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# Analysis of Caustic Soda of Different Manufacturers in Pakistan for Mercerization of Cotton Textiles

BILAL ZAHID\*, SAIRA FAISAL\*, AND SHERAZ HUSSAIN SIDDIQUE\*

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## ABSTRACT

Pakistan has sufficient production capacity of caustic soda to cater the needs of the local industry. Presently, Pakistan has four major plants with production capacity around 435,000 mega ton per year of caustic soda of various grades. Textile industry of Pakistan is the major consumer of produced high grade caustic soda; as the presence of any impurities especially dissolved salts and metals is unfavorable for wet processing of cotton. This study investigates the performance of three different local brands of caustic soda intended for use in textile wet processing of cotton fabric specifically for mercerization process. The brands were selected based on their purity grades. The selected caustic soda samples were chemically analyzed for the presence of impurities. Twenty seven cotton fabric samples (nine samples of each brand) were prepared by mercerization in slack state. The cotton fabric samples were tested for changes in surface morphology, tensile strength (warp and weft), tear strength, air permeability and K/S (Colour Strength). The results were analyzed using one-way ANOVA (Analysis of Variance) to ascertain the statistical equivalence within and between the tested brands. The results showed no significant differences across the tested brands at a 95% confidence level except for air permeability and K/S in blue shade.

**Key Words:** Caustic Soda, Mercerization, One-Way Analysis of Variance, Tensile Strength, Colour Strength, Tear Strength, Air Permeability, Surface Morphology.

## 1. INTRODUCTION

Caustic Soda is one of the basic building blocks of chemical industry and is extensively used in various industrial applications [1]. Pakistan produces around 435,000 mega ton of caustic soda per year with Sitara Chemicals being largest contributor (42%), Ittehad Chemicals (33%), Engro Polymers (23%) and Nimir Industrial Chemicals (2%) [2]. The textile industry of Pakistan consumes approximately 43% caustic soda with compound annual growth rate of 7% [3].

Caustic soda is the strongest alkali and it is commonly produced in various grades of purity. Much of the total production of high grade caustic soda utilized by rayon industry and also textile industry for mercerization of cotton, bleaching and dyeing processes [4]. It is also known that commercial aqueous solution of caustic soda is contaminated with 10-100 ppm of iron unless specially treated. For caustic soda to be consumed in textile industry, the presence of any impurities especially

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dissolved salts and metals is detrimental; and sodium carbonate and iron are particularly undesirable as they cause decrease in K/S [5-6]. Therefore, textile industry published a draft guideline on the limits of impurities, which recommends maximum 150 ppm of sodium chloride, 800 ppm of sodium carbonate and 5 ppm of iron [7].

The textile industry in Pakistan uses a substantial quantity i.e. every 1kg of cotton requires approximately 300g of caustic soda in the mercerization of cotton fabrics [8,9], and it is perhaps the most important commercial process used for modifying the properties of cotton [10]. In particular, this treatment enhances dye substantivity, chemical reactivity, dimensional stability, tensile strength, moisture uptake and overall uniformity of the substrate [11-13].

The aims of this study were (i) to evaluate and compare three different brands of caustic soda available in the local market of Pakistan in terms of purity grades (ii) to evaluate statistical equivalence of three different brands of caustic soda in terms of tensile strength (warp and weft), tear strength, air permeability and K/S of treated cotton. Thus, any significant difference in physical and dye ability of treated samples may be attributable to the impurity level especially sodium carbonate and iron in different brands of caustic soda used.

## **2. MATERIALS AND METHOD**

### **2.1 Materials**

Three brands of caustic soda were collected from the local markets of Multan, Faisalabad, and Karachi. The first test brand (Caustic-A) was the leading brand of local origin of high purity grade, while the other two test brands (Caustic-B and Caustic-C) were selected on the basis that they were of lower purity grades and comparatively economical in price.

