Improvement of Dye Uptake of Indigo and Sulphur Dye in Denim Dyeing and Their Strength Percentage Evaluation in Different Concentration of Alkali Pretreatment

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Abstract

Indigo dyes and Sulphur dyes are measured as very fashionable for clothes as they're having sensible overall fastness properties. Application of Indigo dyes and Sulphur dyes needs an awfully concentration of Caustic soda in the time of pretreatment process. The concentration of Caustic soda plays an important role in denim dyeing. Controlling the concentration of Caustic soda which is used in the pretreatment bath in denim dyeing, increased the dye uptake on warp yarn, good colorfastness, strength, lustrous and it ensured to achieve demanded color efficiency (depth of shed) with the use of lower dyestuffs compared to without controlling the concentration of Caustic soda in the time of pretreatment in denim dyeing. The optimum value was measured accurately because using data color spectrophotometer for measuring color strength and other test values were found from laboratory instrument of Nice Denim Mills Ltd. Scouring and dyeing process is carried out by Padding Mangle. Maximum dye uptake increase was observed at 270 g/l for Indigo Dark Shade, Indigo Light shade, Indigo Medium Shade, Sulfur Dark Shade, Sulfur Light shade. Also, these findings evidenced that tensile strength could be Decreased in this concentration. The optimum concentration of strength is 60g/l for light to medium shade and 90 g/l for dark shade.

Key Words: *Caustic Soda, Cotton yarn, Dyes, Color strength, Yarn strength.*

1. Introduction

In Bangladesh- denim is a very rising sector. But it's a matter of regret there is no well experimental details are available in the literature for the choice of NaOH concentration to be used in pretreatment before denim dyeing. Denim warp yarns travel through scouring baths containing wetting agents, detergents, and sodium hydroxide just prior to the dyeing process. This phase tries to increase the absorbency and whiteness of textile materials by eliminating non-cellulose natural substances, such as lipids, waxes, pectins, and proteins, from the fibers via chemical, physiochemical, and structural alterations to the cellulose (Holme, 2016). Native cellulose (Cellulose I) becomes alkali cellulose I with concentrated sodium hydroxide, while cellulose II is produced after washing and neutralization (Aman, 2013).

As a result of the alkali penetrating the lattice, internal hydrogen bonds are disrupted, and the amount of accessible hydroxyl groups (-OH) in Cellulose II increases by approximately 25%. The alkali treatment and subsequent washing can be carried out such that the fabric or yarn can either contract freely or remain under tension. In both instances, mercerized cotton has an enhanced attraction for both reactive and direct cotton dyes, as well as indigo colors, as well as improved strength and affinity for water. On the fiber level, "Swelling", cross-sectional morphological changes from beam shape to round shape, and Shrinkage occur along the longitudinal direction during Mercerization. At the molecular level, there will be hydrogen bond readjustment, orientation (parallelization) of molecular chains in the amorphous area along the

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fiber length, and orientation of the crystallinity along the fiber length. At the chemical level, chemical changes occur by accelerating the reaction rates of hydrolysis and oxidation; heat is released during caustic treatment (Kiron, 2021).

This research has been done to improve and investigate the dyeability of Indigo and Sulphur dyes on cotton fabric, especially on Denim fabric. Different concentrations of Caustic soda have a different effect on the dyeing kinetics of Denim dyeing. . The Pre-Treatment and Causticization of Cotton Fabric for Enhanced Dye Absorption. It is feasible to prepare a grey cotton fabric for dying and finishing in a single step at a low cost and in a short amount of time (Hosseini, 2013). But in much research has been showing that proper Mercerization with Caustic soda may improve the dye take up. The optimal concentration of caustic soda for efficient mercerization is 25%, which generates the best degree of mercerization and mechanical qualities rather than the highest dyeability based on dye-absorption values (Sameii, 2008). Causticization for improved color yield can be obtained at 14-16% caustic soda concentration which is in agreement. They using the Design Expert Software for accurate results. They also show that Wet-on-wet dyeing gave better color yield compared to dyeing after drying. By their experiment, causticization was carried out on padding mangle and dyeing carried out on jigger machine. They applied causticization of caustic soda on cotton fabric and dye used direct dye (Khalifa, 2017).The dye take up ability may increase by doing treatment with caustic soda both in scouring and mercerization process. Observations indicate that mercerization treatments reduce the cellulose crystallinity index, with the degree of decrease varying according to mercerization settings. Low-temperature mercerization could boost dye uptake. Maximum dye uptake increases were seen at 35 °C for slack mercerized fabrics and 65 °C for tension mercerized fabrics, which were approximately 34 and 28.2% more than un-mercerized cloth (M. Abate, 2017). Many kinds of research have been done on the treatment of fabric with caustic soda to improve the dye take-up ability. But very little researches were done on Denim fabric. We know from much literature that mercerization on Denim fabric (100% cotton) may improve its physical and chemical properties. Generally, it is observed that 20 to 30% concentrated caustic soda at 15 to 18 ^{0}C may increase fiber swelling and shrinkage (JO, 1966)(Spencer, 2008)(Willows, 2016)(Bancroft, 1934)(Neale, 1935)(Goldthwait, 1965).It also increase surface luster of fiber (Spencer, 2008)(Nodder, 2016)(Lyman Fourt A. M., 1951)(Lyman Fourt H. E., 1955), polymer modification and modification in the crystallinity of fiber structure (Lal, 1974) (T. Okano, Mercerization of cellulose. I. X-ray diffraction evidence for intermediate structures, 1984)(T. Okano, Mercerization of cellulose. II. Alkali–cellulose intermediates and a possible mercerization mechanism, 1985),(Hisao Nishimura, 1987), (Menachem Lewin, 2017), (Howard J. Philipp, 1947), somewhere it helps to gain strength also (Tamane Wagaw, 2012), (Wakeham & Spicer, 2019), (B.R. Shelat, 1960), (S. H. Zeronian, 1976), (Jacques J. Hebert, 1973), (J. W. S. Hearle, 1979).Proper causticization also helps to increase dye take up ability, moisture absorption and reduce the immaturity in the fiber (Lenore Cheek, 1987), (MORTON, 1976), (Boer, 1962), (Lewin, 2006), (Karmakar, 1999), (Staff, 1992), (Dr. S.M.Imtiazuddin, 2010), (Saeeda, 2016). From this literature, it is clear that treatment with caustic soda improves dye take-up of Denim fabric. Also, Caustic soda improves the dye take-up ability of fabric dyed with Indigo and Sulphur dyes (Dr. S.M.Imtiazuddin, 2010), but this research did not clearly indicate the strength loss of denim fabric due to the excessive use of caustic soda and also structural changes due to treatment with caustic. To optimize these lacking, this research has been done to observe the dye

take up ability with changes in the strength of the fiber after treatment with different concentrated caustic soda in the pre-treatment stage. Also to find out the optimum rate of caustic soda to use in the dyeing of Denim fabric with Vat dyes and Sulphur dyes with better strength.

