An Investigation on the morphology, chemical, and physical properties of singed and singed-mercerized twisted cotton yarn

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Abstract:

Singeing and mercerization of fibers affect different properties of yarns and fabrics. This research aims to investigate their effect on the physical and chemical properties of cotton fibers and the effect of mercerization on singed yarns using a combed twisted cotton yarn, count 48/2 Ne on a singeing machine with gas and air pressure of 27 and 12 psi, respectively, to singe the yarns. After that, mercerization was performed on the singed yarns using a sodium hydroxide solution(NaOH) with a concentration of 220 g/L Baumé and a low temperature with tension. The three yarn samples were untreated, singed yarn(S), and singed-mercerized (SM), the surface morphology, hairiness, and chemical and physical structure were investigated using a scanning electron microscope(SEM), Microscopic photograph, Fourier transforms infrared spectroscopy (FTIR), and X-ray diffraction (XRD) with Gaussian fitting measurements. The results demonstrated a decrease in the percentage of yarn hairiness in singeing and mercerization, modification of the yarn surface to become more even and lustrous, and a change in the physical and chemical properties of the yarns.

Keywords:

Singeing, mercerization, cotton yarns, IR, XRD, SEM.

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1. Introduction:

Singeing of yarns and fabrics is one of the textile finishing processes that modifies the surface of textiles and affects some properties such as luster, light reflection, printing, dyeing, and draping,(Wang & Xiao, 2020; Hossain et al., 2021). Singeing takes place on the fabric produced from staple fibers which are known to cause hairiness on the yarns and fabric surface. A ratio of hairiness is removed by singeing (Xia et al., 2009) when the yarn passes up to direct flame. This ratio value depends on the yarn pass speed. The singeing process helps increase the pilling resistance of the yarn because it reduces the percentage of hairiness on the surface of the yarn and fabric(El-moursy & Mohamed, 2015) and thus prevents the interlacing of hair on the yarn and fabric surface which leads to reducing the formation of pilling, thus imperfections, and quality is affected.(Asghar et al., 2021; Kumpikaitė et al., 2021). Few studies have focused on the process of singeing yarns and fabrics, despite its widespread use as a treatment for yarns and fabrics, as an important process before printing and dyeing (Ramaiyan & Subramanian, 2023) and during requesting light-reflective fabrics. Little research focused on studying the effect of singeing on hairiness, printing, and mechanical properties. In the study (Xia et al., 2009), it was proven that singeing removes a large percentage of the hairiness of cotton yarns, which increases with the yarn's thickness. , reduces elongation, tenacity, and unevenness, and increases fineness and imperfections which leads to weight loss. On the other hand, singeing was studied on viscose yarns of cellulosic-based fibers, which showed results opposite to the results of the study(Xia et al., 2009) where the tenacity and unevenness increased, and the elongation and imperfections decreased (Ramachandran, & Thirunarayanan, 2015). All the research that has studied the effect of singeing on the properties of yarns and fabrics has not been devoted to studying the effect of singeing on the structure of fibers, which can affect the properties of yarns and fabrics.

Cotton products are widely used around the world because of their comfortable properties in clothing (Lin et al., 2022). Alkali, organic solvents, inorganic salt solutions, and acids cotton treatments are some of the most important requirements for treatments used to modify and improve their properties and increase the purposes of their uses(Remadevi et al., 2023). Cotton mercerization is a physico–chemical process (Brahma et al., 2018) that has been used for a long time and is still at the forefront of processing because it gives the cotton luster, and increases tenacity and smoothness to its surface (Hilal et al., 2020; Remadevi et al., 2023). Mercerization is done on fabrics or yarns in the form of hanks using slack or under tension with different temperatures(Patil et al., 2019). The fibers swell, and shrink, the convolutions are straightened, and the cross-section of the cotton fibers changes from kidney shape to almost rounded as a result of mercerization(Remadevi et al., 2023). Crystallization is affected, depending on the conditions used in mercerizing, as well as the type of alkali used and its concentration ratio (Lin et al., 2022). Either sodium hydroxide or liquid ammonia was used to mercerize cotton fabric to allomorph cellulose II. When both were used together, a mixture of allomorph cellulose II with cellulose III was formed(Lin et al., 2022; Manian et al., 2022).

