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# **Effect of alkali treatment of lower concentrations on the structure and tensile properties of Pakistan's coarse cotton fibre**

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## **Abstract**

Cotton fibres of high Micronaire values usually have adverse spinning performance, either reduction of the fibres' fineness or increase of their tensile strength would improve the fibre spinnability. In this piece of research, the effects of alkali treatment at lower concentrations (0.75M–2.25M) and higher temperatures (70°C–100°C) on the changes of cotton fibre cross-section and tensile strength have been investigated through the observation in scanning electron microscopy (SEM) and single fibre tensile strength testing. It was found that the both the improved roundness of the fibre cross section and the increase of tensile strength of alkali treated cotton fibres have been observed at relatively lower concentration (0.75M) and relatively lower temperature (70°C), which was believed due to possible cellulose dissolution/transformations. It was thus concluded that the alkali treatment of cotton fibres at lower concentrations (0.75M) and 70°C for a shorter period of time (45 mins) could lead to improving tensile strength and roundness of fibre cross-section thereby improving micronaire.

**Keywords:** Micronaire; spinning potential; alkali treatment; Cellulose dissolution; Single Fibre Tensile Strength

## **Introduction**

Cotton is a major natural fibre which exhibits pronounced variation in its properties

(Majumdar, Majumdar, & Sarkar, 2004). The production of cotton fibres of high Micronaire value, which is an indication of both fineness and maturity of cotton fibres, and hence greatly influenced by the geometry of fibre (Montalvo Jr, 2005), has become a quality issue for cotton all around the world. The US Upland cotton crop average Micronaire for the years 2009, 2010, 2011 and at end of September 2012 are 4.18, 4.52, 4.53 and 4.76 respectively showing that the Micronaire is on the rise (Cotton Incorporated, 2012). In Australia, the percentage of cotton in the G5B Micronaire range (3.8–4.5) has fallen since 2000 to 2007 from 75% to 45% of the crop with the percentage of cotton in the coarser G5C range (4.5–4.9) soared from 10% to over 40% in the same period (Van der, Marinus, Gordon, & Long, 2008). A quality survey of Pakistani cotton crop conducted by Pakistan Central Cotton Committee for the year 2009–10 concluded that cotton grown in the Sind and Punjab provinces of Pakistan have average Micronaire values of 4.7 and 5.0 respectively. Percentages of the crop in the Micronaire range of 4.5–4.9 and 5.0–5.4 were 58.1% and 24.3%, and 37.3% and 57.4% for Sindh and Punjab respectively (Ismaili, 2010).

The spinning limit of cotton is dependent on fibre properties and spinning method. To produce high-quality ring-spun textiles, fibres must be fine and have sufficient strength to endure processing (Faulkner, Hequet, Wanjura, & Boman, 2012). Fineness is therefore considered as the dominant factor in determining the limiting count which could be spun out of the cotton fibres. Also, the uniformity of a yarn is largely determined by the average number of fibres in the yarn cross-section, therefore it follows that the finer the fibres, the more uniform is the yarn for a given yarn count; and the finer count that can be spun for a given number of fibres in the yarn cross-section (Morton & Hearle, 2008; Rieter, 2012; Zhu & Ethridge, 1998).

In order to spin cotton fibres into reasonable quality ring-spun yarn, in general the least number of fibres are required in the yarn cross section is 90 fibres for carded yarns and 70 for combed yarns (Steadman, 1997). For open-end yarns and air-jet spun yarns this requirement is at least 110 fibres (Steadman, 1997) and 75 fibres (Bange et al., 2009) respectively. The number of fibres (n) in the yarn cross section can be estimated from the relation:

$$n = \frac{\text{tex}_{\text{yarn}}}{\text{tex}_{\text{fibre}}}$$

Where  $\text{tex}_{\text{yarn}}$  and  $\text{tex}_{\text{fibre}}$  are the linear densities of yarn and fibre respectively (Klein, 1998).

Various indexes have been devised to estimate the overall fibre quality in terms of its yarn spinnability. Fibre Quality Index (FQI) was established using High volume instrument (HVI) results by South Indian Textile Research Association in the following equation (Majumdar, Majumdar, & Sarkar, 2005):

$$\text{FQI}_{\text{HVI}} = \frac{\text{UHML} \times \text{UI} \times \text{FS}}{\text{FF}}$$

Where  $\text{FQI}_{\text{HVI}}$  is the Fibre Quality Index (Dimensionless) based on HVI value, UHML is the Upper Half Mean Length (Inches), UI is the Uniformity Index (Dimensionless), FS is the tensile strength of fibre bundle (g/tex) and FF is the fibre fineness – Micronaire (Dimensionless)

Spinning Consistency Index (SCI) for predicting the spinnability of the US cotton fibres using multiple regression analysis was proposed by Zellweger Uster. It is based on the five-year crop average of US Upland and Pima Cotton and is calculated as follows (Majumdar, et al., 2004):

$$SCI = -414.67 + 2.9FS - 9.32FF + 49.17UHML + 4.74UI + 0.65Rd + 0.36(+b)$$

Where Rd is the reflectance degree and +b is the yellowness of cotton fibre.

Both the above mentioned indices are directly proportional to the spinning potential of the fibre (Majumdar, et al., 2005). Keeping all the other properties constant, both the indices are greater for finer fibres. Therefore, coarser fibres could only be spun into coarser yarns and are not capable of producing fine yarns of sufficient quality (Hake, Bragg, Mauney, & Metzger, 1990) .

