



## Research article

## Effect of neutralizers and silicone softeners on phenolic yellowing phenomenon of OBA treated cotton knitted fabric

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## ABSTRACT

Phenolic yellowing is a complex physicochemical phenomenon of cotton, especially for white and pastel-colored fabrics. This study tried to analyze the effect of neutralizers and silicone softeners on this. Three types of neutralizers of different chemical characteristics (acetic acid, citric acid, and commercial complex acid) were used to achieve core neutralization whereas two unlike ionic nature of silicone softeners (cationic & non-ionic) for the finishing process used. The acetic acid and nonionic softener treated fabric gives the maximum CIE (International Commission on Illumination) whiteness index value of 151 while citric acid and cationic softener have the least rating of 140.93 with a similar chemical dosage of 1.5 g/l by immediate testing. However, after repeated testing for one-month conditioning in atmospheric conditions, complex acid, and nonionic softener treated samples exhibited the maximum whiteness index of 145.86; on the other hand, citric acid and cationic softener treated one had the least rating of 125.85. Moreover, fabric core pH<sub>i</sub> reading was found to be 6.68, 6.15 & 5.13 for acetic acid, citric acid, and complex acid through immediate testing. After one month of conditioning, the values were 7.2, 6.61, and 5.25, respectively. Finally, a maximum phenolic yellowing rating of 4–5 was found with complex acid & nonionic softener in contrast poor rating of 2 with acetic acid & cationic softener for immediate testing. Storage of the samples has a significant impact on phenolic yellowing for all types of chemical concentrations. Lightfastness rating was found identical for all samples while bursting strength had a very negligible impact, Fourier transform infrared (FT-IR) spectroscopy also revealed the presence of noticeable chemical functional groups.

## 1. Introduction

Clothing is the second basic need of humans. Commercially, different types of textile products such as domestic textiles, technical textiles, smart textiles, and functional textiles are available in the market [1, 2]. These products are categorized by fibrous and nonfibrous materials. In fibrous materials, it is divided into natural and manmade fibers. Natural fibers are known as cotton, jute, silk, hemp, ramie, and so on [3, 4]. Among all fibers, cotton is used to fulfill half of the clothing demand around the globe. Cotton cellulose is composed of D-anhydroglucopyranose units, also known as anhydroglucose, joined together by  $\beta$ -1, 4-glycosidic bonds. The repeating unit is generally 5000 [5,6]. It is widely used due to its excellent wearability, superior wearing comfort,

excellent moisture permeability, and outstanding breathability [7]. However, there have been some major drawbacks to the cotton fabric. Phenolic yellowing is one of them [8, 9].

Phenolic yellowing of textiles is the outcome of the phenolic compound present in the surrounding atmosphere and the reaction triggered by suitable pH conditions. These dependent variables are collectively responsible for phenolic yellowing. Butyl-hydroxytoluene (BHT) is the main phenolic compound that can react with the nitrogen oxides (NO<sub>x</sub>) in the air and forms nitrobenzene or quinone, which is a yellow chemical substance in a slightly alkaline medium [10, 11]. None of the nitrous oxides or BHT alone causes yellowing in contrast when they conjugate yellowing product forms. Optically brightened articles or pale shades that are processed with inappropriate core neutralizers always have a

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propensity to cause yellowing. Slightly acidic core pH of the finished goods must have to ensure to avoid this problem. To prevent the aging of olefin, BHT is often added to the production of polyethylene and polypropylene bags. Besides, the impregnation of BHT as an antioxidant can increase the shelf life of packaging bags and avoid air oxidation. BHT is good for packaging material when reacts with textile material causes yellowing. So, textile materials are often advised to not be placed in poly bags which have antioxidants like BHT. BHT is a volatile compound having low vapor pressure thus can be easily transferred to textile goods when comes in contact [12, 13]. The interaction produces a compound named 2, 6-di-tert-butyl-4-nitrophenol (DTNP), which is the main source of phenolic yellowing [14].