Mercerization improves the Physical, and chemical properties, and can remove ratio of the yarn hairiness(EL-Moursy& Mohamed, 2015) which improves dye exhaustion (Hilal et al., 2020), and increases total hand values (THV) (Patil et al., 2019).

This is the first study concerned with the change in the physical and chemical structure of the cotton fiber as a result of the process of singeing and mercerizing for singed yarns through investigation using optical microscope examination, SEM, FTIR, and XRD. Calculate the crystallite size using Gaussian fitting. , the crystallinity index and degree of crystallinity.

Experimental:

Material:

100% cotton with a count of 48/2 Ne, and a twist factor of 4.5 from El-Mahala company. Sodium hydroxide (NaOH) solution with a concentration of 220 g/L

Singeing treatment of yarns(S)

Yarn cones are treated by passing the yarns on the direct flames of fire at speeds of 800 m /min by SSM Swiss machine with gas and air pressure(27, 12 psi), respectively. The yarns became a light yellowish color

NaOH treatment (Mercerization) of Singed yarns(SM)

Yarn cones are converted to hanks by reeling machine and then treated with a solution of NaOH at a concentration of (220 g/L) by machine with the mercerization condition: immersion time (1min), immersion tension (132 cm), fixed dimensions tension (136 cm), fixed dimensions time(5s), washing tension (138 cm) washing time(2.5 min with heat 110° C) NaOH solution heat (18° C), drying heat in the oven (110° C), and PH (7)

Optical microscope examination

Optical microscopy uses visible light with magnifying lenses to examine the hairiness of yarn samples and take a photograph

Scanning electron microscope (SEM)

Untreated and SM yarn samples were scanned by using the scanning electron microscope (Tescan vega 3 SBU, Czech Republic) at an accelerating voltage of 15 kV. Samples were mounted on aluminum microscopy stubs using carbon tape, then coated with gold (Au) for 120 sec using Quorum Techniques Ltd, sputter coater (Q150 t, England)

Fourier Transforms Infrared Spectroscopy Analysis (FTIR)

The IR spectra were obtained in transmiation mode by co-addition of 24 scans with a resolution of 4 cm−1, over the range from 4000 to 400 cm−1. It was recorded using Bruker ALPHA II compact FTIR spectrometer, which was equipped with a Platinum ATR Quick Snap TM module featuring a monolithic diamond crystal.

X-ray diffraction analysis (XRD) and Measurements

The structure of treated and untreated fabric The structure of treated and untreated yarn samples was examined with the X-ray diffraction spectrum of Shimadzu (XRD- 6000 model- with Cu-K α radiation (λ = 1.5418Å) at a range of $10 \leq 2\theta \leq 70$. The crystallite size of the untreated and treated yarn samples was calculated using the Scherrer equation.

$$\text{Crystallite size}(D\ \text{nm}) = k * \lambda/\beta * \cos(\theta) \quad (1)$$

Where θ is the Bragg's angle in degree, k = 0.94, called Scherrer constant, λ is the wavelength of the used X-ray (λ=0.15418 nm), β is the value of full width at half maximum (FWHM) given in radians,(Condurache et al.,2009; Fatimah et al., 2022) The peaks were fitted by the origin software with Gaussian functions. To calculate the Crystallinity Index (CrI) of crystalline Segal ' using the equation:

Crystallinity Index (CrI) =(I002 −Iam/ I002)*100 (2)

Where I002 is the intensity of the crystalline peak at 2θ

Iam is the intensity of the amorphous at 2θ (Diab et al., 2023)

To calculate the degree of crystallinity (Cx) from the XRD pattern using the equation:

degree of crystallinity (Cx) = (Ac /Ac+Aa) *100 (3)

Where Ac is areas under the crystalline peaks in the XRD pattern

Where Aa is areas under the amorphous background in the XRD pattern (Lin et al., 2022)

