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## The effect of glycine treatment on the morphology and tensile properties of cotton yarn

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### ABSTRACT

In this study, a detailed exploration of the effects of glycine treatment on the morphology and tensile properties of cotton yarn is presented. From the cross-sectional morphological studies, it can be noticed that glycine treatment at pH 11 swelled the cotton fibres. Linear density measurements showed that the yarn weight increased after glycine treatment irrespective of the pH. The increased linear density may be due to the weight gained by the yarn because of glycine treatment as well as shrinkage. The results of the tensile properties of cotton yarn treated with glycine showed that glycine treatment protected the samples from the significant drop in yarn tensile strength at acidic pH. The breaking strain was increased significantly in pH 11 glycine treated yarn by 70%. The glycine treatment at acidic condition has also changed the strain value, at pH 4, where the strain value increased by 38%.

### ARTICLE HISTORY

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### KEYWORDS

Glycine; cotton; yarn; morphology; mechanical properties

### Introduction

Cotton fibres swell when treated in alkalis, acids, organic solvents, inorganic salt solutions and with water to varying degrees depending on pre-treatment, the concentration of the swelling medium and the existing morphology of the fibre, e.g. the degree of cell (fibre) wall thickening (Evans & Jeffries, 1970; Lokhande, 1978; Mantanis et al., 1995; Moore et al., 1950; Warwicker, 1969). Swelling cotton fibres are used in the cotton industry to improve the physical and chemical properties of cotton fabrics (Lokhande, 1978; Rousselle et al., 1976; Warwicker et al., 1966). Of all past and potential swelling agents, caustic soda has been the major swelling agent and is the benchmarking agent. Caustic swelling of cotton is used to improve yarn and fabric appearance, dyeing ability and mechanical properties (Warwicker et al., 1966). The outcome obtained from caustic mercerisation is significant, however, the processing route, especially the high concentration of caustic required for the treatment is not environmentally friendly. In the light of these problems ‘greener’ more environmentally friendly agents have been proposed (Cuissinat et al., 2008). An aqueous glycine treatment that cotton fibres can be swelled with aqueous glycine solutions was reported by Remadevi et al. (2017). The authors also reported the glycine treatment improved the tensile properties of cotton fibre treated in the slack form (Remadevi et al., 2018). However, the glycine treatment on cotton in yarn form has been not reported yet.

Rebenfeld et al. studied the effect of mercerisation treatments on cotton yarn (Rebenfeld, 1961) and has reported that in the mercerisation of yarn, the concentration of alkali, time of treatment, treatment temperature and yarn twist are important. The yarn twist is important in terms of reagent penetration into the yarn structure, in view of its effect on the dimensional changes, which the fibres undergo during mercerisation. They also have studied the fundamental difference between single fibre and yarn mercerisation. Their study showed that in fibre treatments the changes in the properties reflect only in the changes in the fibre’s fine structure, although in yarn treatments the changes in the yarn’s structural properties were also affected (Rebenfeld, 1961).

Besides, in the area of slack mercerisation of the cotton yarn, there were reports about the tension mercerisation of cotton yarn (Nelson et al., 1976). For example, M. Nelson et al. studied the mechanical properties of yarn and compared the properties between the ammonia and caustic mercerised yarn. In their work, they prepared yarns from less mature and fully matured cotton fibres. These yarns were then mercerised in liquid ammonia in a continuous process. The authors also reported on the mercerisation performed on the yarn in its skein form under various tensions (Nelson et al., 1976). Both mercerisation treatments had changed the mechanical properties under comparable conditions. Tensioned mercerisation increased the breaking strength and tenacity but decreased the elongation (strain before break). It was found that slack mercerisation in caustic soda allowed a higher elongation at break than the

ammonia mercerisation treatment (Nelson et al., 1976). In this work, Nelson et al. noted a significant difference between the reagents during each mercerisation treatment. When skeins were slack swollen and then re-stretched, a much greater force was required to re-stretch ammonia-swollen skeins in comparison with the caustic-swollen material (Nelson et al., 1976).