During optical brightening treatment (OBA) on cotton fabric, there are possible causes that create phenolic yellowing problems. In some cases, excess OBA is utilized due to oversight or wrong computation. Increasing the centralization of OBA past an immersion limit prompts dulling and yellowing [15]. The presence of metal particles, especially zinc can prompt the dulling of OBA for cotton. Zinc concentration could be expected to unreacted zinc in zinc formaldehyde sulfoxylate in a reductive fade, or zinc salt creates in phenolic yellowing up [16, 17]. Excess drying temperature can likely lead to yellowing. While cotton is dried and completed at 120–140 °C, and polyester and polyester mixes at 170–190 °C, excess temperature and additional contact time at raised temperature can decrease whiteness [18].  $p^H$  can create a problem during OBA treatment. This generally occurs with high-liking OBAs for cotton that are not stable in less than  $p^H$  5.0. Along with these lines in the ensuing balance after OBA treatment and/or during finishing, if pH is highly acidic, the whiteness index is reduce [19, 20].

To overcome this problem, several researchers have performed on it. Certain natural conditioners and silicone conditioners tend to prompt yellowing to the subsequent finishing process. Some of the softeners for whiteness that are non-yellowing such as Gamasoft-KA, Sarasoft-SNY, Sarasoft-1367, Sarasoft-GA, Sarasoft-UK, or Supercone-2100, are suggested particularly for complete whiteness on the fabric [9, 21]. Only softeners can not demonstrate the remarkable properties of anti-yellowing. The treatment with aryl alkyl sulfonic acid salts during cotton fabric finishing shows anti-yellowing properties. Here, to overcome the major drawbacks, it requires additional processing steps, high consumption of water, energy, and treatment time resulting in increased treatment cost. In addition, these chemicals are also not environmentally friendly [22, 23]. Some of the electrospun nanofiber webs have been developed which provide brilliant whiteness surfaces. It shows a higher CIE whiteness index. However, electrospun nanofibers are not possible in every application of cotton fabric [24].

Several types of acid and softeners are used frequently in the textile dyeing industry. This study was mainly conducted to analyze the effect of treatment with neutralizers and silicone softeners. Here, three types of neutralizers (acetic acid, citric acid, and commercial complex acid) and two types of ionic softener (cationic and non-ionic) in different concentrations were used to make the recipe for the finishing process. The fact behind the selection of these three different types of acid lies in their chemical phenomena. First of all, acetic acid is a weak organic acid. In addition, this is a volatile acid. So, neutralization of acetic acid can not give a stable performance, meaning that the pH of the treated fabric becomes on the higher side. Secondly, citric acid is a non-volatile organic acid and comparatively stronger than acetic acid because of its chemical formula. Due to these reasons, citric acid can give better neutralization than acetic acid. In contrast to these two organic acids, the commercial complex acid is a non-volatile inorganic acid. Since it is non-volatile, it does not evaporate while the fabric becomes dry at an elevated temperature, hence a stable core neutralization performance can be found. Then the treated samples are subjected to measure the CIE (International Commission on Illumination) whiteness index value. After one month of conditioning, the whiteness index is again tested. Among all the samples, the best result comes from complex acid & nonionic softener-treated fabrics. This result also indicates a sustainable approach for phenolic

yellowing treatment. Therefore, it can be a potential candidate for anti-yellowing of cotton fabric in the textile industry.

## 2. Experimental details

### 2.1. Materials

Grey cotton (100%) single jersey knitted fabric (160 g/m<sup>2</sup>) having 25 wales/inch and 40 courses/inch were obtained from SQUARE Textile Mills Ltd, Gazipur, Bangladesh. Commercial nonionic silicone softener (alkyl alcohol polyglycolether) and complex acid (sulfuric acid, mono (2-Ethylhexyl) ester, and sodium salt) obtained from Zschimmer & Schwarz, Germany. Moreover, amino-functional cationic silicone (alkyl polyoxyethylene ether) was collected from Xorette specialties, Singapore. Besides, laboratory-grade sodium hydroxide, hydrogen peroxide (50%), acetic acid, and citric acid were obtained from Merck KGaA, Darmstadt, Germany. Commercial sequestering agent, peroxide stabilizer, and detergent sourced from Eurodye CtC, Belgium. A stilbene derivative optical brightening agent (OBA) was collected from Rifa industrial co., ltd (South Korea).