Results and discussion

Surface morphology

Investigation of the surface modifications that occurred to the singeing and mercerization-treated yarns compared to the untreated ones through the image of the hairiness of the yarns as shown in Fig.1a,b, and c. The untreated yarns had a high percentage of hairiness as shown in Fig. 1a, and it decreased after the singeing process as shown in Fig. 1b as a result of the yarns being exposed directly to flame, where a large portion of the lengths of the fibers protruding from the stem of the yarns were removed (Asghar et al., 2021; Xia et al., 2009). After treating the S with a NaOH solution at a concentration of 220 g/L with a temperature of 18˚ C to mercerize the yarns, the percentage of hairiness decreased compared to the signed yarns only as shown in Fig.1c. This may be attributed to the fact that the hairs protruding from the stem of the yarns are free and are not affected by the tension applied to the yarn hanks, so they

swelled more and thus shrinked more and decreased their length so hair decreases, or may be due to the straightening of the convolution that caused the hairs to become loose and fall out, in addition to the effect of singeing in decreasing the percentage of hairiness.

(a)

(b)

(c)

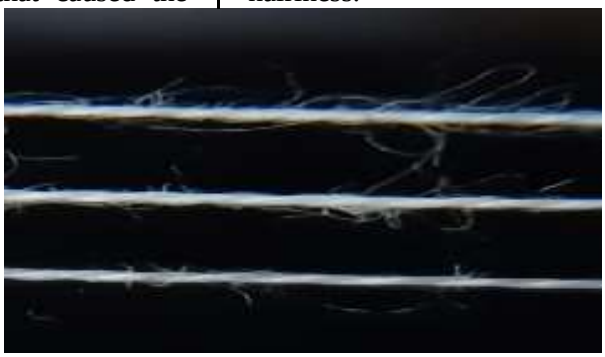
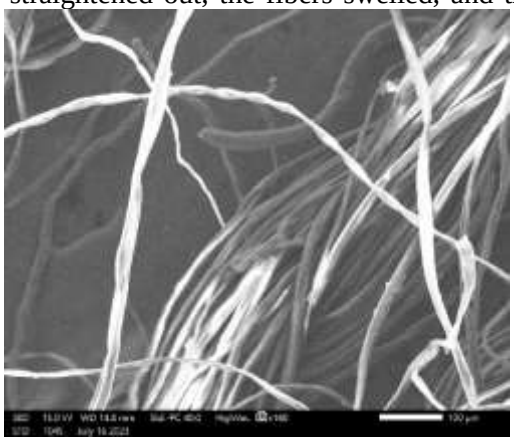
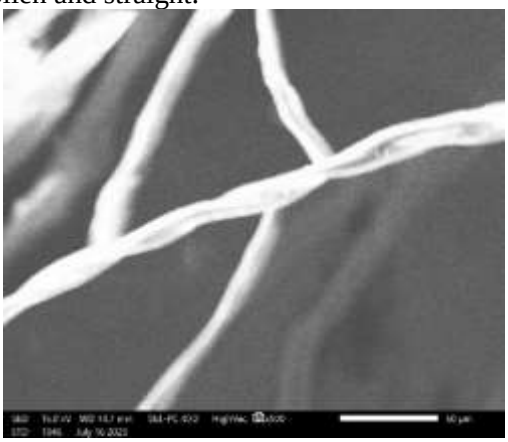


Fig.1 Microscopic photograph of the difference in hairiness between, untreated(a), S(b), and SM(c) cotton yarns

SEM images show the difference between untreated surface and SM yarns, as shown in Fig. 2. Untreated yarns appear in a convoluted ribbon shape along the longitudinal axis of the fibers as shown in Fig.2a and b, which distinguished them from other fibers. After the mercerization and the treatment of cotton with NaOH, the convolutions were straightened out, the fibers swelled, and they were almost rounded in shape (Lin et al., 2022) as shown in Fig. 2 c and d. They had more luster due to the leveling of the surface and the removal of a percentage of the hairiness. This was attributed to the singeing process before mercerization and the penetration of the NaOH solution from the amorphous region inside the fibers to become swollen and straight.