Drimarene Yellow CL-3G and Drimarene Blue CL-BR, Everzol Red CD, Unimercol AWA, Sodium Carbonate, Sodium Chloride and Acetic Acid were purchased from local markets. The fabric used was commercially scoured and bleached 100% cotton fabric (plain weave -140 g/m<sup>2</sup>).

### **2.2 Methods**

The chemical analysis of collected samples of caustic soda was conducted in accordance with ASTM E291-09 [14].

In this research work, twenty seven samples of cotton fabric (nine samples with each test brand) were prepared by mercerizing in the slack state with 20% caustic soda containing 6g/l of unimercol AWA as a wetting agent. After cold wash, neutralizing in diluted acetic acid, and rinsing again, the fabric samples were air dried.

The treated samples were conditioned for 48 hours in atmospheric conditions of  $20 \pm 2^{\circ}\text{C}$  temperature and  $65 \pm 2\%$  relative humidity.

Surface morphology of fabric samples was analyzed by an optical microscope (Nikon - LEICA DM500) along with the digital camera. The images were taken at 640x magnification.

The tensile strength of treated samples in Newton (N) was determined both in warp and weft direction according to the method specified by Standard BS EN ISO 13934-1 [15]. The measurement was performed at a gauge length of 200 mm and an extension speed of 100 mm/min.

The tear strength of the samples in Newton (N) was measured according to the method specified by Standard ASTM D-1424, using Elmandorf Tear Tester [14].

The air permeability of the samples in mL/(cm<sup>2</sup>.s) was measured according to the method specified by Standard BS EN ISO 9237 [16], using Atlas M012S air permeability

tester. The measurements were performed at a constant pressure drop of 100 Pa (5cm<sup>2</sup> test area).

Treated samples were dyed with reactive dyes in red, yellow and blue shades (1% owf) using dyeing method mentioned earlier [17], before being evaluated for K/S using Kubelka-Munk equation [18].

### 2.3 Statistical Analysis

One-way ANOVA was applied on analytical data obtained to determine if variance across the brands were homogenous. The parameters considered were tensile strength (warp and weft directions), tear strength, air permeability and K/S of treated samples. The Tukey's test was used for post-hoc comparisons to determine which brand differs. All statistical tests were performed at the 95% confidence level ( $p < 0.05$ ). Data was analyzed using the Minitab version 16 software. Bar charts are used to show means of experiments.

## 3. RESULTS AND DISCUSSION

### 3.1 Chemical Analysis of Test Brands of Caustic Soda

The chemical analysis results were compared with the guideline impurity levels used in Textile Industry [7] and shown in Fig. 1. From Fig. 1(a-b), it is evident that Caustic-B and Caustic-C had both sodium carbonate and iron content exceeding the recommended levels of 800 and 5 ppm respectively. However, all the tested brands had sodium chloride content below the recommended level of 150 ppm (Fig. 1 (c)).

### 3.2 Microscopic Analysis of Untreated and Treated Samples

Fig. 2 shows the photomicrographs of the untreated and treated cotton fibre surface. From Fig. 2(a-d), it is evident that the surfaces of treated cotton fibers become much