It is therefore become necessary to investigate into the possibility of utilising coarse cotton fibre to produce yarn with better productivity. Various strategies for achieving smaller Micronaire value and increasing the fibre tensile strength to improve potential fibre spinnability have been investigated in previous studies (Abbott & Robinson, 1977; Aboul-Fadl, Zeronian, Kamal, Kim, & Ellison, 1985; Karahan, Özdoğan, Demir, Aydin, & Seventekin, 2009; Karande, Bharimalla, Hadge, Mhaske, & Vigneshwaran, 2011; Lawson & Hertel, 1974; Malek & Holme, 2003; Pionteck, Berger, Morgenstern, & Fengel, 1996; Raes et al., 1971; Thibodeaux & Copeland, 1975; Thorsen, 1971; Wakelyn et al., 2006). However, none of the existing research has reported the effect of lower concentrations, that is, less than 5M, of alkali treatment on the changes of structure and tensile properties of coarse cotton fibres. In this paper, we investigate the effect of alkali treatment at lower concentrations and relatively higher temperatures on the roundness of cross-section geometry and tensile strength of Pakistan`s coarse cotton fibres.

## **Experimental**

## **Material**

Raw cotton fibres used in this research is MNH-93 which is grown in the Punjab province of Pakistan; their physical properties were determined using Uster HVI Spectrum as follows, Micronaire value 5.28, maturity 0.93, length 1.091 inches, length uniformity 82.9%, bundle Strength 31.1 cN/tex. The sample was provided by Amna Industries Private Limited, Karachi, Pakistan and taken following standard procedure of sampling.

The chemicals used in the experiments are as follows: Sodium Carbonate Reagent Plus®  $\geq 99.5\%$  (Sigma-Aldrich, UK), Sodium Hydroxide ACS Reagent  $\geq 97.0\%$  (Sigma-Aldrich, UK), Acetic Acid ACS Reagent  $\geq 99.7\%$  (Sigma-Aldrich, UK) and Sandozin NIN (Clariant, UK) used as received.

## **Scouring of Raw Cotton**

Approximate more than 100g of raw cotton was carded and blended manually and gently using hand card gently. 100g of the resultant hand-carded cotton was taken was spread in a thin layer and small pinches of fibres were taken from 32 different locations to make up a raw cotton sample of 10g. From the remaining 90g of cotton fibres, 60g of them were taken using the same pinching technique for scouring at 60°C for 30 minutes at a liquor ratio of 1:100 in a solution containing 0.2% weight by volume of Sandozin NIN and 0.2M Na<sub>2</sub>CO<sub>3</sub> in a Roaches Roto-Dryer.

The fibres were dried in ambient room for 24 hours and then conditioned for 48 hours under standard laboratory temperature of 20±2 °C and 65±2% relative humidity according to BS EN ISO 20139 (British Standard Institute, 2011). The scoured cotton fibres were carded and blended gently again using hand card.

## Alkali Treatment

In this research work, each treatment was performed in triplicate ( $n = 3$ ). 5g of the scoured cotton fibres was used as control sample, and nine samples (each is of 4.5g) were made utilizing the above pinching technique and were subject to alkali treatment in the Roaches Roto-Dryer with sodium hydroxide for 45 minutes at the liquor ratio of 1:100. Each of the cotton samples was treated in sodium hydroxide solutions of three different concentrations (0.75M, 1.50M and 2.25M) at three different temperatures (70°C, 85°C and 100°C), as shown in Table 1.

After alkali treatment, each sample was washed under warm water flow (temperature between 48 - 52°C) for 10 minutes, then was neutralized using 0.2M acetic acid solution at the liquor ratio of 1:100 for 10 minutes at the same temperature at which the treatment with sodium hydroxide was earlier performed.

After the neutralization, samples were washed using warm water flow (temperature between 48-52°C) for 10 minutes and then using normal tap water flow (temperature between 18-22°C) for 10 minutes. The samples were dried in ambient air for 24 hours and then conditioned for 48 hours under standard laboratory temperature of  $20 \pm 2^\circ\text{C}$  and  $65 \pm 2\%$  relative humidity according to BS EN ISO 20139 (British Standard Institute, 2011). Each sample was then manually carded and blended gently using hand card.

Table 1. Sodium hydroxide treatment scheme (time duration: 45 minutes)

Sample ID	Sodium Hydroxide Concentration (M)	Treatment and Neutralization Temperature (°C)
R	None	
Control	Scoured	
T11	0.75	70
T12	0.75	85
T13	0.75	100
T21	1.5	70
T22	1.5	85
T23	1.5	100
T31	2.25	70

T32	2.25	85
T33	2.25	100

### **Fibre Characteristics and Discussions**

The cross-section and surface morphology of the fibres in each group were examined using Scanning Electron Microscopy (SEM).

### **Fibre Linear Density**

The fibre linear density and corresponding standard deviations (SD) of the raw, scoured and treated cotton fibres were shown in Table 2. The fibre linear density was tested according to gravimetric method specified in standard BS EN ISO 1973:1996 and calculated by using following equation;

$$dtex = \frac{10000 (W)}{L \times N}$$

Where, W is weight of bundle of fibres in mg, L is length of bundle of fibres in mm (15 mm) and N is number of fibres in a bundle (N=500). An average of six bundle weight within each sample was obtained..

It is evident in Table 2 that linear density of cotton fibres decreases from 2.02 dtex (control sample) to 1.65 dtex as the concentration of sodium hydroxide and treatment temperature increases. This decrease in linear density was observed to be due to decrease in fibre mass which shows that weight loss occurred in cellulose. These findings are in accordance with previous studies (Warwicker & Hallam, 1970). Additionally there is no statistically significant difference among alkali treatments (T11-T33).