(a)



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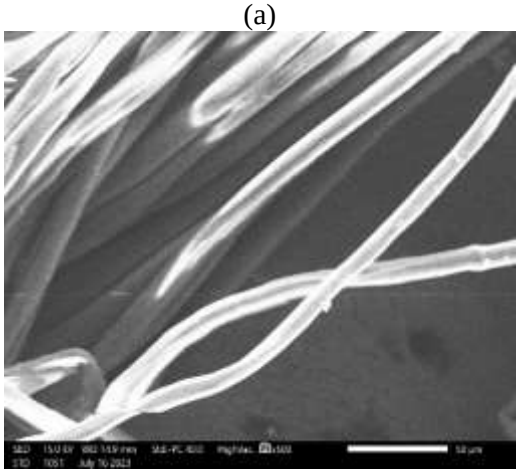


(C)

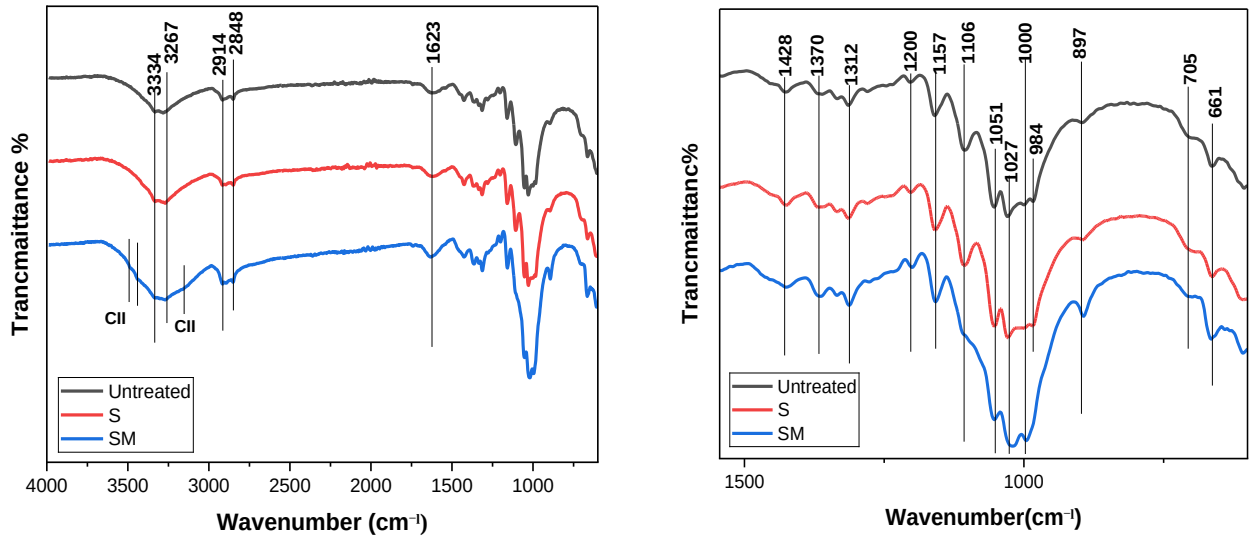
(d)

Fig.2 SEM images(a,b)untreated yarns (c,d) SM yarns treated with NaOH

Fourier Transforms Infrared Spectroscopy Analysis (FTIR)

Infrared spectroscopy analysis of untreated, S, and SM yarn samples, as shown in Figures 3a and b. In untreated, S, and SM, the broader peaks were absorbed at 3334 and 3267 cm−1 stretching vibration OH group they were more broader in SM, which contributed to cellulosic components such as H2O (French & Kim, 2018), and three weak peaks appeared only in SM at 3439, 3491and 3156 cm−1 attributed to CII(Mihajlović et al., 2020). The absorption peak bands at 2848 and 2914 cm−1

stretching while 897 cm−1 (Ilyas et al., 2018) rocking vibration these peaks corresponded to C-H in methyl and methylene groups of cellulose (Huang et al., 2022). A broad peak around 1623 cm−1 stretching vibration of the C=O group in hemicellulose(Vârban et al., 2021) and appeared in untreated, S and SM. The peaks around 1428 and 1370 cm−1 bending vibration of CH2(Ilyas et al., 2018) and bending of OH (Khenblouche et al., 2019), respectively. The peaks at 1312,1200 and 1157 cm−1 were attributed to C-O and C-C stretching and were indicated to be carbohydrates of cellulose(Ilyas et al., 2018). A sharp peak C-O or C-C stretching referred to as an asymmetric ring of glucose appeared at 1106 cm −1 (Mihajlović et al., 2020)in untreated and S while disappeared in SM due to being treated with NaOH which led to a change of cellulose structure and the same way a peak stretching C-O group at 984 cm−1. The peaks at 1051,1027and 1000 cm−1 belonged to the C-O group which increased intensity with a slight shift in SM compared with untreated and S which was affected by NaOH. A peak at 897cm−1 is attributed to C-O-C arising from the polysaccharide components (Margariti, 2019) which appeared sharp with intensity in SM. Two peaks at 705 and 661cm−1 corresponded to OH cellulose or CH2 rocking and OH cellulose /lignin /hemicellulose, respectively (Bouramdane et al., 2022).



