hydrogen monoxide in contrast to VAT dyes. They'll shield against the degrading impact of ultra-violet rays because of glorious lightweight expeditiousness and stable lepton arrangement. There square measure 2 forms of reactive dyes: sultry (monochlorotriazine dyes) and cold brands (dichlorotriazine dyes).

Coloring may be done at temperature as a result of algid brands becomes a plethora of reactive because of the presence of 2 chemical element atoms. Here, reactive dyes were used to dye viscose fabric & later on cross linking agents were also applied on dyed fabric to retain strength & to understand the effect of the chemicals in ameliorating strength.

2. WORKING PROCEDURE

At first pretreatment was carried out. All the chemicals & auxiliaries were transferred into the machine &the temperature was raised to 100^0C& during the raise of the temperature, NaOH and H_2O_2 were provided to the machine by dosing system. At 100^0C, the operation was carried out for 45-60 min. After that the fabric was washed properly. Neutralization was carried out to the fabric by using CH_3-COOH at 1 g/L.

Table1: Pretreatment recipe

Name of chemicals	Used amount (g/L)
Detergent	1
Sequestering agent	0.8
Stabilizer	1
Anticreasing agent	0.5
NaOH	3
H_2O_2	10

After this operation, the impurities present on the fabric was removed & became hydrophilic as well as the natural color of viscose fabric was destructed and a pure, permanent, basic white effect was incorporated in the fabric. When the fabric was fully pretreated, dyeing was carried out using reactive dyes. At first the dye bath was set at 60^0C. Dyeing chemicals & auxiliaries were transferred to the machine. Glauber salt was applied into the machine by 30 minutes dosing system for precaution of viscose fabric. Then the machine was in running condition so that the salt can be uniformly distributed on the fabric. Dyes in solution form were entered into the machine by 40 minutes linear dosing system. Then the machine was kept for running for another 30 minutes. After that caustic soda was applied into the machine by progressive 4 dosing system for 50 minutes. The shade was checked under light box using D65 light source.

Table 2: Dyeing recipe

Name of chemicals	Used amount (g/L)
Sequestering agent	1
Leveling agent	2
Novacron Yellow S3R	5
Novacron Ruby S3B	5.6
Novacron Navy WB	36
Glauber salt	40
Soda Ash	18

Flow chart of application of cross-linking agent is as follows:

Set the application bath temperature at 40^0C.
↓
Mixing of cross-linking agent with softener
↓
pH was adjusted by the addition of acetic acid
↓
After the completion of mixing, the mixed chemicals
were applied on the dyed fabric by padding system at 60^0C.
↓
The sample was dried at mini stenter at 130^0C for 90 sec.

After draining the dye bath water, proper washing was carried out to make the water in the machine clean prior to neutralization. Neutralization was done by using 1 g/L acetic acid. Hot wash was applied using soaping chemical at 90°C. Different types of cross linking agents like Ruco-pur SLR which was Reactive polyether polyurethane type, Ruco-lub KMN 400 was Polyethylene type, Ultratex SI was polydimethylsiloxane type, Fornax W was colloidal polysilicic type &Turpex ACN was colloidal polysilicic type, were applied on the fabric. A variation of quantity of different individual chemical was applied on the fabric. Dyed viscose fabric was treated with three different amounts of quantity like 10g/L, 30g/L, and 60 g/L of the individual chemical. These chemicals were individually applied at 60°C at acidic medium. pH was adjusted by using acetic acid of

0.6-1 g/L. After the application of the chemicals, the viscose fabric was passed through stenter machine in order to basically dry the fabric. Then the fabric was gone through compacting machine to control shrinkage. The application curve of cross-linking argents indicates the similar procedure as mentioned in the flow chart. In the curve, the X-axis indicates the time & Y-axis indicates temperature.