### **Single Fibre Tensile Strength**

The fibre tensile strength and corresponding standard deviations (SD) of the raw,



scoured and treated cotton fibres were shown in Table 2 and compared in Figure 1. The fibre strength was tested according to standard BS EN ISO 5079 (British Standard Institute, 1996) using an INSTRON universal strength tester. It is important to note that Micronaire value cannot be assumed same before and after treatments, therefore, value listed in Table 2 is for raw cotton reference only. It is, for the same reason, that fibre absolute tensile strength are used, as treatments are expected to alter fibre linear density and hence tensile strength. One-way ANOVA was applied on the changes in the fibre tensile strength (gf) and linear density (dtex) of the cotton samples before and after alkali treatment to determine if variance across the alkali treatments were homogenous. The results were expressed as means  $\pm$  SD from three replicates of each treatment and shown in Table 2.

Table 2. Single fibre tensile strength (gf) and linear density (dtex) of untreated and treated samples (raw Cotton micronaire = 5.28).

Sample ID	Tensile strength (gf)	Linear density (dtex)	Difference in linear density (dtex) <sup>±</sup>
R	5.392 $\pm$ 1.375	2.075 $\pm$ 0.109	–
Control	7.272 $\pm$ 2.259	2.021 $\pm$ 0.127	–
T11	7.748 $\pm$ 2.237 <sup>(a)</sup>	1.964 $\pm$ 0.262 <sup>(a)</sup>	0.057 $\pm$ 0.387
T12	5.968 $\pm$ 1.804 <sup>(b,c)</sup>	1.903 $\pm$ 0.097 <sup>(a)</sup>	0.118 $\pm$ 0.072
T13	4.806 $\pm$ 1.510 <sup>(c)</sup>	1.865 $\pm$ 0.305 <sup>(a)</sup>	0.156 $\pm$ 0.382
T21	6.014 $\pm$ 1.735 <sup>(b,c)</sup>	1.880 $\pm$ 0.166 <sup>(a)</sup>	0.140 $\pm$ 0.240
T22	5.563 $\pm$ 2.239 <sup>(b,c)</sup>	1.809 $\pm$ 0.204 <sup>(a)</sup>	0.211 $\pm$ 0.213
T23	5.707 $\pm$ 1.890 <sup>(b,c)</sup>	1.756 $\pm$ 0.225 <sup>(a)</sup>	0.265 $\pm$ 0.171
T31	6.478 $\pm$ 2.458 <sup>(b)</sup>	1.776 $\pm$ 0.193 <sup>(a)</sup>	0.245 $\pm$ 0.169
T32	5.735 $\pm$ 2.057 <sup>(b,c)</sup>	1.691 $\pm$ 0.150 <sup>(a)</sup>	0.329 $\pm$ 0.242
T33	5.512 $\pm$ 1.654 <sup>(b,c)</sup>	1.651 $\pm$ 0.218 <sup>(a)</sup>	0.369 $\pm$ 0.104

Within each column, values with the same letters are not statistically different ( $p > 0.05$ ).

$$^a \text{Difference in linear density} = \text{dtex}_{(\text{control})} - \text{dtex}_{(\text{treatment})}.$$

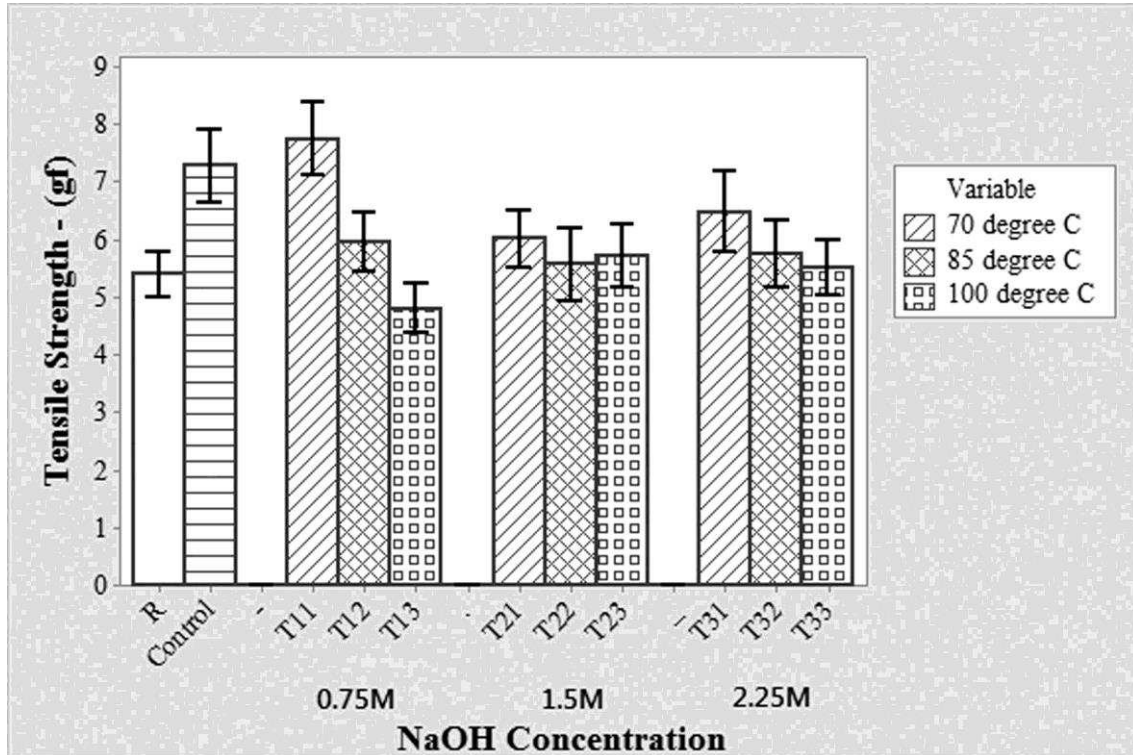


Figure 1. Single fibre tensile strength (gf) of untreated and treated samples; R and Control shows the tensile strength of raw and scoured samples. Error lines represent  $\pm$  standard deviation of the mean

It is evident from Figure 1 that after scouring the fibre tensile strength increased considerably from the raw cotton, this could be attributed to the increase of moisture regain of the cotton by scouring (Kang & Epps, 2009). It is also evident that after alkaline treatment the strength of treated cotton fibres increased, this could be attributed to the elimination of non-cellulose materials from the cellulose (Rajkumar, Manikandan, & Saravanakumar, 2016) and increased moisture regain imparted by the treatments.