2. Experimental

2.1. Materials

All yarns are collected from local spinning industries Ismail spinning LTD & Noman spinning LTD. In this experiment different counted yarn such as 7, 10, 12, 16 & 18 were used. Chemicals and dyestuffs used in this experimental work are presented in ***Table 1***.

Table 1: List of dyes and chemicals used in this experiment

Sl no	Name of chemicals	Type	Company	Origin
1	Denisol Indigo 30	Dyes 305 liquid	Archroma	Spain
2	Diresul Black RdT DBD	Sulphur dyes liquid	Archroma	Spain
3	Caustic Soda	Sodium hydroxide flakes with 97%	Chinese	China
4	PrimasolNf	Anionic Wetting agent with non-foaming agents	Archroma	Spain
5	Kieralon EH	Anionic wetting agent with high cloud point.	Archroma	Spain
6	Mercerol QWH	Anionic mercerizing agent with high caustic conc.	Archroma	Spain
7	Sodium Hydrosulphite	Reducing agent for indigo	BASF	Germany
8	Secron	Sequestering agent	Pulcra	Turkey
9	Reductant powder D	Reducing agent glucoseforSulphur black	Archroma	Spain

2.2. Methods

This research has been done in the continuous Denim dyeing machine of Nice Denim LTD Bangladesh. All the experiment for this research has been done under laboratory graded standards. All purity of the chemicals and dyes was tested in the laboratory of the Nice Denim LTD. In this research work, several steps had been followed sequentially to make the research successful. The research work had been done as the given below flow chart in ***Figure 1***.

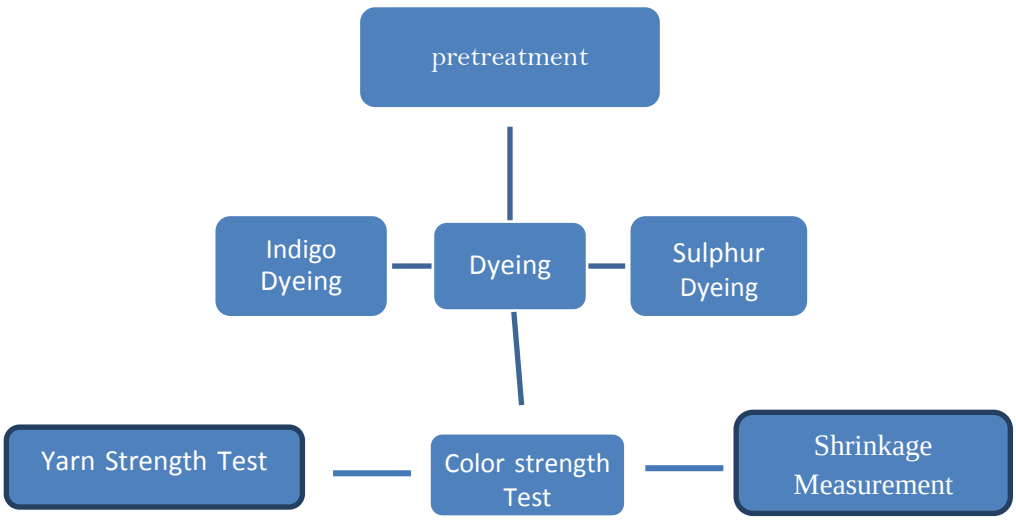

Figure 1: Method of denim dyeing

2.3. Pretreatment process with caustic soda solution

The caustic soda flasks used in the experiment were laboratory graded. Based on the result of the strength of caustic soda, the yarns of the samples were treated with varying caustic soda concentrations from 20 g/l to 270 g/l. Pretreatment baths, which generally contain wetting agents, sequestering agents, and sodium hydroxide. For pretreatment, a padding Mangle machine is used. The yarn is first padded in a padding mangle with a caustic soda solution. The yarn is then passed in between the infrared heater to dry the yarn. Ten samples of three different yarn counts (12, 16 & 10) were treated with caustic soda. Other chemicals like the wetting agent and sequestering agent amount were kept 1 g/l for every sample.

2.4. Dyeing Process

The samples of yarn used for causticization are dyed with two different types of dyestuff, one is Indigo (different shades) and Sulphur (different shades).Indigo and Sulphur dyes both are naturally water-insoluble dyestuff (Saeeda, 2016). But these two dyestuffs are very suitable for denim fabric. The list of chemicals used in the research work are given in ***Table 2*** & ***Table 3***,

Table 2: List of chemicals used in Indigo dyes

Name of Chemical	Gram per litter
Free $Na_2S_2O_4$	0.6 g/l
pH	11.95-12
Wetting agent	1 g/l
Sequestering agent	1 g/l
Sample weight	1 gm
Stock Solution	1%
M:L	1:30
Temperature	80°C
Time	40 Min

The fabric sample was dyed with three different shade percentages of Indigo dyestuff. Liquid Indigo of Denisol Indigo 30 (Archroma) with light shade 1% (o.w.f), medium shade 2% (o.w.f) and deep shade 3.5% (o.w.f) was used on the different denim yarn sample. Ten samples had been dyed based on shade variation and Yarn types. All other chemicals and auxiliaries for dyeing were kept the same for every sample only variation happened in shade percentage. A padding Mangle sample dyeing machine was used for the dyeing process. The textile materials (yarn) were first padded in a padding mangle with the dye solution. After the completion of the dying process, dyed materials were dried by an infrared dryer. Then after treatment and finishing were done.For dyeing with Sulphur dyes, following condition was followed;

Table 3: List of chemicals used in Sulphur dyes

Name of Chemical	Gram per litter
pH	12.95-13
Wetting agent	4 g/l
Sequestering agent	4 g/l
Reducing Agent	11 g/l
Sample Weight	1 gm
Stock Solution	1%

Sulphur dyes are also water-insoluble dyes like Indigo dyes. It has to be made water-soluble before applying in the dyeing process (Saeeda, 2016)Generally Sulphur dyes are used in denim fabric especially for black, cherry, grey, rust, mustard, and lime, and also to get better dyeing quality. But because of its water insolubility, these dyestuffs have to be made water-soluble by reducing agents like Indigo dyes (Chi-wai Kan, 2017) .