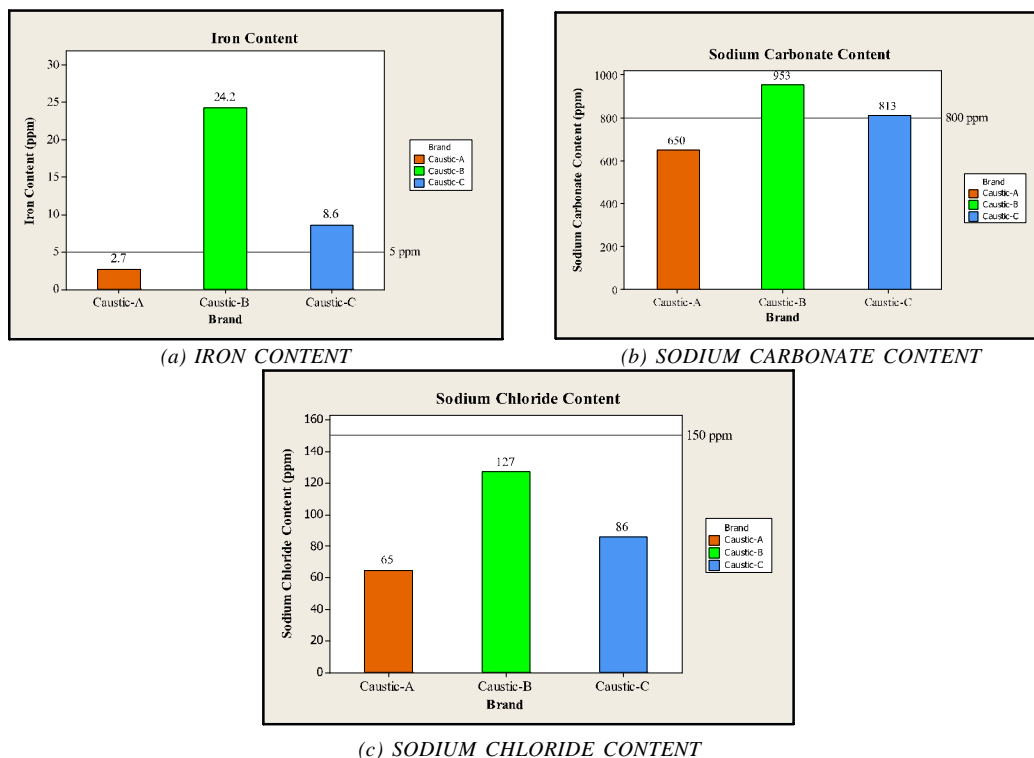


FIG. 1. BAR CHARTS SHOWING IMPURITIES LEVELS IN TEST BRANDS CAUSTIC-A, CAUSTIC-B AND CAUSTIC-C. REFERENCE LINE SHOWS THE MAXIMUM ALLOWABLE IMPURITY LEVEL

more smooth, de-convoluted and swollen (Fig. 2(b-c)) as compared to untreated sample (Fig. 2(a)). The untwisting, swelling and smoothness of treated samples observed by optical microscopy is consistent with an earlier findings [19].

### 3.3 Effect of Impurity Level in Test Brands on Tensile Strength (Warp and Weft) and Tear Strength of Cotton Fabric

From **Fig. 3(a-c)**, it is evident that mercerization in slack state results in significant increase of the tensile and tear strength of cotton fabric. This increase in strength of cotton fibre may be attributed to increased uniformity of strength along the fibre length [10,13,19]. Mercerization also increases the cohesion between individual cotton fibres and this closer embedding of the fibres in the yarn

not only increases the strength but makes it more uniform in strength [20].

It is also observed that tensile strength in weft direction (Fig. 3(b)) is lesser than that of in warp direction (Fig. 3(a)) in untreated as well as treated samples; this is attributed to the significant lesser number of threads in weft direction than warp threads.

To determine whether the impurity levels in test brands has a significant effect on tensile and tear properties of treated samples, one-way ANOVA was carried out with the tensile and tear strength data. The results shown in Table 1 revealed that the test brands appeared to be indistinguishable with respect to tensile strength in both directions as well as tear strength ( $p>0.05$ ); this suggests that the effect of impurity levels in test brands are similar.