The tensile strength of samples following alkali treatments shows a particular trend. For any given concentration of sodium hydroxide, the tensile strength decreases as the temperature is increased from 70°C to 85°C and 100°C. For higher concentration of alkali at both 1.50M and 2.25M, the tensile strength of the treated fibres is nearly the same level around 5.7 gf. For the fibres obtained from all of the nine experiments of combined alkali concentration and treatment temperature, the greatest fibre tensile strength (7.7 gf) is obtained at the processing conditions at 0.75M of the alkali concentration and 70°C in temperature while the smallest tensile strength of 4.8 gf for sample treated in 0.75M of alkali concentration at 100°C. The degree of hydration of alkali hydroxide ions affects their ability to enter and swell cellulose fibres (Wakelyn, et al., 2006). At low concentrations of sodium hydroxide, the diameters of the hydrated ions are too large for easy penetration into the high-order crystalline regions of the fibres (Krässig, 1993; Wakelyn, et al., 2006). But these hydrated ions can penetrate into the cellulose in the primary cell wall which is a low order crystalline region having lower molecular weight and lower degree of polymerization between 2000 to 6000 (Hsieh, 2007).

The alkali treatment always causes a reduction in the thickness of fibre due to the removal of alkali soluble fractions like waxy layer, lignin etc. This treatment produces small voids and thus causes a rougher surface for the fibre (Sreekumar et al., 2009). A detailed review of cellulose dissolution in sodium hydroxide is presented elsewhere (Budtova & Navard, 2016; Medronho & Lindman, 2014). Additionally, Tukey's test (Bewick, Cheek, & Ball, 2004) was used for post-hoc comparisons to determine which treatment differs. All statistical tests were performed at the 95% confidence level ( $p < 0.05$ ). Data was analysed using the Minitab software version 17. Significant

differences between treatment T11 and all other treatments (T12 – T33) was observed at the  $p < 0.05$  level (Table 2).

On effect of temperature, cellulose dissolution is favoured by a decreased temperature (Lindman, Karlström, & Stigsson, 2010). This is suggested to be due to possible conformational changes in the cellulose chain, which would make the polymer less polar at higher temperatures reducing the attractive interactions with the polar solvent. Alternatively, the more polar configurations are suggested to be favoured at lower temperature due to better interaction with the solvent and hence support dissolution (Medronho & Lindman, 2014).

It is interesting to note that the highest fibre tensile strength is obtained with the lowest NaOH concentration and temperature. This means that the positive effect of alkali treatment on coarse cotton fibres could be stronger at even lower concentrations of NaOH and/or lower temperature; the effect of further combinations of the two variables as well as with the combination of the time durations of the alkali treatments are under further investigation and this will be reported in future study.

### **Fibre Morphology**

The Scanning Electron Microscope images of the surfaces and cross-sections of the untreated and treated cotton fibres are shown in the Figure 3 – Figure 6. Following observations were considered while analysing these images:

- For the surface images, any sign of cellulose dissolution from surface erosion or cracking,
- For the cross-sectional images, the cross-sectional swelling (increase in area) or slimming (dissolution), the visibility of pores in the section and fuzziness of the edges.

These effects are shown in the Figure 2.