The sample was dyed with two different shade percentages of Sulphur dyes. Liquid Sulphur dyes of Diresul Black RdT DBD (Archroma) were used for dyeing. Light shade percentage was 3% o.w.f. and deep one was 6% o.w.f. ten samples were dyed for each shade and the padding mangle was used for dyeing.

2.5. Color Strength Measurement

The color Strength of the samples was determined by using Kubelka-Monk (k/S) equation. A spectrophotometer of Data color brand version 650 was used to determine the color yield of the samples dyed with Indigo and Sulphur dyes.

2.6. Tensile Strength Measurement

Tensile strength was tested to determine the effect of caustic soda on the dyed samples. A universal strength tester was used to test the strength of the sample. For the tensile strength test, the ASTM D5034 method was followed. The ASTM D5034 standard is used to evaluate the breaking strength and elongation of textile fibers. The ASTM D5034 standard pertains to the majority of woven textile materials, such as weather-resistant barriers, although it can also be applied to fabrics made using alternative techniques. To completely comprehend the test setup, procedure, and results criteria, it is essential to peruse the full standard.

3. Results

Well-known is the influence of causticization on the enhancement of color yield. Measurements of color in the dyed yarn are examined in a variety of ways, but the KubelkaMunk function K/S, where K is an absorption coefficient and S is a scattering coefficient, has been utilized by the majority of employees to compare color yield changes due to mercerization and causticization(Khalifa, 2017). As shade depth increases, the K/S ratio rises. Instead of providing the precise amount of dye present on the dyed sample, the measurement of K/S provides the comparative color yield or dye uptake. The major goal of the current research is to determine experimentally the ideal caustic soda concentration to be employed for causticization. Besides that,the effect of caustic soda concentration on various types of counted yarns was tried to find out. To establish the result of this research after dyeing, all works are mainly divided into two parts. One is the effect of caustic soda concentration on color yield and another one is the Effect of caustic soda concentration onthe tensile strength of the samples. For each shade and each count of yarn, ten samples were tested for this research.

3.1. Effect of caustic soda concentration on color yield

Various caustic soda solutions (20 g/l to 180 g/l) were used to treat samples of commercial cotton yarn. On samples treated with various doses of caustic soda, the percent increase in K/S values showed an increase in color yield. The objective of the current experiment was to determine the

ideal caustic concentration to reap the benefits of the increased color yield. Therefore, experiments were conducted to determine how caustic soda concentration in the restricted range of 150 g/l-270 g/l affected dye uptake and yarn strength.

3.2. Color Strength for Indigo dyes Light Shade

The tests of all samples are arranged based on shade and yarn count. The results of the effect of caustic soda concentration on color yield measured as K/S are shown below in ***Figure 2***,

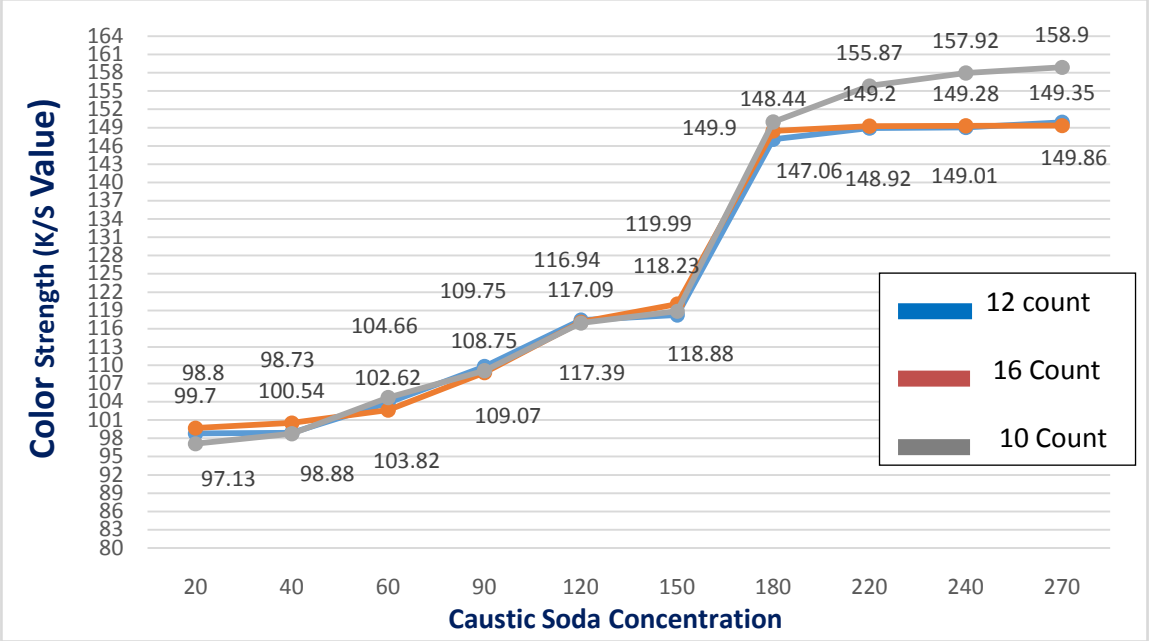

Figure 2: Graphical representation of color strength (K/S Value) of Indigo light shade

It shows that with the increase of the concentration of caustic soda value in the pretreatment, the strength of the color also increased. But from the graph show that in caustic soda concentration 20 to 60 g/l, the color strength increased very slowly, from 60 to 150 g/l, the color strength increased gradually, from 150 to 180 g/l, the color strength increased rapidly and from 180 to 270 g/l, color strength increased very slowly.

3.3. Color Strength for Indigo dyes Medium Shade

The experiment was also done by the medium-shaded indigo dyestuff. Ten sampleswere developed for each counted yarn. After the dyeing process, all samples were tested by spectrophotometer to measure the color strength to see the effect of caustic soda concentration on the fiber material. The results of the effect of caustic soda concentration on color yield measured as K/S are shown belowin ***Figure 3***,