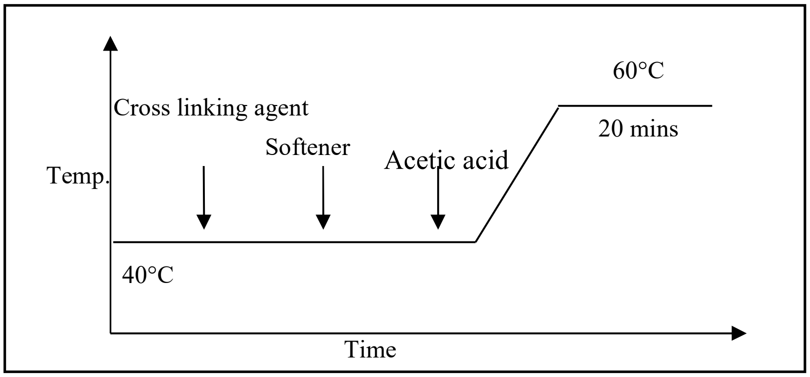

Figure 1: Cross linking agent application curve

3. RESULTS AND DISCUSSION

At first the morphological view of pretreated, dyed & different finished fabric was observed under Scanning Electronic Microscopic (SEM) machine. Pretreated viscose fabric, dyed fabric & the finished fabric treated with different cross-linking agents were checked under scanning electronic microscope to understand the morphological change. Pretreated viscose fabric amorphous region was more because the treatment was in wet condition. Amorphous region % became increase for the dyed fabric as it was processed in wet condition along with chemicals. When the dyed fabric was finished with cross linking agents, the amorphous % became less which ultimately increases the crystallinity of the fabric. Compactness of the fiber was observed for all the fabric finished with RucoPur SLR, RucoLub KMN-400, Ultratex SI, Fornax W, TurpexACN.

All the finished fabric morphological views indicate that the compactness or crystallinity of the fiber increased which ultimately positively affect the strength of the viscose fabric & the crystallinity of finished viscose fabric is more in comparison to the grey, pretreated & dyed viscose fabric. Cross-link could be a bond that links one compound chain to a different. These links might take the shape of valence bonds or ionic bonds and therefore the polymers will be either artificial polymers or natural polymers (such as proteins). Cross-linking typically refers to the employment of cross-links to market a modification within the polymers' physical properties.

Cross linking is that the general term for the method of forming valence bonds or comparatively short sequences of chemical bonds to affix 2 chemical compound chains along. The term solidification refers to the cross linking of thermo set resins, like unsaturated polyester and epoxy. Once chemical compound chains square measure cross linked, the fabric becomes additional rigid. An artificial compound is claimed to be "cross-linked", it always implies that the whole bulk of the compound has been exposed to the cross-linking methodology. The ensuing modification of mechanical properties depends powerfully on the cross-link density. Low cross-link densities decrease the viscosities of compound melts. Intermediate cross-link densities rework sticky polymers into materials that have elastomeric properties and probably high strengths. Very high cross-link densities will cause materials to become terribly rigid or glassy, like phenol-formaldehyde materials. Where chemical cross-links area unit valence bonds, physical cross-links area unit shaped by weak interactions. In this way, the cross-linking agents used here cross linked with the fiber.

Cross linking agents create chemical bond between polymer molecule to form a three-dimensional network in the fiber. This generally restricts swelling in inhibits solubility and alters elastic recovery. Cross linking agent react with this fiber here as follows:

cell – OH + cross linking agent + OH – ⟶ cell cross linked cell + $2H_2O$

From the SEM test result, this was illustrated that the crystallinity was maximum when the fabric was treated with Ruco-pur SLR. For the fabric treated with Ultratex SI, the increased crystallinty was second most. The increased crystallinty was third most for Ruco-LubKmn 400 and then the performance of the crystallinty was increased at maximum amount, it subsequently increased the compactness of the fabric as well as the improvement of strength.

In grey stage the tear resistance of grey fabric was 4.15 N in warp direction & 3.2 N in weft direction. Strength test like tear resistance as well as GSM, absorbency was tested for the pretreated fabric. Tear resistance was checked in both warp & weft direction. In warp direction, the tear resistance was 4.05N & in weft direction, the tear resistance was 3.12N for pretreated viscose fabric.