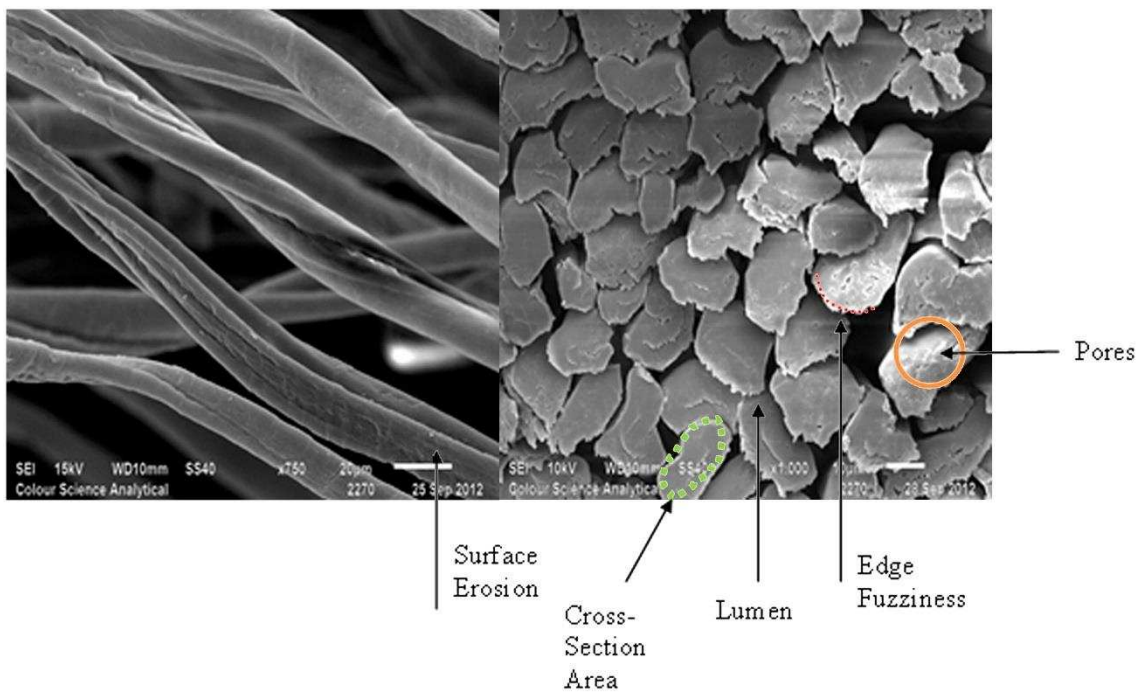


Figure 2. Observed effects in cotton fibres

A comparative and qualitative analysis of the surface and cross-section SEM images shown in Figure 3, having above mentioned effects is utilized to give insight into the changes in the cotton fibre morphology brought about by the alkali treatment. It is evident from Figure 3(A-1), raw cotton have characteristic parallel ridges and grooves (Yonghua & Hardin, 1997).

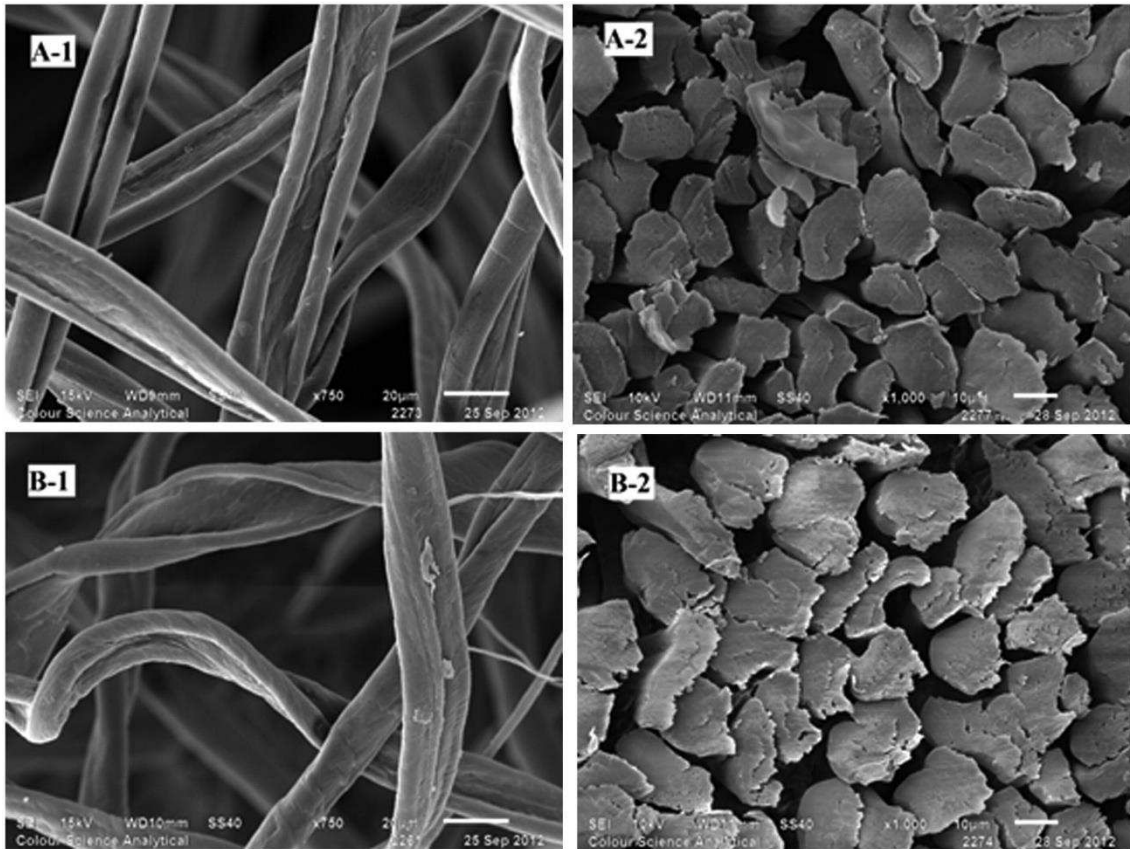


Figure 3: SEM images of the surface and cross-session of (A) raw cotton, (B) scoured cotton

Figure 4 shows SEM images of the surface and cross-session view of cotton fibre treated with different concentration of sodium hydroxide at 70°C.