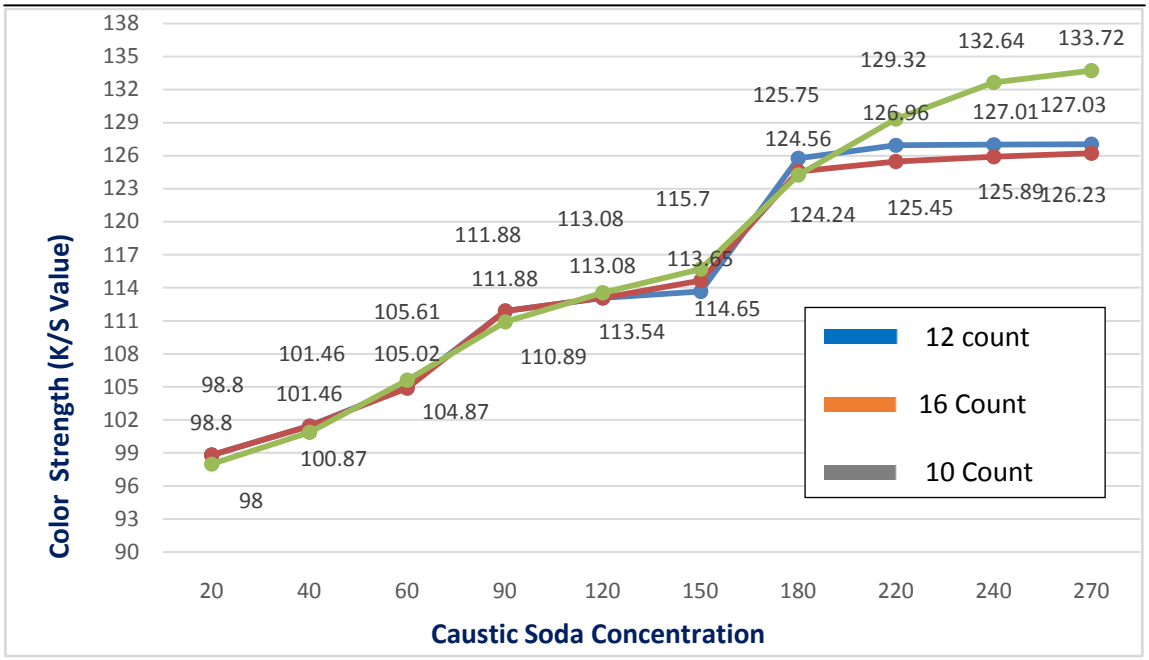

Figure 3: Graphical representation of color strength (K/S Value) of Indigo medium shade

Where it was shown that color strength is increased gradually for all samples with the increased value of the caustic soda concentration. It is observed from the graph that from caustic soda concentration 20 to 60 g/l the color strength of medium shaded sample was increased gradually but very slowly but after 60 g/l concentration the color strength increased a little fast and between 150 to 180 g/l concentration, the color strength growth is very rapid. It shows color strength growth in this portion is Between 113 to 127. After 220 to 270 g/l color strength growth is slow again.

3.4. Color Strength for Indigo dyes Dark Shade

For Indigo dark shade, ten samples for each counted yarn were experimented with for this research work. 4% shade percentage was used for indigo dark shade for this experiment. Ten samples were developed for each counted yarn. After the dyeing process, all samples were tested by spectrophotometer to measure the color strength to see the effect of caustic soda concentration on the fiber material. The results of the effect of caustic soda concentration on color yield measured as K/S are shown in ***Figure 4***. Graphical representation of color strength (K/S Value) of Indigo medium shade for yarn count 12, 16 & 10. From these experiments and test reports of color strength value from spectrophotometer, the graphical analysis has shown that for dark shaded indigo dyes the growth of the color strength is increased gradually. From the graph, it is shown that for dark shaded indigo dyes, the color strength was increased very smoothly from 20 g/l to 270 g/l. It represents an almost curve line in the graph paper. Here the color strength of the dyed yarn shows 86.78 to 120.03.

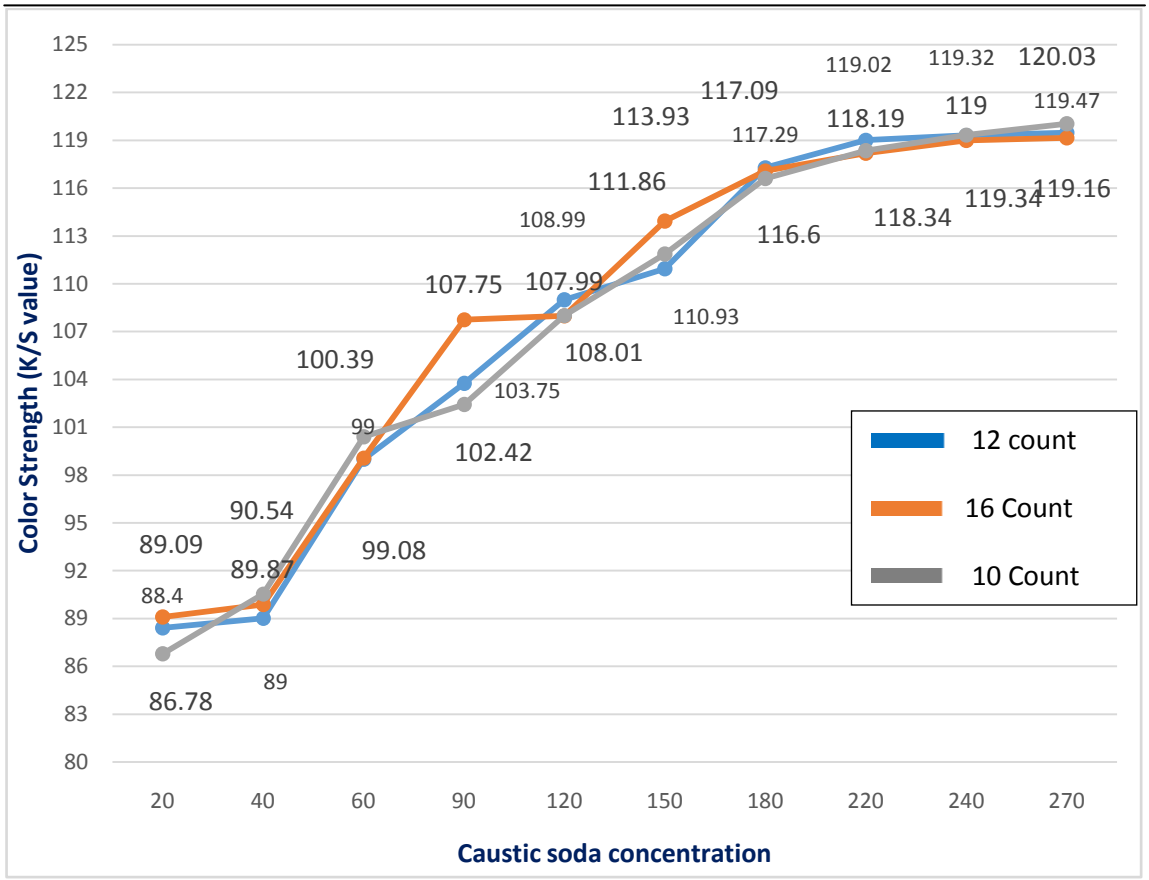

Figure 4: Graphical representation of color strength (K/S Value) of Indigo dark shade