### 2.2. Instrumentation

A 300ml bath capacity, suitable for the 10 gm of cotton fabric processing in the laboratory dyeing machine (Ahiba infrared dyeing machine, UK) used for bleaching, neutralizing, and softening. Similarly, an oven (James heal, UK) was used with a maximum heating capacity of 100 °C for drying. Phenolic yellowing testing apparatus named perspirometer also from James heal, UK. Digital pH meter for pH measurement from Mettler Toledo, USA. All the phenolic yellowing testing consumables such as Impregnated test paper, Butyl hydroxytoluene (BHT) free polyethylene, Polyamide control fabric collected from the society of dyers and colorists (SDC), UK. Bursting strength of the treated fabrics tested according to the ISO 13938:2 1999 using Truburst 4 (James heal, UK). Lightfastness testing was carried out with the help of xenon arc chamber B02 (Q-Sun, USA) following ISO 105 B02:2014. FT-IR spectra of the samples were taken using Bruker advance (Bruker, Germany) spectrometer.

### 2.3. Methodology

Ahiba infrared exhaust machine (SDL, UK) was used in this study for all chemical processing. A 300 ml bath capacity pot is prepared for the scouring-bleaching. To make such happening 10 gm grey fabric was cut and weighed correctly using the digital balance (Ohaus, USA) having a measurement precision capacity of 0.001 gm. The M: L (material: liquor) ratio at 1:10 was maintained throughout the experiment. Sodium hydroxide (NaOH)-3 g/l, hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>)- 6 g/l, sequestering agent- 1 g/l, detergent- 1 g/l, peroxide stabilizer- 1 g/l, and 1% optical brightening agent (OBA) added to the scouring-bleaching bath. Stock solution prepared for all chemicals for the precision of measurement. The required amount of chemical solution was taken using a digital pipette (Rainin, USA). Scouring-bleaching was carried out for 60 min at 100 °C with the machine at 40 rpm. Once the bleaching is completed the samples are washed with hot water for 10 min at 80 °C to remove the natural oil, wax, and impurities that come out from the grey cotton. Thorough rinsing is followed by hot washing to clean the extracted impurities. The neutralization conditions were optimized using different concentrations (0.5 g/l, 1 g/l, and 1.5 g/l) of the acids (acetic acid, citric acid, and complex acid) at 50 °C for 20 min. Once the neutralization was completed, different concentrations (1 g/l, 2 g/l, and 3 g/l) of both silicone softeners (nonionic and cationic) were applied separately at ambient temperature for another 20 min. Finally, the samples were squeezed with a laboratory padder (Mathis, Switzerland) and dried in an oven (James heal, UK) at 100 °C for 20 min. The process sequence is represented in Figure 1.



Figure 1. Process Sequence of Phenolic Yellowing treatment.

### 3. Result and discussion

#### 3.1. Mechanism of phenolic yellowing

Phenolic yellowing is a complex phenomenon and time-consuming physicochemical process. This chemical reaction is a conjugation process of oxidation and nitration. Quinone and nitrophenol are the outcomes of such chemical mechanisms in the presence of nitrous oxide. 2, 6 di-tert-4-nitrophenol is formed from 2, 6 di-tert butyl p-cresol in the presence of NOX which is a nitration reaction. Quinone forms in the presence of NOX. This is a visible yellow compound. Moreover, these nitrophenols and quinones are colorless in an acidic medium. By contrast,

these become bright yellow in an alkaline medium. The mechanism of phenolic yellowing is represented in Figure 2.