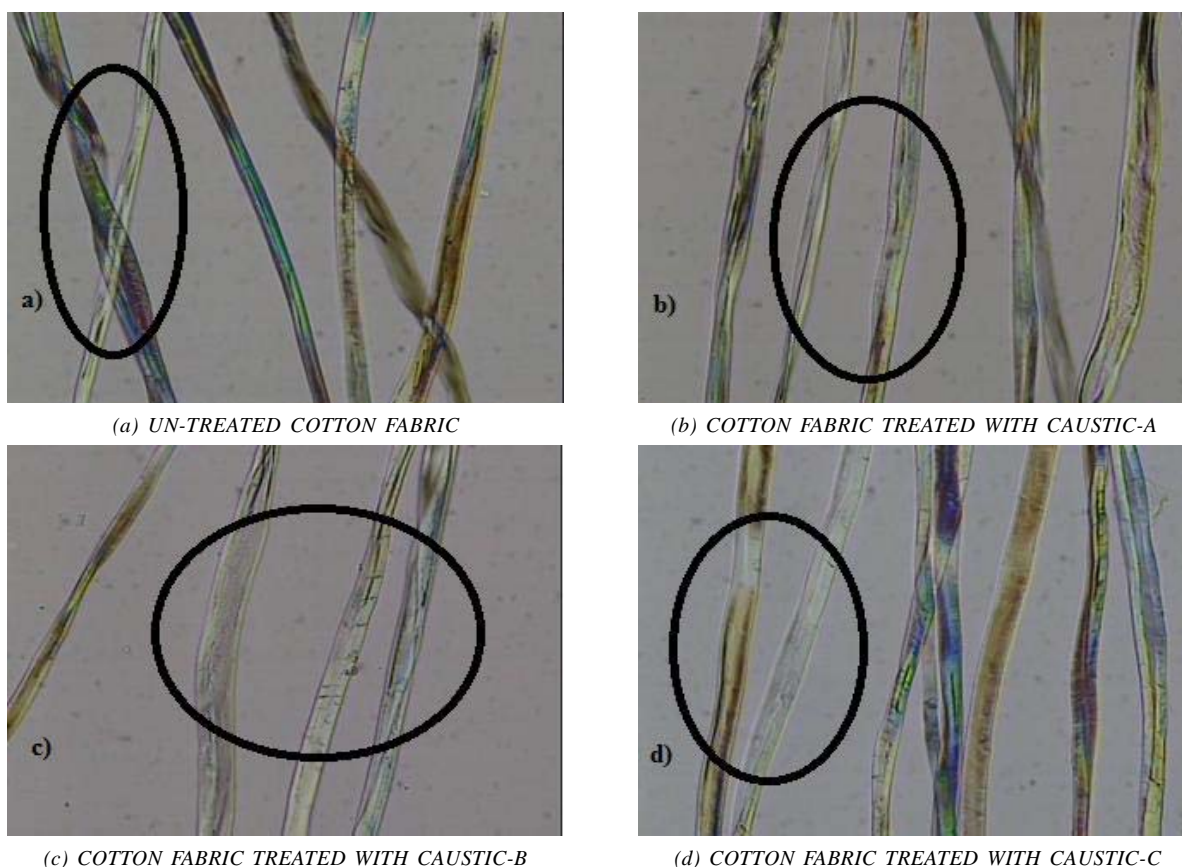


FIG. 2. SURFACE MORPHOLOGY OF UNTREATED AND TREATED COTTON FABRIC

### 3.4 Effect of Impurity Level in Test Brands on Air Permeability of Cotton Fabric

From Fig. 4, it is observed that the air permeability of the treated samples is decreased as compared to the untreated sample. This decrease in air permeability after mercerization in slack state could be attributed to the swelling of fibres after treatment thereby reducing the air spaces available between the fibres and thus the yarns in the fabric.

One-way ANOVA was used to determine whether there was a significant difference in air permeability of fabric samples treated with different test brands (Table 1); and the result shows that there is statistically significant difference between the test brands ( $P < 0.05$ ). The post hoc comparison (Table 2) revealed differences between fabric samples treated with Caustic-B and fabric samples

treated with Caustic-A or Caustic-C. However, there was no significant difference between Caustic-A and Caustic-C treated fabrics samples with regard to air permeability. It also revealed that when cotton fabric samples were treated with Caustic-A or Caustic-C, they attained highest degree of compactness of fabric (low air permeability).