Furthermore, there are works reported on the mercerisation treatments on rotor spun yarn, open spun and ring spun cotton yarn (Hari et al., 1985; Hunter & Andrews, 1977; Pillay & Nagaraja, 1984). Hari et al. have found that mercerisation has improved the tenacity of rotor-spun yarn significantly: improvement was substantial at low twist levels. In their work, they have hypothesised that the increase in yarn tenacity, due to tension mercerisation, at low twist levels is mainly because of the improved fibre packing, and at high twist levels, it is because of the combined effect of fibre packing, fibre orientation, and improvement in fibre tenacity (Hari et al., 1985). It was reported that the stress-strain properties of open end and ring spun yarns have shown structural differences between the yarns and the changes in the structure resulting from the mercerisation and stretching (Pillay & Nagaraja, 1984). It was also found that the lustre of both open end and ring spun yarn's lustre has improved after mercerisation; however, the open spun yarn has a lower lustre than the ring spun yarn. Moreover, in the case of shrinkage, the open spun yarn has shown a higher shrinkage than the ring spun yarn (Pillay & Nagaraja, 1984).

The natural wax present on the cotton fibres' plays an important role in the physical processing of the fibres (Cui et al., 2002). Cotton wax is a chemically complex matter containing a high molecular weight of acids and alcohols (Clifford & Probert, 1924; Price et al., 2002). Regardless of the nature of the wax present on the cotton fibres, the cotton wax is very effective in promising adequate cohesion for web formation at the card/draw frame, allowing the fibres to slide each other during the drafting process (Price et al., 2002). Removal of wax from the cotton yarn before knitting/weaving creates problems. The interest for carrying out this research study is because scouring prior to the glycine treatment removes the cotton wax rendering any post-treatment mechanical processing, e.g. carding, drawing, and spinning, of the fibre difficult due to the lack of cotton wax. Therefore, it hinders the fibre treatment and thereby, the scalability in the cotton processing industry. However, if the treatment can be conducted on yarn and the physical properties of the yarn can be improved through the glycine treatment, then it becomes an attractive proposition. Furthermore, in a yarn, fibres are assembled with twists and hence the structure is compact. Therefore, it was of interest to understand how the treatment enables the glycine to penetrate the yarn structure and changes the fibre at various locations along the yarn cross-section. Thus, here in this work, we have studied the effect of glycine treatment on the morphology and mechanical properties of cotton yarn. In addition to this, the present work has also included a study on the shrinkage and linear density of the glycine-treated yarns.

## Materials and methods

Australian ring spun cotton yarn (11 Tex) supplied by the Commonwealth Scientific and Industrial Research Organisation (CSIRO) Manufacturing Business Unit, Waurn Ponds, VIC Australia was used in this study. Sodium hydroxide (NaOH), Glycine and hydrochloric acid were purchased from Sigma Aldrich and were used as received. All reagents were of analytical grade and all aqueous solutions were prepared with deionised water. Table 1 lists the applied treatments on yarns and their respective sample codes.

### Scouring of cotton yarn hanks

Around 10 g of raw cotton yarn samples (11 tex yarn) were scoured using a conventional scouring method to remove wax (Buschle-Diller & Zeronian, 1992). Scouring was done with the yarn hanks in a slack state using aqueous NaOH (4%) at 100 °C for 90 min (at a fibre-to-liquor ratio of 1:100). Scoured yarns were washed with copious amounts of deionised water until the fibres were free of any traces of the scouring agent. After washing, samples were dried overnight (12 h) in an oven at 50 °C. The scouring of yarns was done in a reflux system.

### Glycine treatment on cotton yarn hanks

The scoured and rinsed cotton yarn samples were treated with an aqueous solution of glycine (20% w/v) in a 1:100 yarn to liquor ratio at 100 °C for 24 h under reflux with constant stirring. The yarn was treated in a slack state at different pHs i.e. 3, 4, 7 and 11, with the pH of the solution adjusted using either sodium hydroxide (10% w/v) or hydrochloric acid (32% w/v). Control treatments at different pHs without glycine were also applied. At the end of each treatment, yarn samples were thoroughly washed with deionised water and dried overnight in a convection oven at 50 °C.