#### 3.2. FT-IR analysis

The presence of chemical compounds on the surface of treated cotton fabric is investigated by using Fourier Transform Infrared spectroscopy. The analysis is represented in Figure 3. Where Figure 3a is the non-treated cotton fabric and Figure 3 (b-g) is the treated cotton fabric using different types and amounts of softener and acid. The characteristic peaks of cotton fabric due to cellulose structure were observed at  $3330\text{ cm}^{-1}$  for O–H stretching,  $2900\text{ cm}^{-1}$  for C–H stretching,  $1430\text{ cm}^{-1}$  for C–H wagging,  $1314\text{ cm}^{-1}$  for C–H bending, and  $1026\text{ cm}^{-1}$  for C–O stretching. When the cotton fabric is treated with acid and softener, then the peak intensity is slightly decreased. In some cases, the peaks appear in the same band with different wavelengths because the same stretching and bending are possible for useable acid and softener. For complex acid, there is an interaction between acid and cellulosic cotton which can form sulfoxides. The peaks at  $1050\text{ cm}^{-1}$  for S=O stretching give the evidence in Figure 3 (b-c). In addition, the peak at  $1010\text{ cm}^{-1}$  for C–O stretching ensures the interactions of citric acid on cotton fabric Figure 3 (d-e). Moreover, the peaks at  $3276\text{ cm}^{-1}$  for O–H stretching and  $1025\text{ cm}^{-1}$  for C–O stretching also provide the interactions of acetic acid on cotton fabric Figure 3 (f-g). Therefore, the overall analysis of FTIR spectra indicates that cotton fabrics are made interactions with the chemicals.

#### 3.3. CIE whiteness index

The CIE whiteness index is determined with the help of a reflectance spectrophotometer (Datacolor 650, USA). The instrument was calibrated accordingly with the setup of  $10^\circ$  viewing angle, a specular component included and larger aperture size. The fabric CIE whiteness index analysis is represented in Figure 4. Nonionic softener treated samples show a better whiteness index for all three types of acid. In contrast, cationic softener shows a lower whiteness index rating for all cases. Positive amino group nuclei of cationic softener negatively affect the whiteness index because of yellowing caused by the same. The positive charge reacts with the cellulose in an aqueous medium and causes yellowing in the presence of a suitable condition. Since the nonionic softener does not pose any active charge, therefore it does not react with the cellulose to affect the whiteness index. One more reason behind the lower whiteness rating by cationic softener may be the lower  $p^H$  (4.7) value of the product as compared to the nonionic softener ( $p^H = 6.1$ ). Major OBAs are not stable in acidic  $p^H$ , thus appearing yellow to greener because of reflectance value negatively impacted by the acidic  $p^H$ , showing a lower