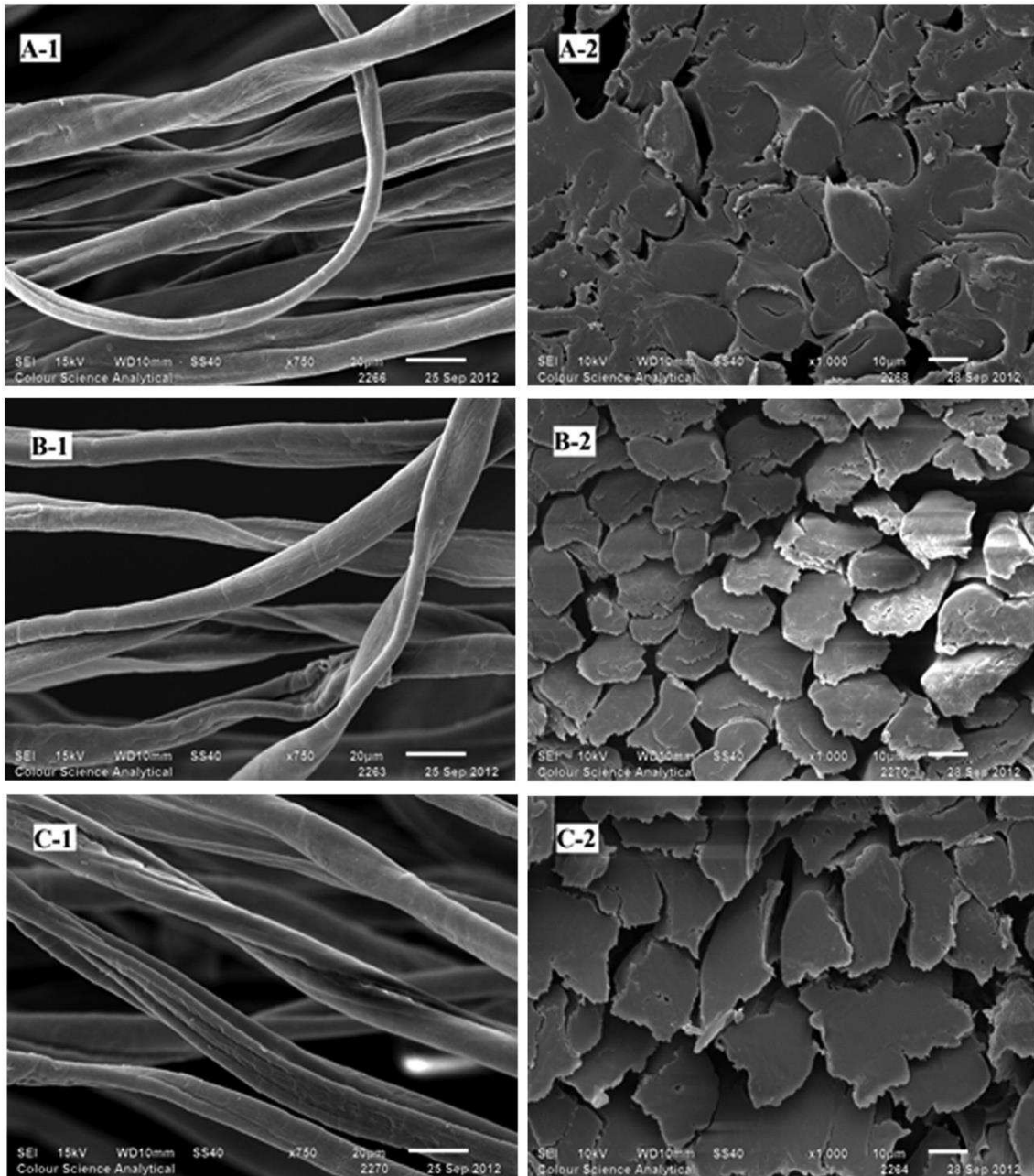


Figure 4. SEM micrographs of the surface and cross-session of cotton fibres treated at different concentration of sodium hydroxide at 70°C (A) 0.75 M (T11), (B) 1.5 M (T21), and (C) 2.25 M (T31)

Sample T11 (0.75M NaOH at 70°C): The surface and cross-section SEM images (Figure 4(A-1) and Figure 4(A-2)) of fibre sample treated at 0.75M concentration and 70°C shows that the surface of the treated fibre looks smooth with cross-section without

noticeable fuzziness. Diluted alkali penetrates only in the accessible regions of the fibres (Wakelyn, et al., 2006) thus the least changes in linear density(dtex) (see Table 2) was observed at the lowest concentration and the lowest temperature, the cross-section of this sample is seen more circular, giving this sample highest tensile strength (7.7 gf).

Sample T21 (1.5M NaOH at 70°C): When the cotton fibres were treated in a higher concentration keeping the treatment temperature at 70°C, the effect will be more than the sample T11. SEM image (Figure 4(B-1)) of the surface of the treated cotton fibres show erosion on the surface; less number of fibres have narrow ribbon shapes in their cross-section (Figure 4(B-2)) which is indicative that cellulose swollen and erosion had occurred while the presence of some flat fibres was also observed and the decreases of tensile strength (6.0 gf) of this sample was noticed.

Sample T31 (2.25M NaOH at 70°C): When the cotton fibres treated in a further higher concentration of 2.25M at the temperature of 70°C, the characteristic parallel ridges and grooves on the surface raw cotton fibres were hardly observed, this indicates the eroded fibre surfaces (Figure 4(C-1)). At the same time, this sample (Figure 4(C-2)) shows considerable swollen fibre of larger cross-section areas. This might explain the slight increases of their tensile strength in comparison with the sample T21, the cotton fibres treated in a lower concentration of 1.5M.

SEM images of the surface and cross-section of cotton fibres treated with different concentrations of sodium hydroxide at 85°C are shown in Figure 5.