(a) (b)

Fig.3 FTIR of the untreated, S and SM of cotton yarns

From the above, it was found that there was a change in the region below 1500 cm−1 when treating the S with NaOH solution, which is the fingerprint region, which distinguished each material structure from another. It disappeared and new bonds were formed in this region, while the functional groups increased, in the region above 1500 cm−1, and on the other side, there was no change in the S.

The X-ray diffraction (XRD):

The XRD pattern of the untreated, S, and SM samples, as shown in Fig. 5, and Table 1, revealed four peaks for each of the untreated and S samples. These peaks appeared at positions 2θ at 15.2˚, 16.8˚, 23.2˚, and 34.9˚ for the untreated sample and 15.1˚, 16.6˚, 23.4˚, and 35.6˚ for the S sample. These peaks are attributed to planes((1-10),(110),(200), and (004), respectively for both untreated and S samples and responsible for cellulose I (CI) according to Miller indices(French, 2014). By comparing the diffraction patterns between the S and untreated samples, a significant decrease in the intensity of the sharp main peak was observed as well as a slight decrease in the intensity of the peak at 34.4˚. The peak at 15.6˚ almost disappeared after singeing treatment. This decrease may be due to the heat of singeing, which affected the cotton chains and led to them moving away from each other and becoming unoriented and unordered, and so, increasing the amorphous areas. This was consistent with what the IR analysis showed, that the bonds between the atoms and molecules were not affected by the singeing. Perhaps Van Derwaals and some hydrogen bonds were broken.

The pattern of the SM cotton yarns exhibited five peaks, shifted from the original peak positions of the untreated sample, which were at 2θ values of 12.7,16.7, 21.2, 23.3, and 35.6˚. These peaks were attributed to planes (1-10/100),(110),(110),(020), and (004), respectively according to Miller indices(French, 2014). Two peaks in SM appeared after treatment with NaOH solution at 2θ values of 12.7˚ and 21.2˚. Additionally, the main peak at 23.3˚ increased in intensity by a large amount compared to the intensity of the S peak and by a small amount compared to the untreated peak at the

same position of 2θ with a slight shift of the plane which became (020). An increase in the intensity of the peak was also seen at 35.5˚. It was sharp compared to S and untreated in the same position approximately. The peak in S at 15.2˚, disappeared and fused with the neighboring peak at 16.6˚ appearing together in one peak at 16.7˚, and became broadened in SM. All these were due to the NaOH penetration through both amorphous and crystalline, causing the fibers to swell and combine with cellulose, forming sodium cellulose(Lin et al., 2022), and converting CI to cellulose II(CII) after washing and removing NaOH.

Table 1. Miller indices with planes (hkI) of untreated, S, and SM of cotton yarns

Samples	2θ	Miller indices hkl
Untreated	15.2	(1-10)
	16.8	(110)
	23.2	(200)
	34.9	(004)
Treated S	15.1	(1-10)
	16.6	(110)
	23.5	(200)
	35.6	(004)
Treated SM	12.7	(1-10/ 100)
	16.7	(110)
	21.1	(110)
	23.3	(020)
	35.6	(004)