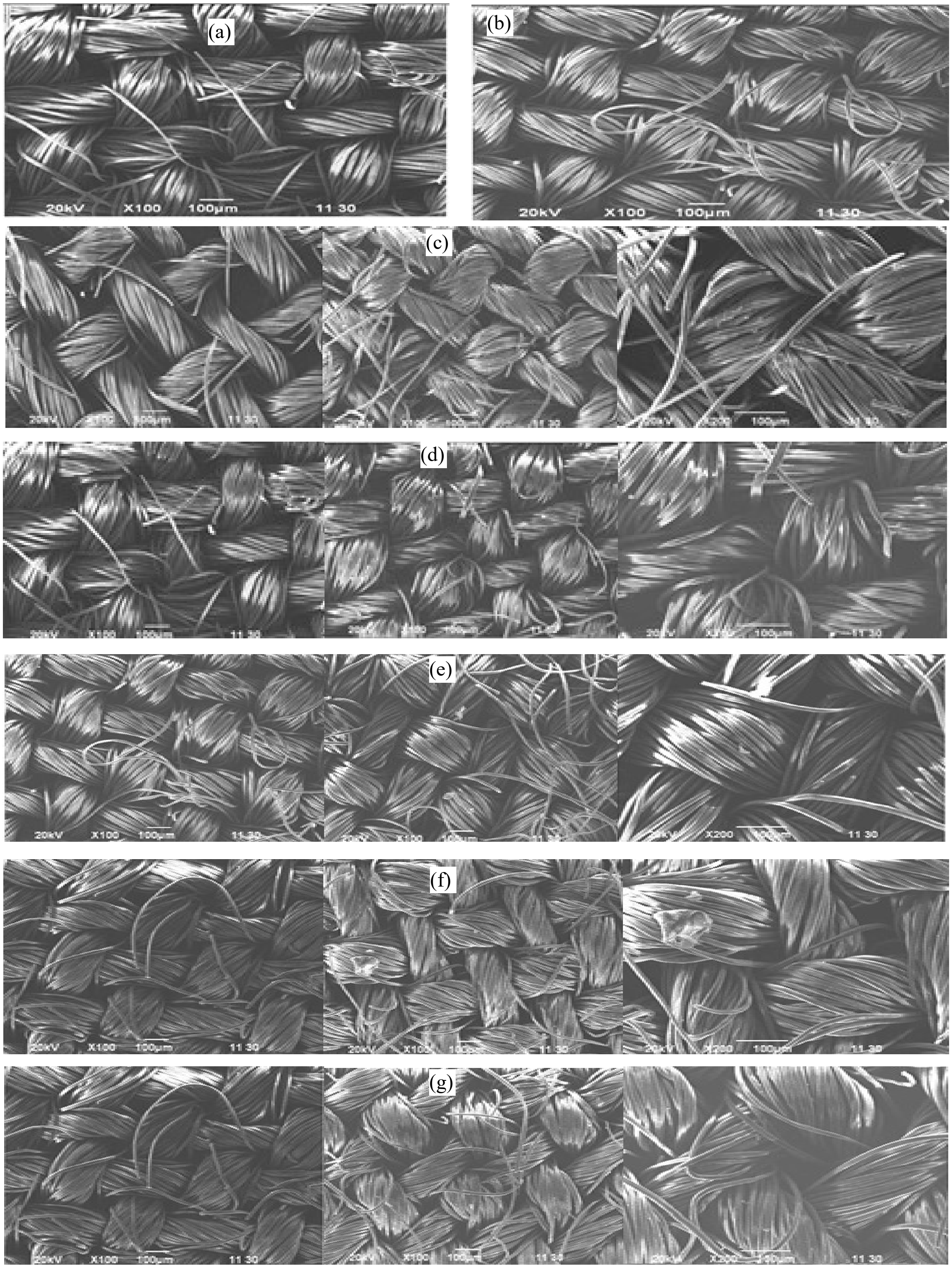

Figure 2: Microscopic view under SEM of a) pretreated, b) dyed and 3 different view of fabric finished with c) RucoPur SLR, d) RucoLub KMN-400, e) Ultratex SI, f) Fornax W, and g) Turpex ACN.