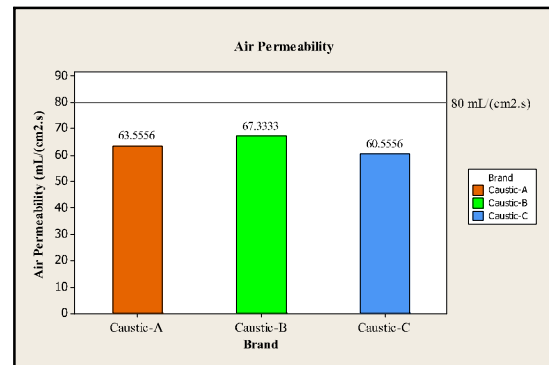


FIG. 4. BAR CHART SHOWING AIR PERMEABILITY. REFERENCE LINE SHOWS AIR PERMEABILITY OF UNTREATED SAMPLE

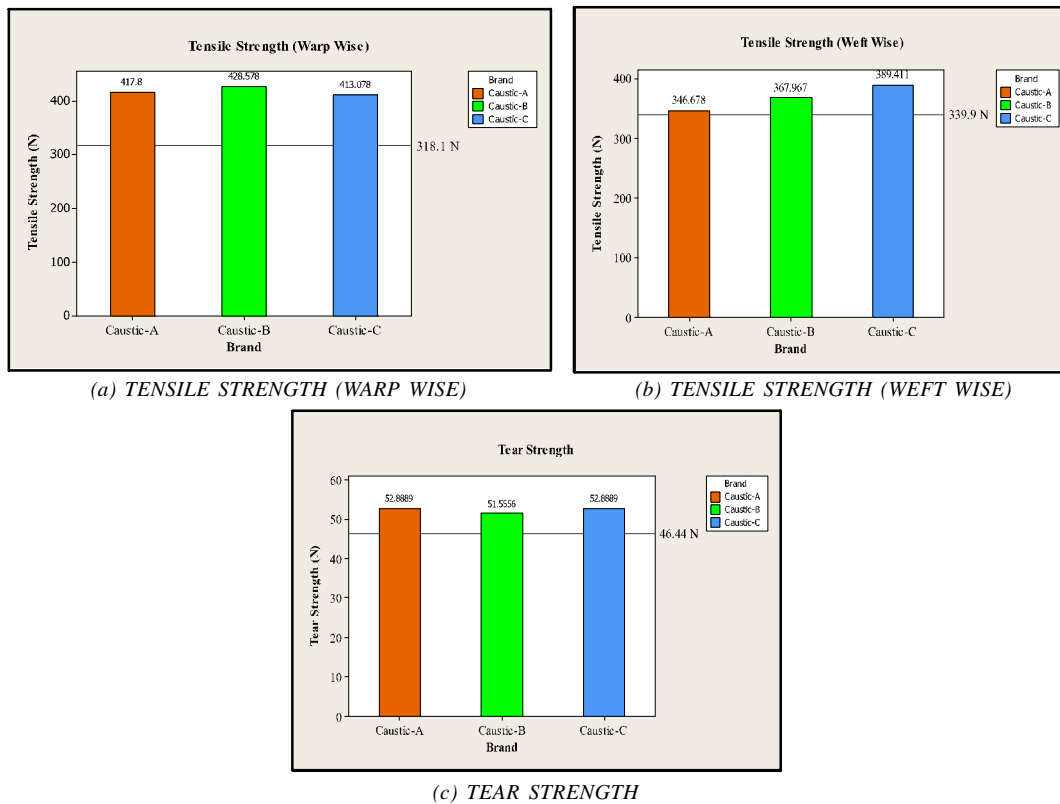


FIG. 3. BAR CHARTS SHOWING TENSILE STRENGTH (WARP AND WEFT) AND TEAR STRENGTH. REFERENCE LINES SHOWS RESPECTIVE VALUES OF UNTREATED SAMPLE

However, when fabric is treated with Caustic-B the compactness is not very pronounced, which could be

attributable to presence of comparatively high levels of impurities in this caustic soda.