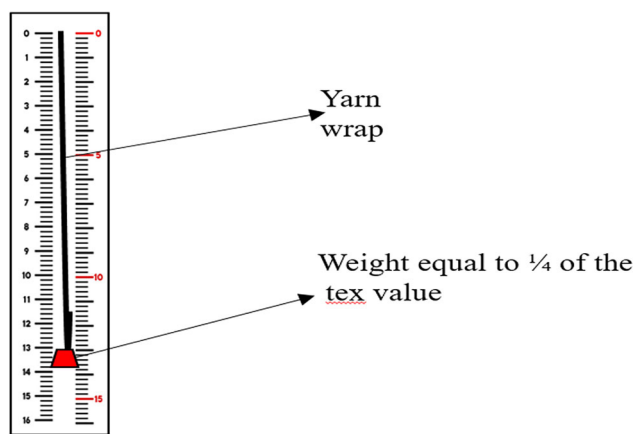
## Characterisation

### Change in morphology

The change in cross sectional morphology of cotton yarn before and after treatments were observed using Scanning Electron Microscopy (SEM, ZEISS Supra 55 SEM VP microscope -Karl Zeiss, Germany). Treated and untreated fibres were embedded in TAAB TLV medium resin and then sectioned into 100–200 nm slices using an ultra-microtome (Leica EM UC6 Ultra microtome). The as-prepared cross-sections were gold sputter coated (Bal-Tec Sputter Coater SCD

**Table 1.** Yarn treatments and codes.

Number	Treatment	Code
1	Untreated cotton	UTC
2	Scoured cotton	SC
3	Treated at pH3(controlled sample)	W3
4	Glycine treated at pH 3	CG3
5	Glycine treated at pH 4	CG4
6	Treated at pH 4 (controlled sample)	W4
7	Glycine treated at pH 7	CG7
8	Treated at pH 11(controlled sample)	W11
9	Glycine treated at pH 11	CG11



**Figure 1.** Schematic representation of the measurement of shrinkage (%) of yarns.

050) and then observed under a Zeiss Supra 55vp SEM at an accelerating voltage of 3 KV.

### Linear density of yarn

The linear density of yarn was measured using ASTM D1577 – 07 method. The conditioned (conditioned at 21 °C and 65% RH for 48 h prior to testing) yarn samples were wound on reels as skeins (1 m) and weighed. The linear density of the yarn is computed from the mass and length of the skein.

### Yarn shrinkage

The percentage of shrinkage of the yarns was determined according to ASTM standard method (D2259). For shrinkage (%) measurement, the conditioned yarn in the form of the wrap was mounted on a hook attached to a frame at one end. A load was hanged on the other end based on the respective yarns one fourth of tex value (Figure 1) (Hebsiba & Thambiduraia, 2007). The initial and final lengths were noted using a scale. The shrinkage percentage of treated yarn was calculated using the following Equation 1 (Hebsiba & Thambiduraia, 2007).

$$\text{Shrinkage}(\%) = \frac{(\text{Scoured yarn length} - \text{Treated yarn length})}{(\text{Scoured yarn length})} \times 100 \quad (1)$$

### Yarn tensile testing

The tensile properties of slack treated yarns were tested on Instron tensile tester using a 100 N load cell and with a gauge length of 10 cm (strain rate 20 mm/min). Conditioned yarns were mounted between the jaws, and load and extension at break were determined. The sample size (number of tests) used for the yarn tensile testing was 100.

## Results and discussion

### Morphology of treated yarn

To understand the swelling effect of glycine on the cotton yarn at various sections from outside to inside, cross sections of the yarn samples were examined.

Cross-sectional SEM images of treated and untreated yarn samples are presented in Figure 2. The difference in the cross-sectional shape of fibres in scoured cotton yarn (see Figure 2(b)) and that of the CG11 (see Figure 2(g)) is clear; fibres swelled throughout the yarn cross section. However, there was no swelling evident at pH 11 without glycine (w11); shown in Figure 2(d). In CG3 cross sections variation in shape is evident (Figure 2(e)); i.e. fibres in the outer part of the yarn swelled and turned round but not in the core of the yarn. The controlled samples at respective pH didn't show any appreciable change in cross-sectional morphology. Overall, it can be observed that the fibre cross-sectional shape of the yarn was changed after the glycine treatments pH 3 and 11 (CG11 and CG3); shown in Figure 2(e,g).