Table 3: Test result chart for different kind of tests of pretreated viscose fabric

Test name	Test method	Direction	For pretreated fabric
GSM	ASTMD 3776		85 g/m^2
Absorbency	Column Test		3.8
Tear	ENISO 13937	Warp	4.05N
resistance	2000 pts. 2to4	Weft	3.12N

After dyeing, the strength was also checked for the dyed fabric. Dyed viscose fabric tear resistance in warp direction was 4N & in weft direction was 3N which was indicated that the strength was slightly decreased in dyed fabric. This was illustrated that the tear resistance of pretreated fabric was less than the raw/grey viscose fabric & it was decreased more in dyed fabric than the pretreated fabric.

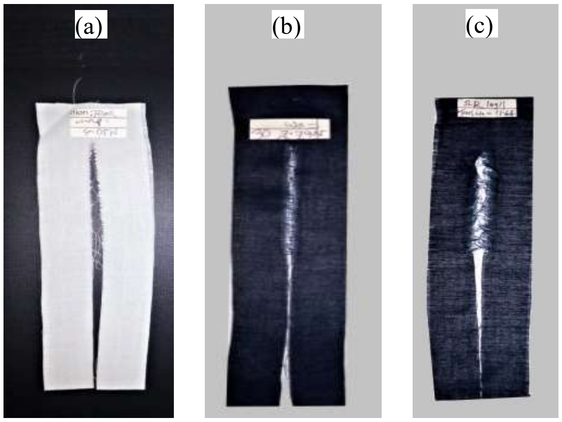

Figure 3: Tear resistance test of a) pretreated, b) dyed, c) finished fabric

Due to water sensitivity of viscose fabric & its amorphous region, the strength of viscose fabric fell in wet condition. For dyed fabric, the strength fall was more than that of pretreated fabric & for pretreated fabric, the strength fall was also more than that of grey fabric. In grey stage the tear resistance of grey fabric was 4.15 N in warp direction & 3.2 N in weft direction. Due to water sensitivity of viscose fabric & its amorphous region, the strength of viscose fabric fell in wet condition. For dyed fabric, the strength fall was more than that of pretreated fabric & for pretreated fabric, the strength fall was also more than that of grey fabric.

Table 4: Test results of dyed viscose fabric

Test name	Test method	Direction	For dyed fabric
GSM	ASTMD 3776		87 g/m^2
Absorbency	Column Test		3.8
ear	ENISO 13937	Warp	4.0 N
resistance	2000 pts. 2to4	Weft	3.0 N

The cross-linking agents were applied in different quantities to the dyed viscose fabric. After the application, the tear resistance was checked for all the finished fabrics. This was shown from the (Table5) that tear resistance of the finished fabric was increased in every case in respect to the dyed & pretreated fabric. Tear resistance for the finished fabric with the application of Rucopur SLR was more both in warp & weft direction using 10 g/L. For the finished fabric with the application of Ruco-LubKmn 400, the tear resistance both in warp &weft direction was more for the application quantity 60 g/L. The similar result found for the finished fabric with the application of Ultratex SI. In this case, the tear resistance was more both in warp & weft direction for the application quantity 60 g/L. The finished fabric with the application of Fornax W, the tear resistance was more both in warp & weft direction for the application quantity 10g/L. Finally, the fabric, which was finished with Turpex CAN, the tear resistance was more both in warp & weft direction for the application quantity 30 g/L. So, tear resistance was increased with the application quantity 10 g/L for the fabric finished with Ruco-pur SLR, Fornax W. The finished fabric with application of Ruco-LubKmn 400 60 g/L the tear resistance was more, Ultratex SI. Tear resistance became more with the application quantity 30 g/L for the finished fabric treated with Turpex CAN. This was illustrated that tear resistance was increased for the finished fabric treated with cross linking agents than that of the grey, pretreated and dyed viscose fabric.