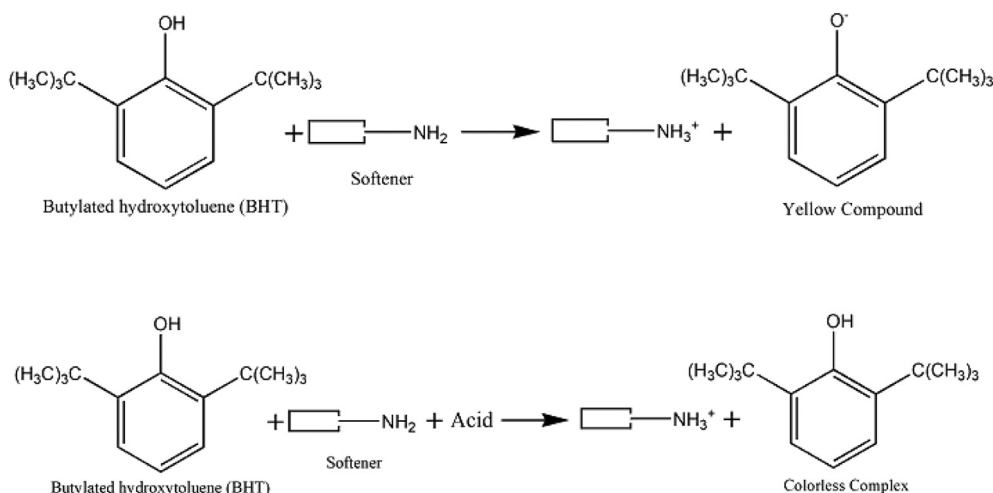
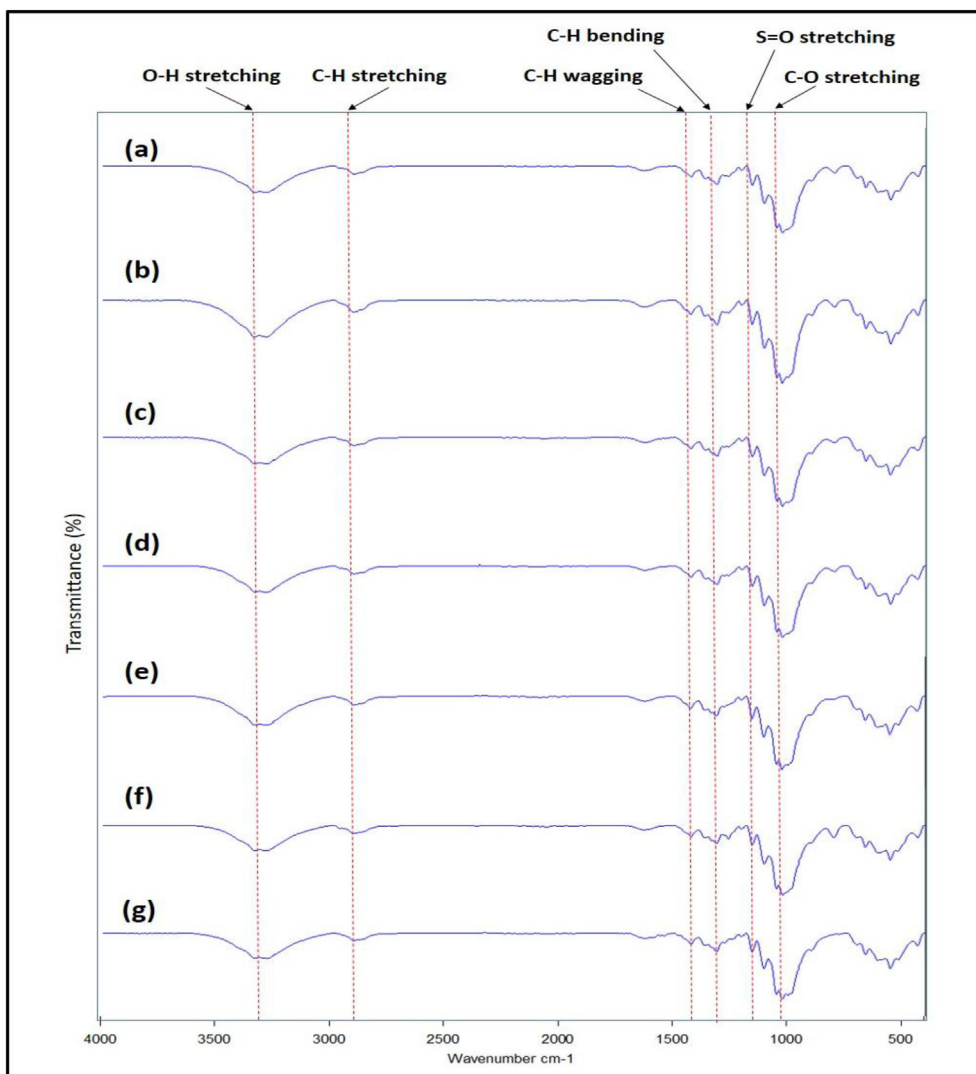