The swelling of cotton yarn with glycine is due to the interaction between the amphoteric natured glycine and the hydrogen bonds within cotton cellulose. The swelling is a low homogeneous swelling with no dissolution of the cellulose (Goldthwait, 1965). At acidic pH, glycine solutions can act as an acid (Xu et al., 2003) which together with the relatively high temperature of the treatment caused swelling in cotton (Remadevi et al., 2018). The carboxyl group of an amino acid forms esters by reacting with –OH groups at acidic pH (Bose et al., 2012). Thus, at acidic pH, the glycine formed esters with cotton cellulose. In an alkaline pH, the nature of glycine changes into a base. It was reported that bases and acids can swell the cotton cellulose by altering the intermolecular hydrogen bonding (Rowland & Howley, 1988). Thus, the swelling of cotton yarn could be due to the change in intermolecular hydrogen bonding in the cotton cellulose.

### Measurement of shrinkage

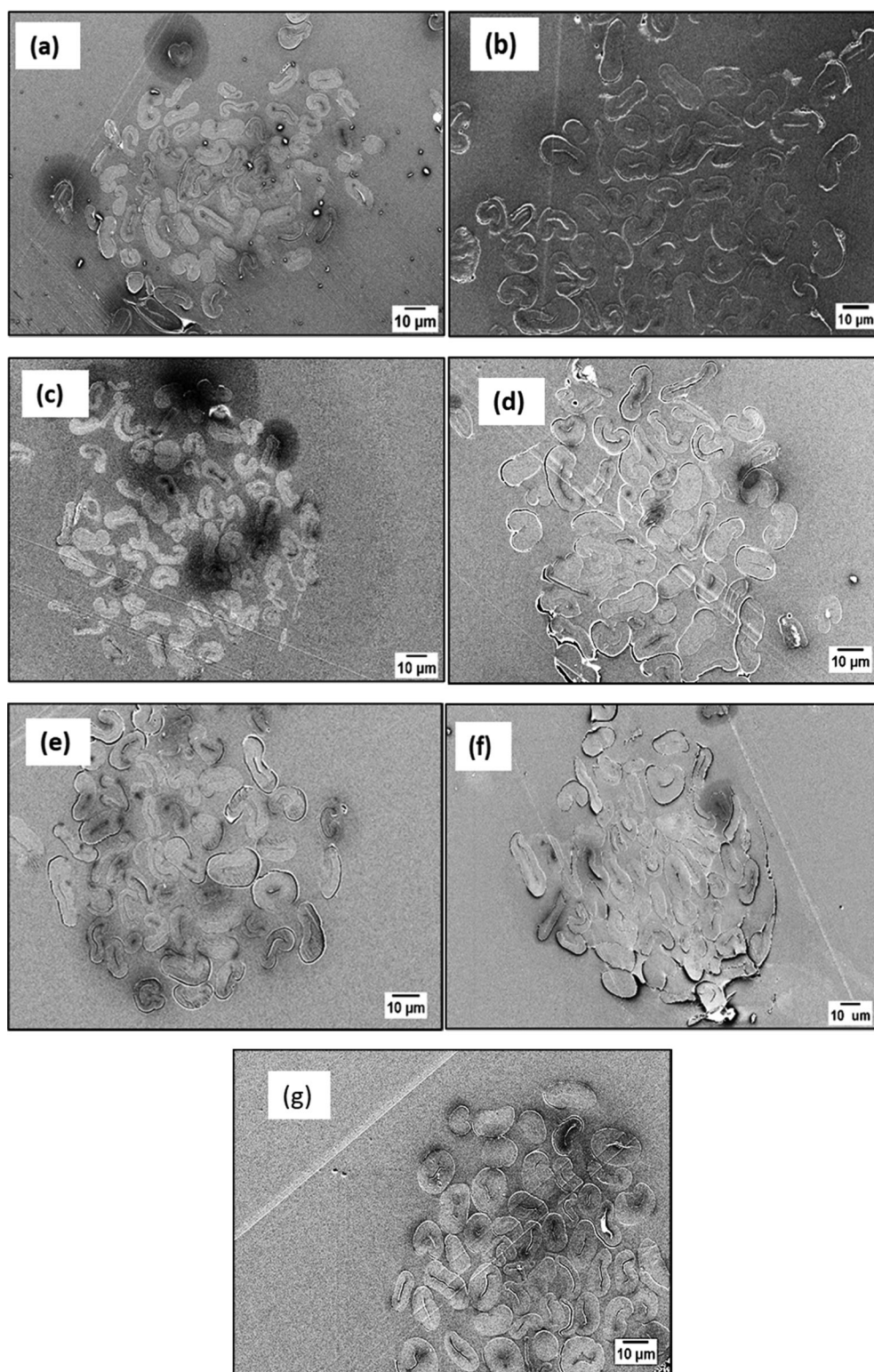
The shrinkage of yarn that occurred with treatments was measured using ASTM standard (D2259). Seven test specimens per sample were measured and the average of shrinkage obtained is shown in Figure 3.