3.5. Color Strength for Sulphur dyes light Shade

Sulphur dyes are also used in the denim fabric dyeing process. It is very popular for black color in the denim industry. Like all other experiments, ten samples were developed for the test for each counted yarn. After the dyeing process, all the samples were tested under a spectrophotometer to see the color strength. Also it was observed was the effect of caustic soda concentration on the dyed materials. The results of the effect of caustic soda concentration on color yield measured as K/S are shown below,

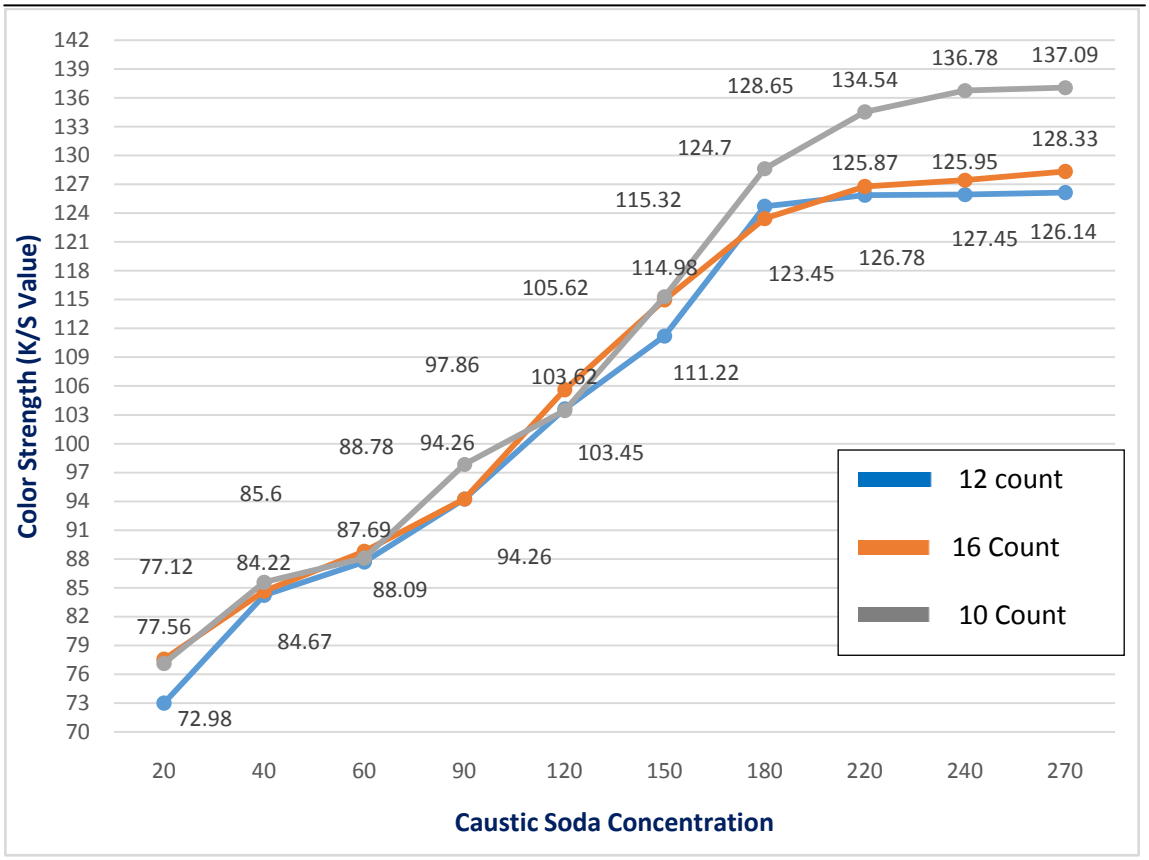

Figure 5: Graphical representation of color strength (K/S Value) of Sulphur light shade

It is shown that the color strength of Sulphur dyed samples was increased with the increased value of caustic soda concentration. From the graphical analysis, it replaced that from 20 to 60 g/l caustic soda concentration the growth of color strength is increased in a moderate fast way. But from caustic soda concentration 60 g/l to 180 g/l, the color strength growth of the dyed sample was very rapid. Here the color strength value increased with the increasing value of caustic soda. But from 180 to 270 g/l caustic soda concentration, the color strength growth increased slowly.

3.6. Color Strength for Sulphur dyes dark Shade

Sulphur dyes are also used in the denim fabric dyeing process. It is very popular for black color in the denim industry. Like all other experiments, for Sulphur dyes dark shade ten sample was developed for the test for each counted yarn. After the dyeing process, all the samples were tested under a spectrophotometer to see the color strength and also observed the effect of caustic soda concentration on the dyed materials. The results of the effect of caustic soda concentration on color yield measured as K/S are shown below in ***Figure 6***,