Table 5: Comparison of tear resistance test of finished viscose fabric

SL no.	Chemical name	Unit	Direction	Amount		
				10 (g/L)	30 (g/L)	60 (g/L)
01	Ruco-pur SLR	Newton	Warp	11.66 N	7.79 N	7.75 N
			Weft	12.14	7.67	7.56
02	Ruco-LubKmn 400		Warp	8.34	7.71	8.60
			Weft	7.07	9.09	8.75
03	Ultratex SI		Warp	7.39	9.49	9.63
			Weft	7.73	9.76	9.84
04	Fornax W		Warp	4.56	3.82	3.05
			Weft	3.80	3.0	3.74
05	Turpex ACN		Warp	8.23	8.35	6.90
			Weft	7.49	8.37	7.40

FTIR Spectroscopy is a nondestructive method for studying the physico-chemical properties of Fibrous materials. The FTIR spectrum of untreated and treated viscose fabric is shown in Figures 4 and 5 respectively. The FTIR spectrum of untreated & treated viscose sample show peaks at 3373.12- 3422.26 cm^{-1}can be assigned to C-H stretching. The broad shoulder around 3383.14 cm^{-1} for untreated sample and 3381.21cm^{-1} for treated sample can support the existence of C-H stretching. FTIR spectrum shows pick at 1036.42-1382.17 cm^{-1}can be assigned to the existence of C-O bond. The broad shoulder around 1159.22 cm^{-1} for untreated sample and 1363.76cm^{-1} for treated sample can support the existence of C-O bond. In both samples FTIR spectrum picks at 1028.06cm^{-1} can be assigned to the vibration of C-O in alcohol hydroxyl group.

FTIR spectrum shows peaks at 2918.20-2954.49 cm^{-1} can be assigned to the presence of C-H bond. In treated sample (Figure 5), the FTIR spectrum peak at 2927.94 cm^{-1} which strongly indicates the presence of strong C-H bond. The presence of C-H bonds ameliorates the internal strength of fabric which has been happened here of treated viscose sample.

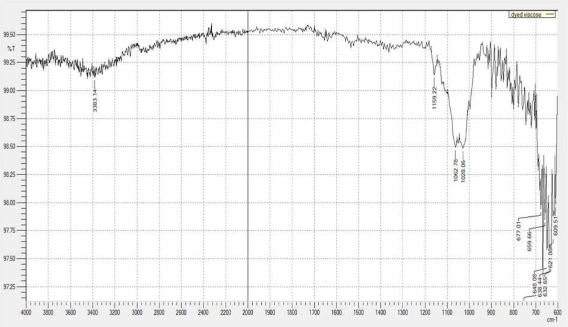

Figure 4: Untreated viscose fiber

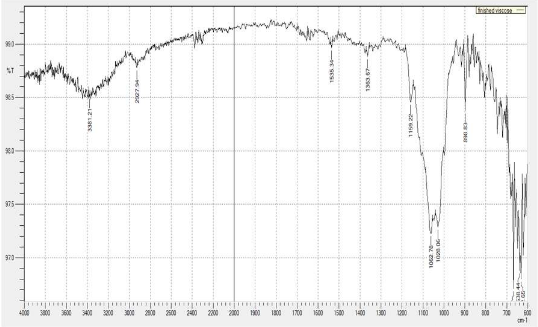

Figure 5: Treated viscose fiber

So, the strength of viscose fabric can be retained after pretreatment as well as dyeing. Safetssue like Carcinogenic Arylamines (use of Banned Azo Colourants) test was also done for the applied chemicals & it was not found in all the finished fabric.

4. CONCLUSIONS

Generally viscose strength falls due to its water sensitivity & amorphous region. Due to the presence of hydroxyl group present in viscose, it can react with the chemical containing functional group. If the cellulose molecules of viscose fabric can cross link with chemicals, it reduces the freedom of movement of the molecules with respect to each other and has the effect of reducing water absorption and swelling in water and alkali and increasing the wet initial modulus. Here different cross-linking agents were used for this purpose as well as to retain the strength of viscose fabric which would be treated in wet condition. When the dyed fabric was treated with different types of cross-linking agents, the strength like tear resistance was increased in the finished fabric in comparison to the grey and pretreated, and dyed viscose fabric. Performance of different cross lining agents was evaluated, and the strength i.e. tear resistance was increased most when the fabric was treated with Ruco-pur SLR at application amount of 10 g/l because it increased the maximum amount of crystallinity of the viscose fabric. All the applied chemicals were safe as Carcinogenic Arylamines test was done. This improvement in resistance is reflected in increased fabric quality. So, strength of viscose can be ameliorated by using cross linking agents.

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