From the shrinkage measurement, it was found that CG11 had significant shrinkage compared to other samples. The results further support the morphological changes of CG11 cotton both in fibre and yarn forms. The shrinkage could be due to the swelling of the fibres in the yarn. A similar kind of shrinkage was reported in the case of caustic mercerised cotton yarns (Goldthwait, 1965). In the case of caustic mercerised yarn, the shrinkage occurred due to the swelling of the fibres in the yarn.

### Linear density of yarn

The linear density of the treated yarns was calculated following ASTM D1–77 – 07 method and summarised in Table 2. The number of specimens measured for each sample was 20.

The linear density of the yarn changed after the treatments. The overall trend shows that the density has increased after treatment irrespective of the pH. The increased linear density may be due to the weight gained by the yarn as a result of glycine treatment as well as shrinkage. The general trend other than at pH4 suggests that



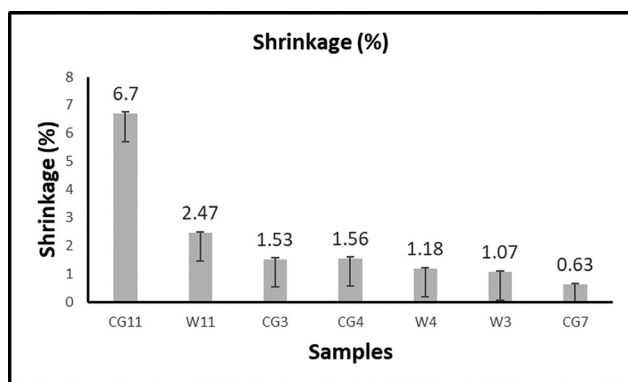
**Figure 2.** SEM micrographs of cotton yarn cross sections; (a) UTC, (b) SC, (c) W3, (d) W11, (e) CG3, (f) CG7 and (g) CG11.

glycine treated yarns had higher linear density compared to control samples at the same pH.

#### **Tensile properties of the yarn**

The treated yarns were tested for tensile properties. The distribution curves of stress values of a single yarn are shown in Figure 4. Glycine treatment reduced the specific stress

values to some extent but protected the samples from the significant drop in tensile strength at acidic pH (pH 3,4). In the SEM micrographs, it can be seen that the packing of the fibres in the yarn was changed after treatments. Therefore, the above-described decrease in stress may be due to this change in packing and deformation of the structure during the treatment in addition to an increase in linear density, which caused a drop in normalised strength.



**Figure 3.** Shrinkage percentage of treated samples. Error bars indicate 95% confidence intervals for shrinkage means.

**Table 2.** Linear density of yarns and t-test statistics for the difference between UTC and other glycine/pH treatments.