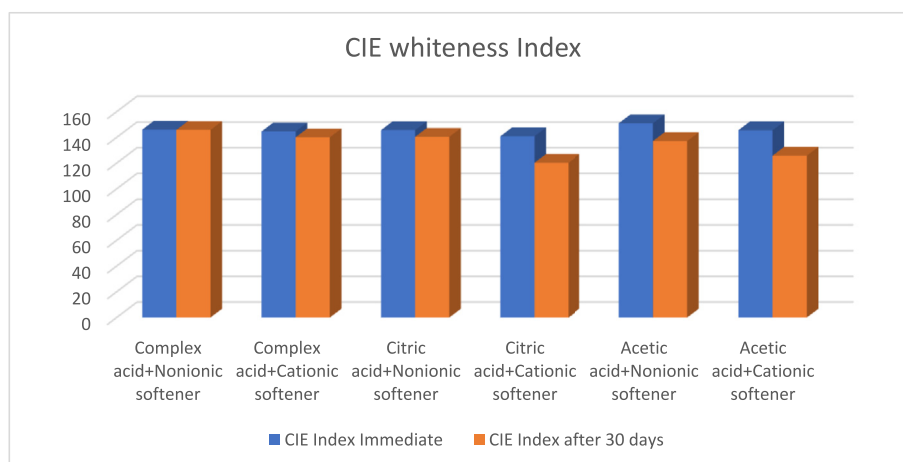
Figure 2. Mechanism of phenolic yellowing.



**Figure 3.** FTIR spectra (a) Non-treated cotton fabric, treated cotton using (b) 1.5 g/l complex acid & 3 g/l nonionic softener, (c) 1.5 g/l complex acid & 3 g/l cationic softener (d) 1.5 g/l citric acid & 3 g/l nonionic softener (e) 1.5 g/l citric acid & 3 g/l cationic softener (f) 1.5 g/l acetic acid & 3 g/l non-ionic softener, (g) 1.5 g/l acetic acid & 3 g/l cationic softener.

whiteness index. The samples were again tested after one-month conditioning in ambient conditions to determine the impact of the atmospheric condition on the whiteness index. Test results depict that nonionic

softener and complex acid have outstanding whiteness indexes of 146.05 and 145.86, respectively for immediate testing and after one month of conditioning. Besides the cationic softener with complex acid has some



**Figure 4.** Fabric CIE whiteness Index analysis with 1.5 g/l Acid & 3 g/l Softener treatment.

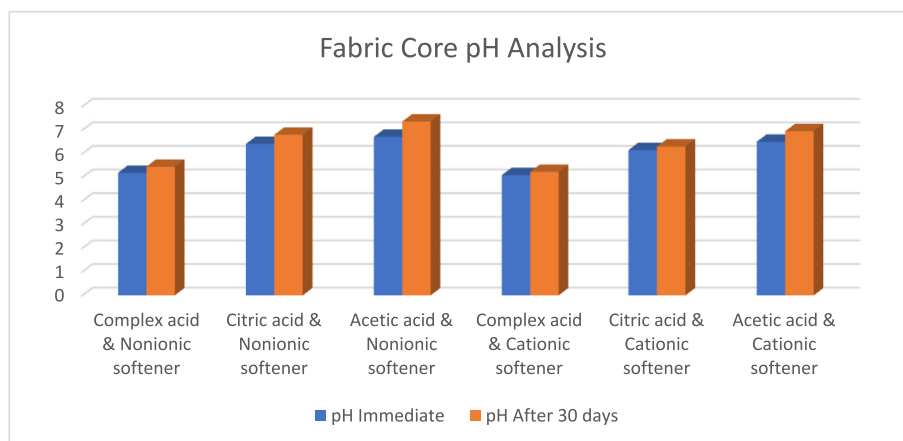


Figure 5. Fabric core pH analysis with 1.5 g/l Acid & 3 g/l Softener treatment.

influence on the whiteness index the reading is 144.67 and 140.1 for immediate and after conditioning. Similarly, citric acid and nonionic softener show a similar whiteness index of 145.69 and 140.5, respectively. While citric acid with the cationic softener the value was found to be 140.93 and 120.3, respectively. Moreover, acetic acid and nonionic softener give a whiteness index of 151 and 137.16, respectively. While acetic acid with cationic softener gives 145.61 and 125.85 whiteness index, respectively. The reason behind the lower CIE whiteness indexes after 30 days for all samples is due to the phenolic yellowing phenomenon. The oxides of nitrogen react with the fabric sample in a complex way to undergo yellowing. This complex chemical compound is yellow on a slightly higher  $p^H$  medium. Under the atmospheric condition, pH slightly increases in the presence of moisture which accelerates the reaction. Since the yellow compound forms CIE whiteness rating which also goes down.