**TABLE 1. ONE-WAY ANOVA – EFFECT OF IMPURITY LEVEL IN TEST BRANDS ON TENSILE STRENGTH (WARP), TENSILE STRENGTH (WEFT), TEAR STRENGTH, AND AIR PERMEABILITY OF TREATED SAMPLES**

Source		Degree of Freedom	Sum of Squares	Mean of Squares	F-Statistic	P-Value
Tensile Strength (Warp Wise)	Between Brands	2	1136	568	0.34	0.716
	Within Brands	24	40261	1678	-	-
	Total	26	41397	-	-	-
Tensile Strength (Weft Wise)	Between Brands	2	6269	3134	2.87	0.076
	Within Brands	24	26221	1093	-	-
	Total	26	32490	-	-	-
Tear Strength	Between Brands	2	10.67	5.33	0.85	0.439
	Within Brands	24	150.00	6.25	-	-
	Total	26	160.67	-	-	-
Air Permeability	Between Brands	2	207.63	103.81	12.31	0.000*
	Within Brands	24	202.44	8.44	-	-
	Total	26	410.07	-	-	-
*Statistically Significant (p < 0.05)						

**TABLE 2. GROUPING INFORMATION USING POST HOC TUKEY'S TEST**

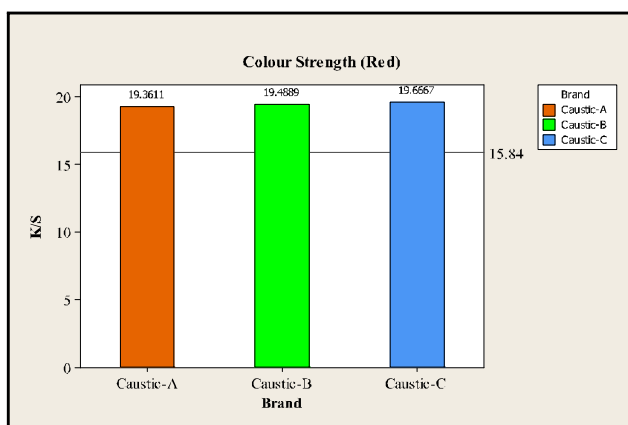
Source	Brands	Newton	Mean	Grouping	
Air Permeability	Caustic-B	9	67.333	X	
	Caustic-A	9	63.556		Y
	Caustic-C	9	60.556		Y
Means that do Not Share a Letter are Significantly Different					



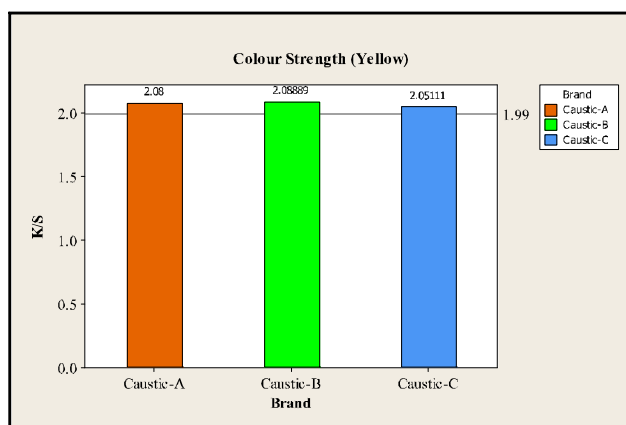
### 3.5 Effect of Impurity Level in Test Brands on Colour Strength of Cotton Fabric

It is evident from Fig. 5(a-c) that treated cotton samples illustrate increased K/S. This increased in K/S of treated cotton samples may be attributed to the swelling of fibre from bean-shaped to more circular configuration which affects the internal scattering of light [12,21]. It is also well known that mercerization increase in the proportion of amorphous region and availability of hydroxyl groups which directly relates to the increase dye absorption of mercerized cotton [22].