Samples	Linear density (TEX) $\pm$ SD	t Value	p Value
UTC	11.0 $\pm$ 0.28	–	–
SC	10.7 $\pm$ 0.88	2.05	.014
CG11	13.0 $\pm$ 0.26	2.03	.003
W11	12.3 $\pm$ 0.24	2.02	.002
CG3	12.3 $\pm$ 0.27	2.02	.002
W3	11.5 $\pm$ 0.30	2.01	.004
CG4	12.6 $\pm$ 0.26	2.05	.003
W4	12.8 $\pm$ 0.24	2.07	.005

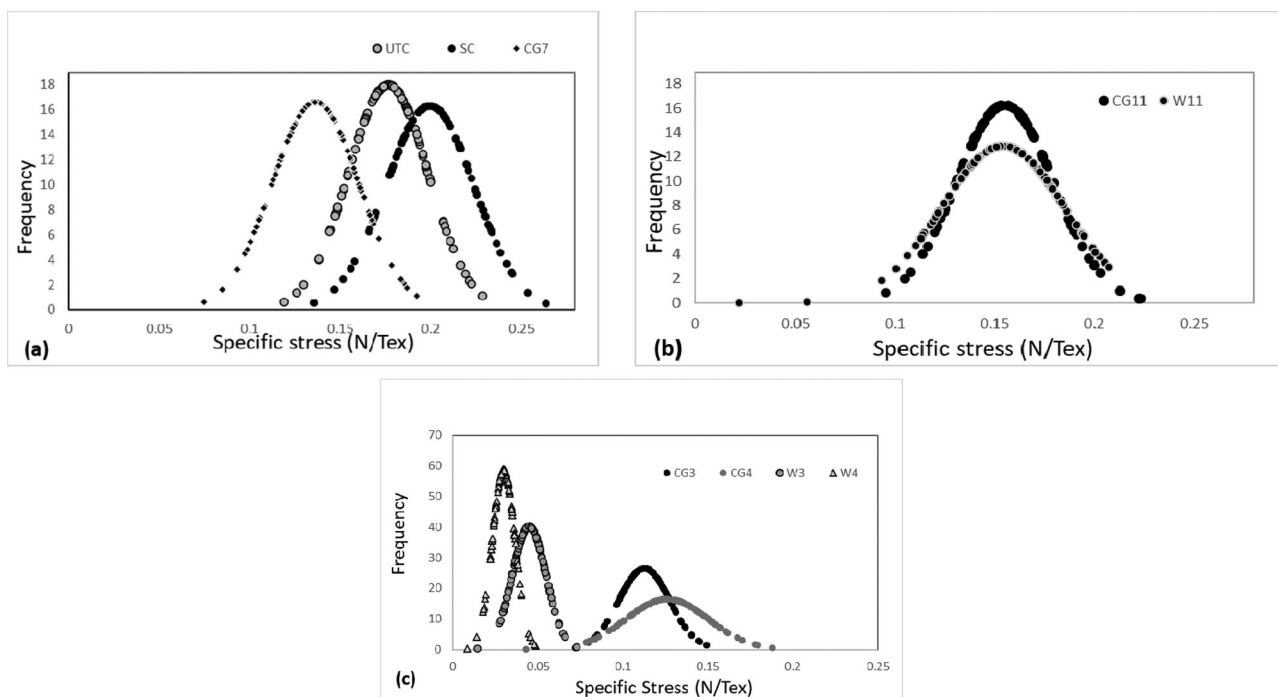
The distribution curves of the strain values of yarn are shown in Figure 5. Cotton yarn treated with glycine showed a substantial change in strain. In particular, breaking strain increased significantly in CG 11. The influence of glycine is clear when CG 11 is compared with buffer only treatment, i.e. W11. These results are comparable with fibre tensile test results where improvement in breaking strain was evident, particularly in single fibre tests. Remarkably higher breaking

strain of CG3 and CG4 compared to W3 and W4 demonstrate the positive effect of glycine on yarn tensile properties.

The average values of the load, the elongation at break and specific stress (N/tex) obtained from single yarn tensile tests (specimen size of 100 per sample) are listed in Table 3.

The tensile tests revealed that after treating with aqueous glycine solution at pH11 the breaking strain of the scoured cotton increased by 70%. In an identical case, the glycine treatment at acidic conditions also changed the strain value, especially at pH4, where the strain increase was 38%.