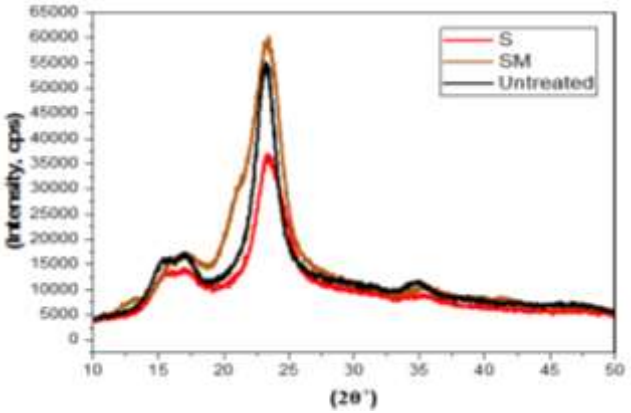


Fig. 5 XRD of untreated, S, and SM of cotton yarns

As a result of singeing the yarns over direct flame and in the presence of air, they became a light yellowish color, and this was attributed to the trapped center phenomenon that occurs in amorphous materials such as cotton. When the yarns were treated with singeing, the free radicals gained thermal energy that moved them upward to settle in the trapped center leaving holes in their place. This acquired energy was responsible for the color of the trapped center(Abbaszadeh et al., 2019) so that the slightly yellowish color appeared.

Gaussian function software of Origin program was used for peak fitting as shown in Fig.5 a,b,c to determine(FWHM) and (DÅ) using Equation(1). Origin software was used to determine the CrI using Equation (2) and to calculate Cx using Equation (3). The results are summarized in Table 2. The results in Table 2 and Equation(1) showed that narrow peak patterns with small FWHM contained large size (DÅ) and vice versa(Fatimah et al., 2022). The large Crystallite size reduced the distances between atoms and thus increased the ability to form more bonds and crystallinity, on the other hand, the small crystallite size increased distances between atoms and thus decreased the ability to form more bonds and crystallinity.

In S, the CrI was quite low and recorded (71%) compared to the untreated ones (79%). This was attributed to the effect of the flame on the cellulose chains leading to the disintegration of some of them and the increases of the amorphous region followed by decreased CrI. The yarns mercerizing after singeing them repaired what was caused by the singeing, and restored some of the amorphous regions to crystallinity ones, due to NaOH ability to easily penetrate the amorphous regions, especially those that singed and form new bonds and bring the chains closer together, thus increasing the crystallinity regions followed by increased CrI (75%) compared to S (71%)