### 3.4. pH analysis

Fabric core  $p^H$  was analyzed with the standard AATCC 81 (2012) test method. The samples were cut into small pieces according to the method and boiled at 80 °C in distilled water to have an aqueous extraction for the  $p^H$  measurement. The results are shown in Figure 5. Acetic acid has the least neutralization performance means more  $p^H$  reading as compared to all three dosages. In contrast, the complex acid has the lowest pH reading in all concentrations which is the outcome of better neutralization. Similarly, citric acid also shows a good neutralization behavior. The

core  $p^H$  of the fabrics with 1.5 g/l acid treatment was found to be 6.68, 6.15, and 5.13, respectively for acetic acid, citric acid, and complex acid. The samples were stored for one month in atmospheric conditions to analyze the  $p^H$  variation effect. Repeated testing after one month depicted a slightly increased pH reading for all samples meaning that atmospheric conditions had some impact on the sample which was mainly caused by humidity. Under the atmospheric conditions, the presence of humidity caused slightly increased  $p^H$ . This happens due to the presence of residual alkali in the fabric reacting with the acids in the presence of moisture and produces sodium salt of that respective acid which is the main reason for the increased  $p^H$ . The acetic acid reacts with caustic soda produces sodium acetate, similarly citric acid produces sodium citrates, and commercial acid produces sodium sulfate. Since all these three salts have slightly alkaline  $p^H$ , hence the fabric pH increases accordingly. After conditioning of the sample, complex acid-treated fabric shows a  $p^H$  value of 5.25, citric acid shows a pH value of 6.61, and acetic acid shows a pH value of 7.20. Analyzed data depicts that acetic acid-treated samples have a slightly increased  $p^H$  phenomenon while complex acid has the least.

### 3.5. Phenolic yellowing test

Assessment of phenolic yellowing of the treated samples were carried out according to the standard method of ISO 105- X18 (2007). According to the testing method, a maximum of six samples can test once at a time. Due to the number of samples, several tests were needed

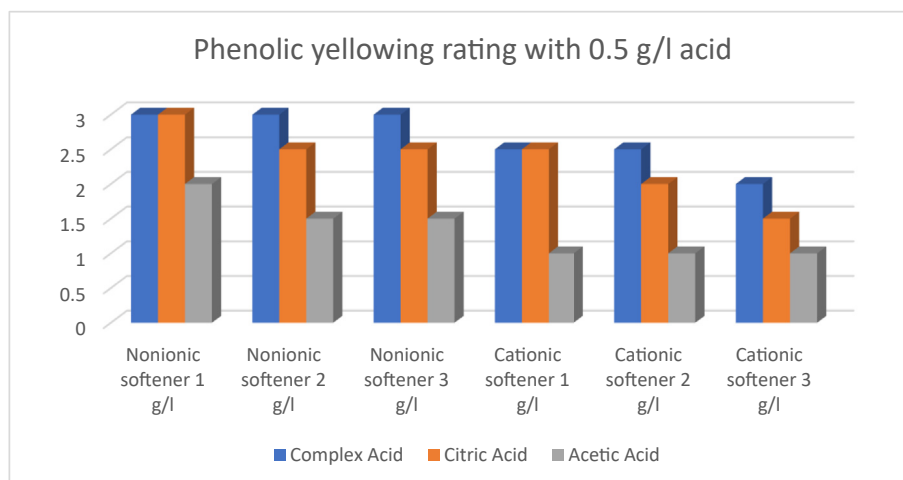


Figure 6. Phenolic yellowing analysis with 0.5 g/l Acid treatment.