It was reported that under acidic conditions, at pH 3, the  $-\text{COO}-$  part of the glycine zwitterion picks up a hydrogen ion (Xu et al., 2003) and in alkaline pH, the hydrogen ion is removed from the  $-\text{NH}_3^+$  part of the zwitterion. Thus, an esterification reaction between glycine and cotton cellulose structure occurs at pH 3. However, at pH 11 an amino complex is formed between glycine and cotton cellulose (Remadevi et al., 2018). The increase in strain at pH 11 could be due to the formation of an amino complex wherein the amino complex formation increases the amorphous content in the cotton material (Remadevi et al., 2018). Furthermore, it was reported earlier (Remadevi et al., 2018), that the crystallinity of cotton didn't change with the glycine treatment. This indicates that the glycine interactions didn't disrupt the crystalline regions of the cotton cellulose, suggesting that this interaction mainly occurred to the surface, amorphous regions, which in turn provides a greater degree of extension (strain) before the break. Overall, the tensile properties of the glycine treated yarn were improved and thus it can be used as a green pre-treatment for yarn.



**Figure 4.** Distribution of yarn specific stress (N/tex) values for (a) UTC, SC and CG7, (b) W11 and CG11 (c) CG3, CG4, W3 and W4.

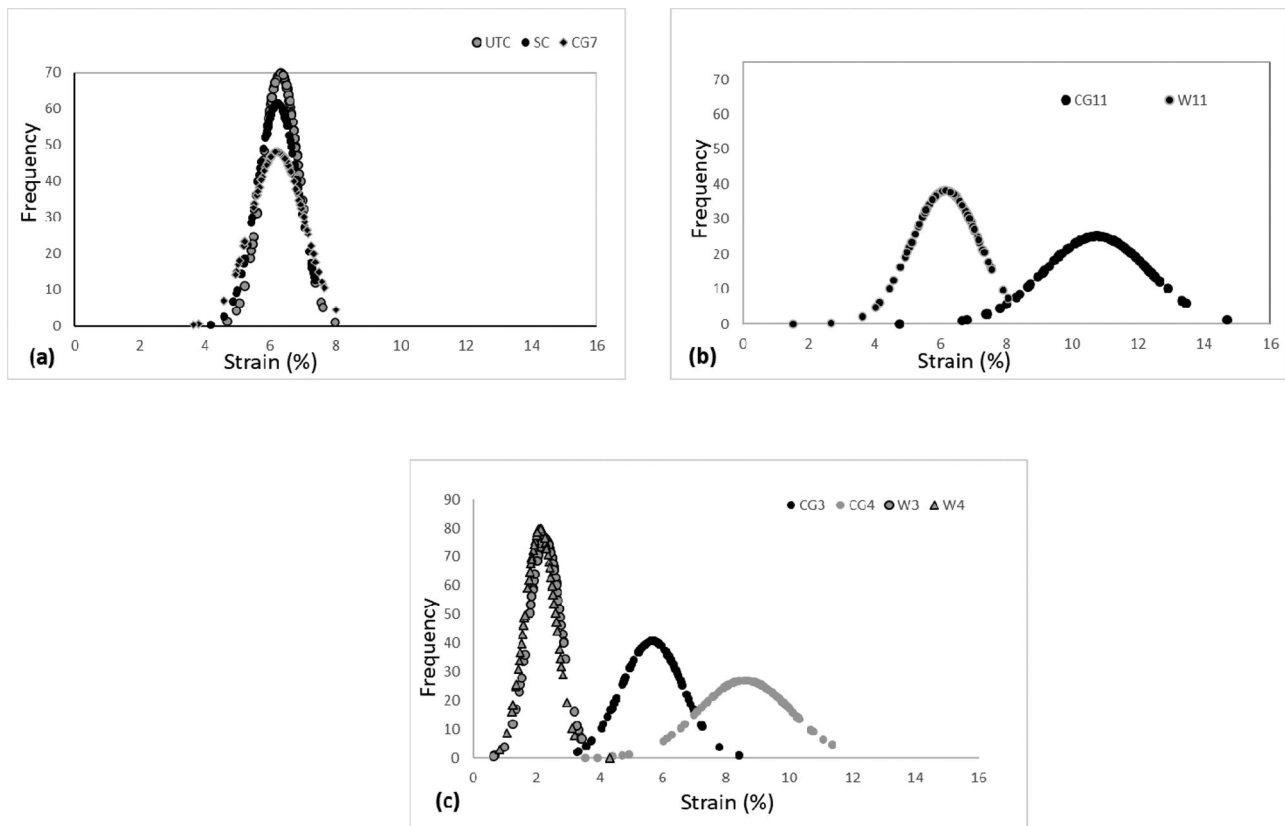


Figure 5. Distribution of yarn strain (%) values for (a) UTC, SC and CG7, (b) W11 and CG11 (c) CG3, CG4, W3 and W4.