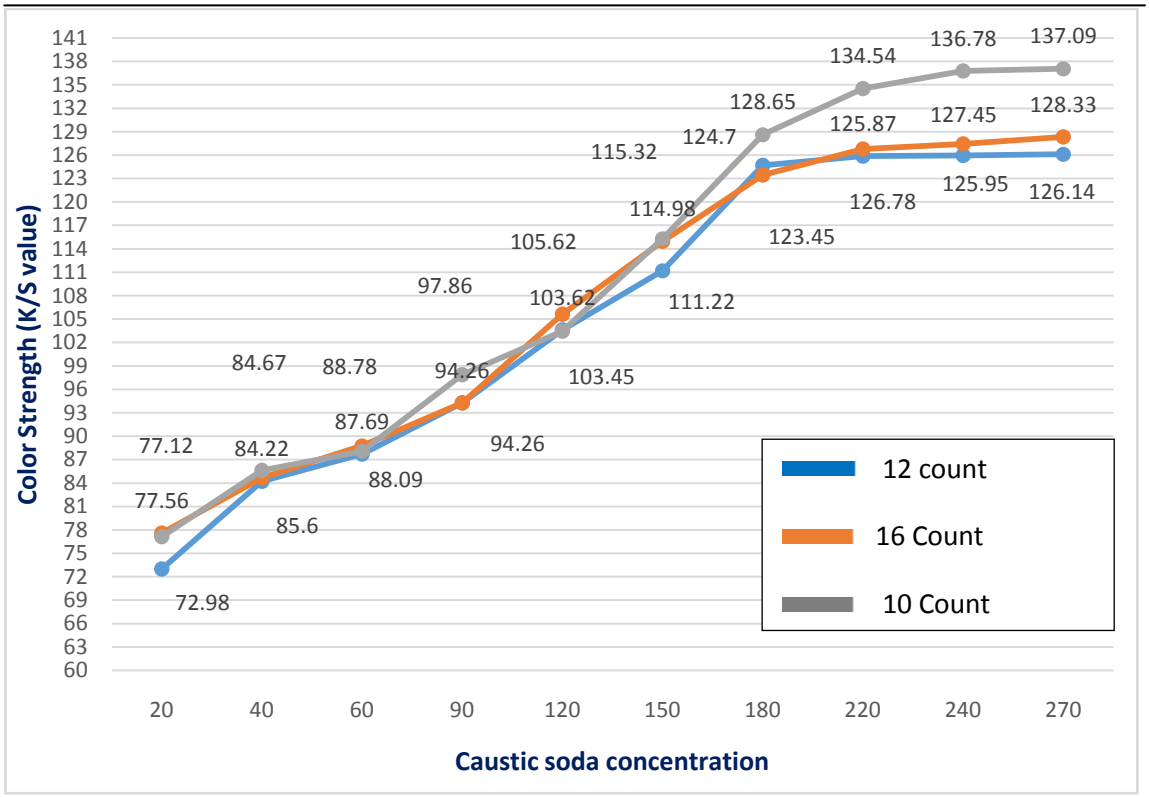

Figure 6: Graphical representation of color strength (K/S Value) of Sulphur light shade

It is shown that the color strength of Sulphur dyed samples was increased with the increased value of caustic soda concentration. From the graphical analysis, it replaced that from 20 to 60 g/l caustic soda concentration the growth of color strength is increased in a moderate fast way. But from caustic soda concentration 60 g/l to 180 g/l, the color strength growth of the dyed sample was very rapid. Here the color strength value increased with the increasing value of caustic soda. But from 180 to 270 g/l caustic soda concentration, the color strength growth increased slowly.

3.7. Effect of caustic soda concentration of Tensile Strength

The color strength of the samples was analyzed and it is shown that with the increased value of caustic soda concentration the color strength also gradually increased. But with the increased value of caustic soda, the molecular bond of cellulose may be affected. Excessive caustic soda may cause the breaking of intermolecular bonding of cellulose (Jürgen Kunze, 2005).To check the strength of the yarn for thedyed sample, a universal strength tester was used in this experiment. Ten tests were done for each shade.

3.8. Yarn Strength for Indigo Light Shade

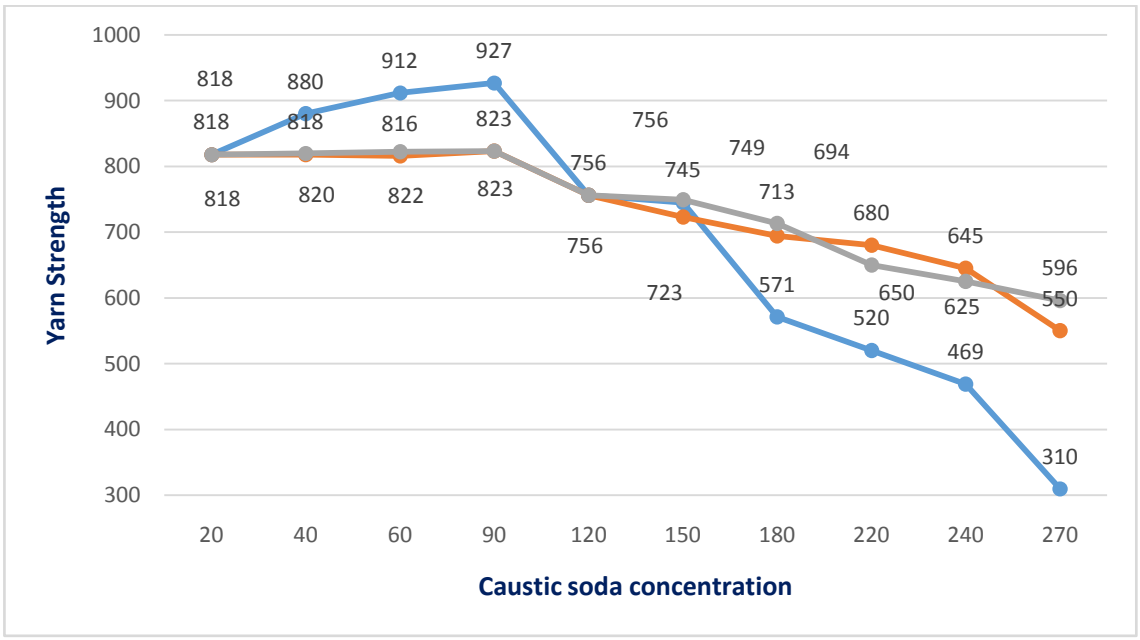

Figure 7: Graphical representation of yarn strength for Indigo light shade dyed yarn

3.9. Yarn Strength for Indigo medium Shade

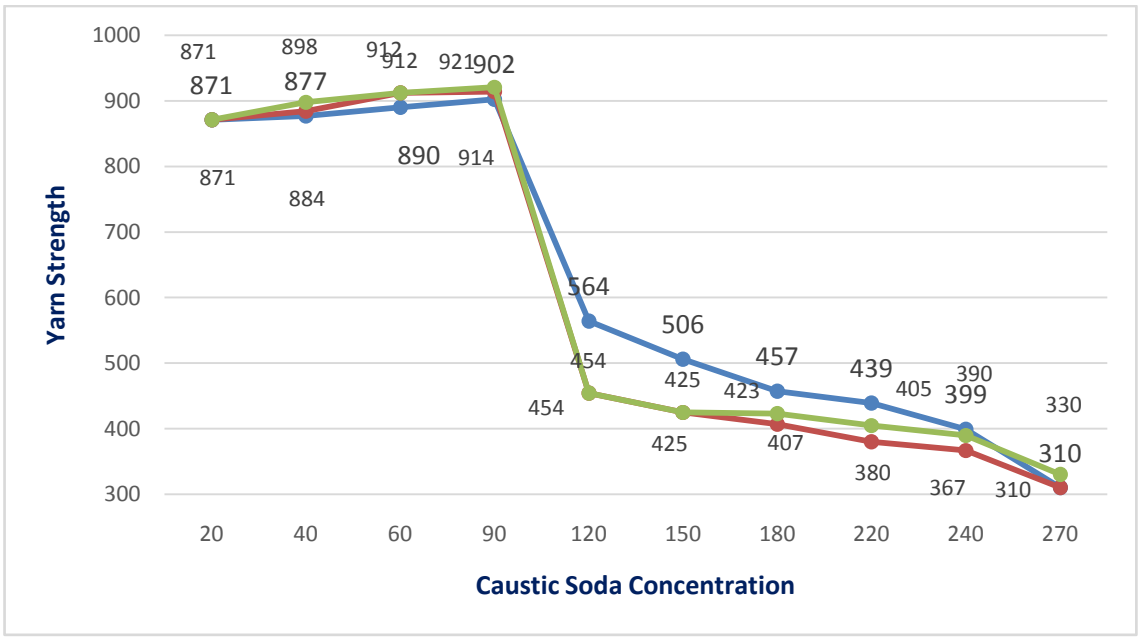

Figure 8: Graphical representation of yarn strength for Indigo medium shade dyed yarn