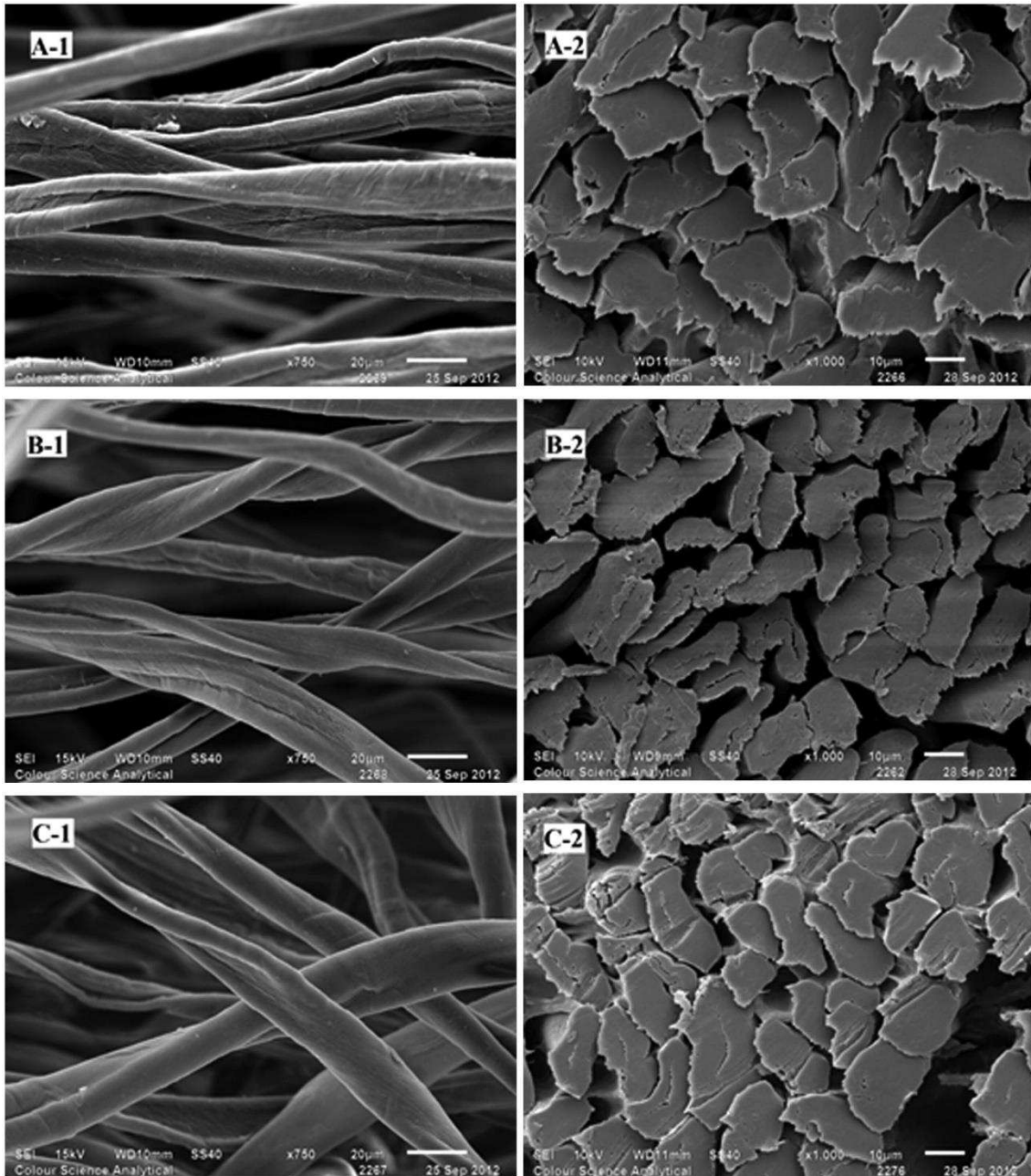


Figure 5. SEM micrographs of the surface and cross-section of cotton fibres treated at different concentration of sodium hydroxide at 85°C (A) 0.75 M (T12), (B) 1.5 M (T22), and (C) 2.25 M (T32)

Sample T12 (0.75M NaOH at 85°C): Most of the fibres treated show apparent swollen fibre cross-sections and fibres having ribbon cross section were hardly shown in the SEM image (Figure 5(A-2)) of the sample treated at 0.75M and a relatively higher

temperature of 85°C, while a few fibres still have characteristic parallel ridges and grooves on the fibre surfaces (Figure 5(A-1)) indicating the occurrence of possible cellulose dissolution and mass losses. It should be noted that this was not observed in sample T11. This differences might be due to the penetration of alkali into the fibre is higher when temperature is increased due to kinetics of the chemical reaction (Wakelyn, et al., 2006).

Sample T22 (1.5M NaOH at 85°C): Upon doubling the concentration than previous sample and keeping the treatment temperature at 85°C, more deterioration on the fibre surfaces were observed (Figure 5(B-1)) while less number of fibres swollen in cross-section was shown (Figure 5(B-2)).

Sample T32 (2.25M NaOH at 85°C): Further increase in the concentration of sodium hydroxide solution to 2.25M keeping the temperature at 85 °C, has led to the swollen of most of the fibres in cross-sections (Figure 5(C-2)) while similar sever fibre erosion on fibre surfaces (Figure 5(C-1)) to the sample T22 was observed, this might be due to severer penetration of alkali into the microstructure of cotton celluloses.

Figure 6 shows the SEM images of the surface and cross-section view of cotton fibres treated with different concentrations of sodium hydroxide at 100°C.