Table 3. Single yarn tensile test results ( $n=100$  single yarn in each sample) and t-test statistics for the difference between UTC and other glycine/pH treatments.

Samples	Load (N)	Strain (%)	Specific stress (N/tex)	t Value	p Value
	Avg. $\pm$ SD	Avg. $\pm$ SD	Avg. $\pm$ SD		
UTC	1.94 $\pm$ 0.24	6.32 $\pm$ 0.57	0.18 $\pm$ 0.02	–	–
SC	2.15 $\pm$ 0.26	6.23 $\pm$ 0.65	0.19 $\pm$ 0.02	Load.1.97 Elon.1.97	2.26E – 08 .32
CG3	1.39 $\pm$ 0.18	5.67 $\pm$ 0.97	0.11 $\pm$ 0.02	Load. 1.97 Elon.1.97	3.59E – 44 4.59E – 08
W3	0.52 $\pm$ 0.11	2.27 $\pm$ 0.52	0.04 $\pm$ 0.01	Load. 1.97 Elon.1.97	7.25E – 119 7.91E – 118
CG4	1.59 $\pm$ 0.31	8.59 $\pm$ 1.47	0.13 $\pm$ 0.02	Load.1.97 Elon.1.97	1.13E – 16 9.76E – 33
W4	0.38 $\pm$ 0.09	2.11 $\pm$ 0.50	0.03 $\pm$ 0.01	Load.1.97 Elon.1.97	2.91E – 129 1.99E – 122
CG7	1.80 $\pm$ 0.31	6.22 $\pm$ 0.83	0.14 $\pm$ 0.02	Load. 1.97 Elon.1.97	.001 .34
CG11	2.02 $\pm$ 0.32	10.74 $\pm$ 1.58	0.16 $\pm$ 0.02	Load.1.97 Elon.1.97	.069 2.40E – 66
W11	1.89 $\pm$ 0.38	6.16 $\pm$ 1.04	0.15 $\pm$ 0.03	Load. 1.97 Elon.1.97	.31 .18

## Conclusion

In this work, the effect of aqueous glycine treatment on the physical and mechanical properties of cotton yarn was studied. The CG11 sample's scanning electron microscopic images showed morphological changes. The swelling was seen even in fibres in the yarn core. However, in CG3, fibres only in the outer part of the yarn cross-sections swelled. From the shrinkage measurement, it was found that glycine treatment at pH11 yarn samples had significant shrinkage compared to other yarn samples. This is supporting the electron microscopic results. The linear density

results showed that the linear density of the yarn changed after the treatments. The overall trend showed that the density has increased after treatment irrespective of the pH. The increased linear density could be due to the weight gained by the yarn because of glycine treatment as well as shrinkage. The general trend other than at pH4 suggests that glycine treated yarns had a higher linear density compared to control samples at the same pH. From the yarn tensile tests, it was found that yarn tenacity decreased after the glycine treatment but at pH 3 and 4, glycine treatment produced stronger yarn compared to buffer only treated yarns. The most interesting results from yarn tensile tests

are the significant increase in breaking strain of CG11 yarns, which recorded a 70% increase compared to untreated yarn. In the case of CG4, the breaking elongation increased by 38%. Glycine treatment caused yarn shrinkage particularly evident in CG11. Considering the non-hazardous, environmentally friendly nature of glycine (Bose et al., 2012) these findings will help the development of nonhazardous amino acid-based pre-treatment for cotton yarn to improve the appearance with enhanced mechanical properties.

## Disclosure statement

No potential conflict of interest was reported by the author(s).

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