3.10. Yarn Strength for Indigo dark shade

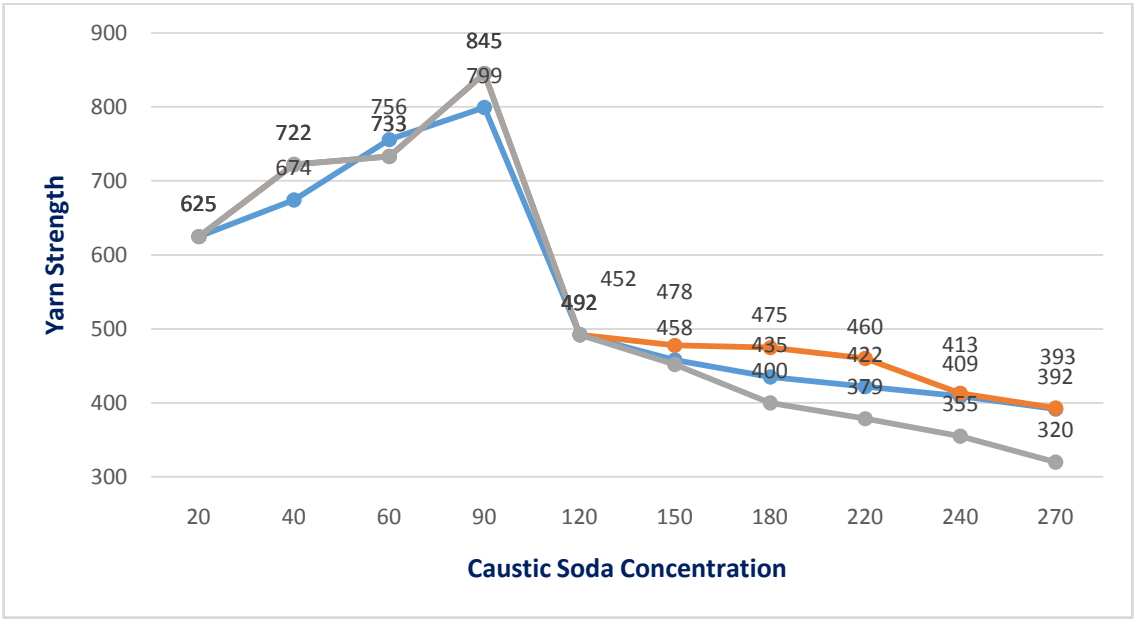

Figure 9: Graphical representation of yarn strength for Indigo dark shade dyed yarn

3.11. Yarn Strength for Sulphur dyed light Shade

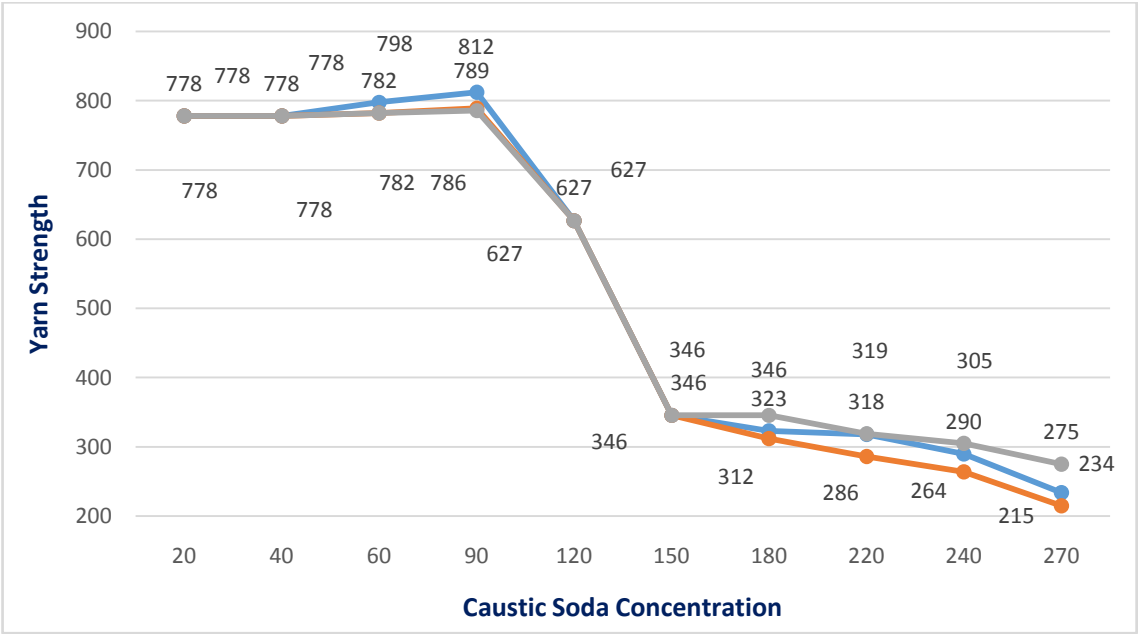

Figure 10: Graphical representation of yarn strength for Sulphur light shade dyed yarn