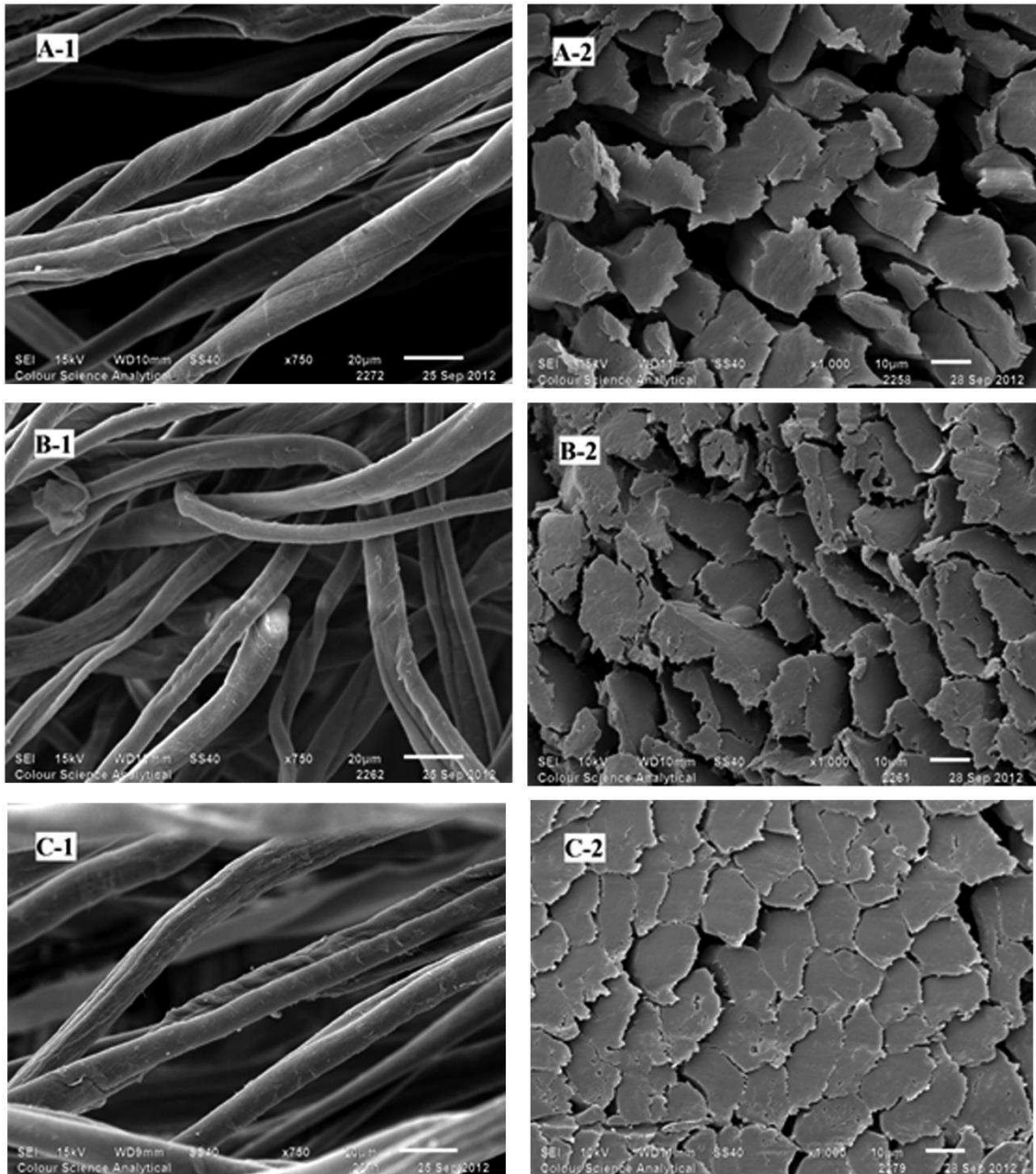


Figure 6. SEM micrographs of the surface and cross-session of cotton fibres treated at different concentration of sodium hydroxide at 100°C (A) 0.75 M (T13), (B) 1.5 M (T23), and (C) 2.25 M (T33)

Sample T13 (0.75M NaOH at 100°C): SEM images of sample T13 (Figure 6(A-1) and Figure 6(A-2)) has shown similar trends to sample T12 (Figure 5A). This is likely to be due to the mechanism that low concentration of sodium hydroxide are limited in

penetration into the ordered regions of the secondary wall due to large hydrated ions (Wakelyn, et al., 2006), the increases in temperature increased activity of alkali on the peripheral regions (primary wall).

Sample T23 (1.5M NaOH at 100°C): In Sample T23 more deterioration on the fibre surfaces was observed (Figure 6(B-1). While most of the fibres swollen in a larger cross-section area were visible, the fibre cross-section was not in more circular but still kept in irregular ribbon shapes (Figure 6(B-2)).

Sample T33 (2.25M NaOH at 100°C): There were fibres showing visible cracks on the surfaces (Figure 6(C-1)), and most of the fibres appearing in almost perfect circular cross-sections (Figure 6(C-2)) which is indicative of the rapid and sever swelling of the fibres. Therefore, cotton fibres treated in sodium hydroxide solutions of relatively higher concentration greater than 2.25M and higher temperature (100°C) could be severely deteriorated.

It is also worth to note that the roundness of the fibre cross section is improved due to fibre swollen effect shown in Figure 4 (A, B and C). Moreover, the areas of the pores in lumen have disappeared after the treatment, impacting positively on the Micronaire value.

#### **4. Conclusions**

In a summary, it was found that coarse cotton fibres treated with sodium hydroxide of low concentrations (0.75M) at lower and higher temperatures behave differently. Cotton fibres treated at lower concentrations (0.75M) and lower temperatures (70°C) favour the improvement of the roundness of fibre cross-sections with an increase of fibre tensile strength. Therefore, it is possible to change the cotton fibre fineness of coarse fibres (thus improving the spinning potential of the fibres) without comprising their tensile

strength by controlling fibre cross-sections and dissolution of cellulose by using sodium hydroxide treatment in lower concentrations. While in the absence of quantitative measure for cross-sectional area, no quantitative conclusions could be given in this paper, this research suggests a novel route to be considered for improving the utilisation of coarse cotton fibres in the cotton textile industries.

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### **Disclosure statement**

No potential conflict of interest was reported by the authors.

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