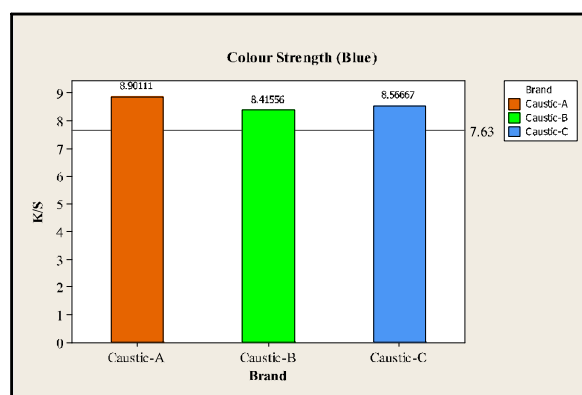
One-way ANOVA showed no significant difference in K/S among the three test brands ( $p > 0.05$ ) in case of red and yellow shade. However, it shows significant difference ( $p < 0.05$ ) in blue shade (Table 3). The post hoc comparison revealed significant differences in K/S between fabric samples treated with Caustic-A and fabric samples treated with Caustic-B (Table 4). Also K/S of samples treated with Caustic-A and Caustic-C are not significantly different from each other, but are significantly higher than the Caustic-B, this can be attributed to higher impurity levels in Caustic-B.



(a) RED



(b) YELLOW



(c) BLUE

FIG. 5. BAR CHARTS SHOWING COLOUR STRENGTH. REFERENCE LINES SHOWS COLOUR STRENGTH VALUES OF UNTREATED SAMPLE



**TABLE 3. ONE-WAY ANOVA – EFFECT OF IMPURITY LEVEL IN TEST BRANDS ON COLOUR STRENGTH OF TREATED SAMPLES**

Source		Degree of Freedom	Sum of Squares	Mean of Squares	F-Statistic	P-Value
Red	Between Brands	2	0.424	0.212	0.74	0.486
	Within Brands	24	6.850	0.285	-	-
	Total	26	7.273	-	-	-
Blue	Between Brands	2	1.111	0.556	5.34	0.012*
	Within Brands	24	2.497	0.104	-	-
	Total	26	3.608	-	-	-
Yellow	Between Brands	2	0.00347	0.00173	0.33	0.723
	Within Brands	24	0.12680	0.00528	-	-
	Total	26	0.13027	-	-	-
*Statistically Significant ( $p < 0.05$ )						

**TABLE 4. GROUPING INFORMATION USING POST HOC TUKEY'S TEST**

Source	Brands	Newton	Mean	Grouping	
Colour Strength- Blue	Caustic-A	9	8.9011	X	
	Caustic-C	9	8.5667	X	Y
	Caustic-B	9	8.4156		Y
Means that do Not Share a Letter are Significantly Different					

#### 4. CONCLUSIONS

Present study provides important findings regarding the quality i.e. impurity levels of different brands of caustic soda produced in Pakistan. Moreover, the selected brands were also analyzed for their suitability in textile industry of Pakistan specifically for mercerization process. The important findings are:

- (i) The chemicals analysis of three test brands showed that only Caustic-A, which was the leading brand of local origin, has impurity level within the recommended guideline. However, Caustic-B and Caustic-C has impurity level higher than the recommended guideline. Among three brands Caustic-B has highest levels of sodium carbonate and iron.

- (ii) Experimental comparison made on the properties of cotton fabric treated with Caustic-A, Caustic-B and Caustic-C revealed that the tensile (both directions) and tear strength of the fabric samples increased for all cases, whereas, the air permeability of the fabric had reduced. It also revealed that the K/S of the treated samples was also increased in all cases.
- (iii) Statistical analysis using one-way ANOVA revealed that all of the three tested brands are equivalent i.e. impurity level in caustic soda has no effect on tensile strength, tear strength and K/S (Red and Yellow Shade) of treated samples.
- (iv) One-way ANOVA followed post hoc Tukey's test revealed that the three tested brands are not equivalent i.e. impurity level in caustic soda has effect on air permeability of treated samples.
- (v) Statistical analysis revealed K/S of samples treated with Caustic-A and Caustic-C dyed in blue shade are not significantly different from each other, however, they are significantly different than the Caustic-B, and this can be attributed to highest level of sodium carbonate and iron in Caustic-B among the three brands.

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