3.12. Yarn Strength for Sulphur dyed dark Shade

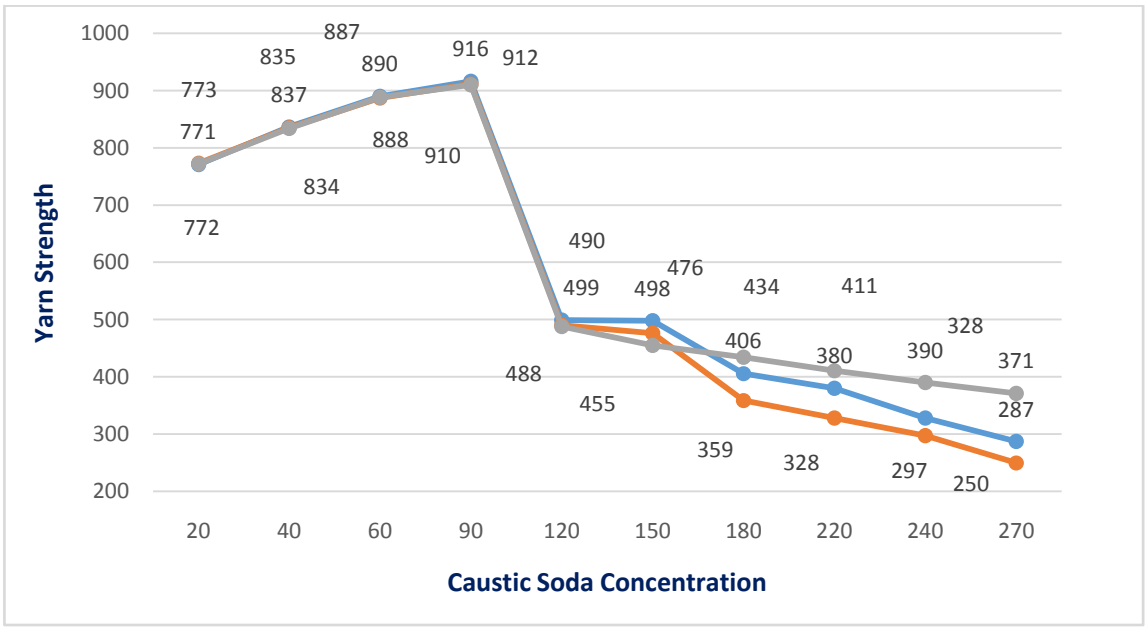

Figure 11: Graphical representation of yarn strength for Sulphur dark shade dyed yarn

After completing all the tests of tensile strength of each sample, it showed the effect of causticization on the cotton denim yarn. For Indigo dyed light shaded sample, the graph (***Figure 7***) showed that 20 g/l caustic soda treated samples tensile strength is 818, 40 g/l caustic soda treated samples tensile strength is 880, for 60 g/l tensile strength is 912 and it increased to 927 for 90 g/l caustic soda treated samples. But after 90 g/l, the tensile strength of the samples are gradually decreased like for 120 g/l it is 756, for it is 571 and for 270 it is 310. The tensile strength for other samples like Indigo dyed medium shaded sample (***Figure 8***), Indigo dyed deep shaded sample (***Figure 9***), Sulphur dyed light shaded sample (***Figure 10***) and Sulphur dyed deep shaded (***Figure 11)*** sample are also initially increased to 20 g/l to 90 g/l but after 90 g/l the strength is gradually decreased.

4. Findings and Discussion

From all the analyses of Indigo and Sulphur dyed samples, yarn strength was tested by a universal strength tester. From the result of all strength tests, it is observed that strength is gradually increased from 20 g/l to 90 g/l. From the Indigo light shade strength-caustic concentration graph, it is shown that at 20 g/l caustic concentration the strength is 818, at 40 g/l strength is 880, at 60 g/l strength is 912 and at 90 g/l it is 927. But from 90 g/l to 120 g/l, the yarn strength is gradually decreased. At 120 g/l the yarn strength shows 756, at 150 g/l it shows 745, at 180 g/lit shows 571, at 220 it shows 520, at 250 g/l it shows 469 and at 270 g/l it shows 310. In the analysis of other experiments, it is replaced that the strength increase and decrease pattern is almost the same as the indigo light-shaded dyed sample. In all samples, strength is increased between 20 g/l to 90 g/l but after 90 g/l to 270 g/l, the strength is decreased gradually. That means after an optimum rate of caustic concentration, caustic soda affects the molecular bonding of cellulose that's why the strength of the cellulose also decreased.

The outcomes in each example can be understood based on how sodium hydroxide hydrates at various concentrations and how that affects swelling. There is some proof that alkali hydroxide ions' capacity to penetrate and swell cellulose fibers depends on how hydrated they are. The sizes of the hydrated ions are too big for simple penetration into the fibers at low sodium hydroxide concentrations. The amount of water molecules accessible for the creation of hydrates declines as the concentration of caustic soda rises, and as a result, so does their size. Small hydrates can permeate into the pores and low-order areas of cellulose, as well as into the high order, or crystalline regions. Based on the aforementioned information, conclusions on how caustic soda concentrations affect color yield can be drawn. There may not be further hydrogen bond breakage of already water-swollen fiber due to high degree of hydration at low concentration caustic soda as evidenced by yield being minimally lowered up to 20 – 60 g/l caustic soda concentration influence on color. The sluggish increase in color output indicates that the fiber swelling tends to grow between 90 and 150 g/l of caustic soda concentration. The increase in color yield between 180 and 270 g/l caustic soda concentration can be attributed to the rapid swelling rate and potential hydrogen bond breakage in the intercrystalline areas.

For the yarn strength, the results indicated that between 20-60 g/l caustic soda concentration increases in yarn strength were low, a rapid rise in strength was observed between 90 g/l caustic soda concentrations. Beyond 120-270 g/l caustic soda concentration the strength is decreased. The above results indicated that the causticization at 270 g/l concentration of caustic soda resulted in an increase in color yield in the case of Indigo Dark Shade, Indigo Light Shade, Indigo Medium Shade, Sulphur Light Shade, Sulphur Dark Shade and 90 g/l concentration of caustic soda resulted in an increase in Yarn Strength in case of Indigo Dark Shade, Indigo Light Shade, Indigo Medium Shade, Sulphur Light Shade.

5. Conclusion

In this research work, the appropriate concentration of Caustic soda has been found out for the Pretreatment process. It is very important to know the ideal concentration of Caustic during scouring in the dyeing industry. Less concentration of caustic soda gives a poor result and more concentration is a big threat for fiber damage which reduces the strength of the textile product. The process is carried out in the exhaustion method by using high concentration caustic soda in scouring and bleaching bath.The K/S value of all dyed samples with Indigo and Sulphur dyes in different concentrations is also measured. Dye up-take for indigo and Sulphur dyes is maximum at 270 g/l concentration of caustic soda but strength is maximum at 90 g/l concentration of caustic soda. From this work, it is found out that the dyeing rate is increased with the increased value of caustic soda. The color yield increased from the 20 g/l concentrated caustic soda application on the textile materials, and it increased the value gradually to the last value of the caustic soda concentration in this research is 270 g/l. But on the other hand yarn strength is not proportional to caustic soda concentration. The cellulosic yarn strength increased at a definite point of caustic soda concentration. But after a certain point, the strength of the cellulose yarn is gradually decreased. So from the research work, it is found out that for light to medium types of shade the optimum rate of caustic soda concentration should be 60 g/l, and for dark shade, the caustic soda concentration might be 90 g/l. This concentration is good for both strength and color yield.

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