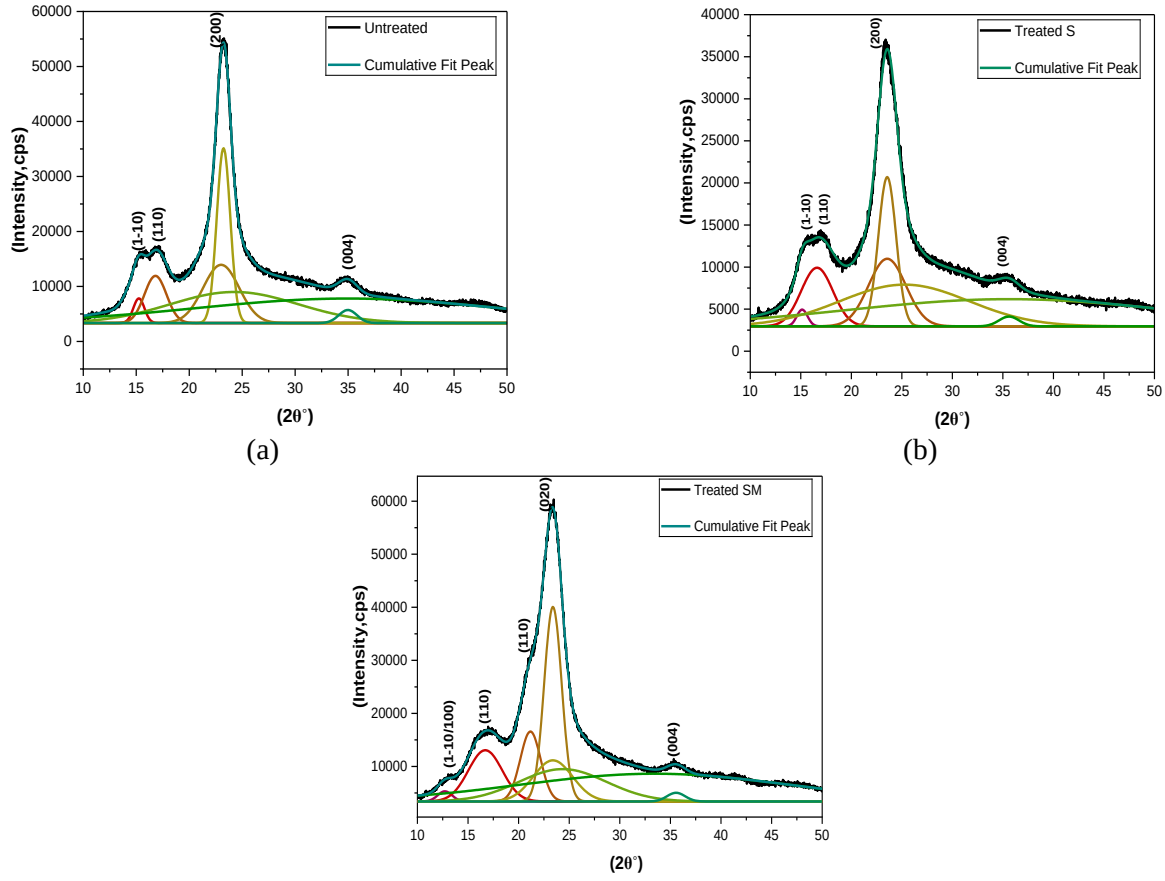


Fig. 6. Gaussian fitting of XRD of untreated(a), S(b) and SM(c) of cotton yarns

Cx in SM increased by about 10% over S because the singeing decreased CrI which led to an increase in amorphous in addition to the elimination of non-cellulosic substances as a result of treatment with NaOH solution which created empty spaces that helped the ability to arrange and order the chains with orientation in the longitudinal direction of the fibers axis to record the highest Cx value of (40%). Regarding the decrease of Cx in the S (30%) compared to untreated (33%) as a result of the singeing process, it helped the chains to move away from each other and became unable to rearrange and order their chains again due to the absence of any factor contributing to this arrangement.

Table 2. 2θ, FWHM, D, CrI, and Cx at of untreated, S, and SM of cotton yarns

	untreated	S	SM
2θ	-	-	12.71341
FWHM	-	-	1.67825
D Å	-	-	49.78519518
2θ	15.24862	15.13598	-
FWHM	1.23641	1.5405	-
D Å	67.7598666	54.37714696	-
2θ	16.81449	16.60869	16.71176
FWHM	2.45127	3.6867	3.94927
DÅ	34.24361667	22.76241919	21.25184237
2θ	-	-	21.17156
FWHM	-	-	2.0383
DÅ	-	-	35.63035744
2θ	23.24007	23.55364	23.3924
FWHM	1.50613	2.06668	2.0383
DÅ	56.28717667	41.04353529	41.60283605
2θ	34.97963	35.60583	35.56079
FWHM	2.01617	2.3347	2.10659
D Å	43.18247633	37.35584515	41.39566403
CrI	79%	71%	75%
CX	33.78%	30.32%	40.66%

From the previous, it was clear that the singeing process had a positive effect on the mercerization process. It led to a decrease in CrI and facilitated the penetration of the NaOH solution to reach more places. Thus, the effect of mercerization appeared on the singed yarns, which was represented by an increase in Cx. Both singeing and mercerization affected each other.

Conclusion:

After performing singeing and mercerization of the singed yarns on the twisted cotton yarns and investigation through optical microscopic, SEM, IR, and XRD analyses, modifications appeared on the surface of the yarn and its properties chemical and physical were changed. The main results were as follows:

- The percentage of hairiness in the S decreased and the yarn became lustrous with yellowish color and it decreased even more after mercerization in SM compared with untreated.
- The fibers swelled after treatment with NaOH solution, flattened their surface, and increased their luster compared to untreated and S
- Increase in functional groups and change in the fingerprint area in SM according to chemical bonds and change of CI to CII
- The S did not change their chemical bonds, nor did the structure of the cellulose.
- A significant decrease in the CrI of S compared to untreated and SM ones
- The highest degree of Cx was the SM, followed by untreated, and S.
- The size of the crystallite differed at 2θ. The sharp peaks contained larger sizes than the broad peaks, and thus an inverse relationship between the crystallite size and FWHM.

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