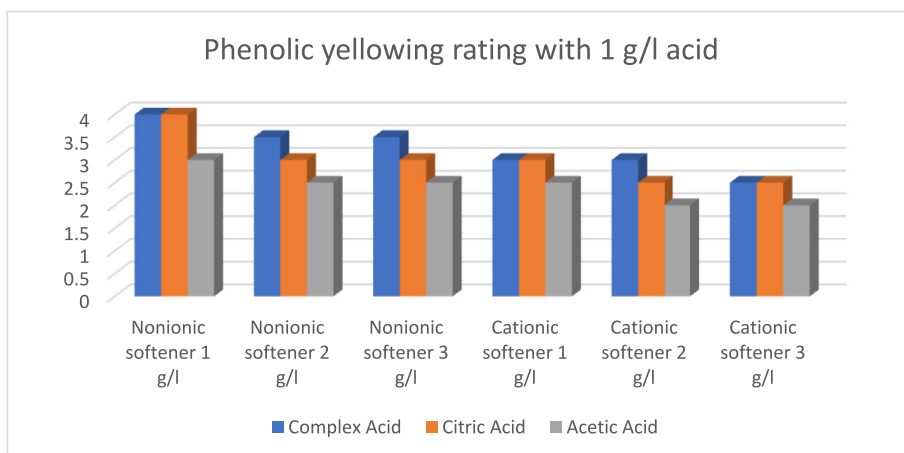


Figure 7. Phenolic yellowing analysis with 1.0 g/l Acid treatment.

for complete testing. Standard testing conditions of 50 °C for 16 h were maintained accordingly. Once the testing was completed, the samples were assessed using the greyscale (James heal, UK), and specific ratings had done separately. All the test results are represented in Figure 6, Figure 7, Figure 8, and Figure 9. Nonionic softener & complex acid gives the best (4–5) rating in all concentration of acid. While acetic acid and cationic softener have the worst rating of 2 for even maximum dosage of acid and minimum dosage of softener. However, citric acid performs a good rating (4–5) with nonionic softener and moderate for cationic softener in all concentrations. One common phenomenon

found for all samples that better phenolic yellowing rating with the increased acid concentration was the outcome of proper core neutralization. Repeated testing after conditioning for one month gave a lower rating in all cases.

Acetic acid is a volatile substance and comparatively weak organic acid. In contrast, citric acid is not a volatile one like the commercial complex acid. The volatility of acetic acid leaves the fabric surface in insufficient neutralization compared to the citric and complex acid. Besides, the cationic softener would create a yellow complex reacting group with the BHT which be visible after testing. p<sup>H</sup> act as a catalyst for any

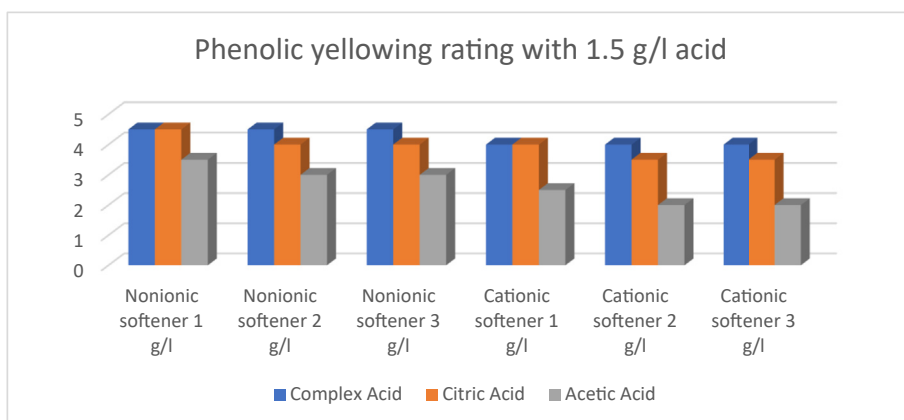


Figure 8. Phenolic yellowing analysis with 1.5 g/l Acid treatment.

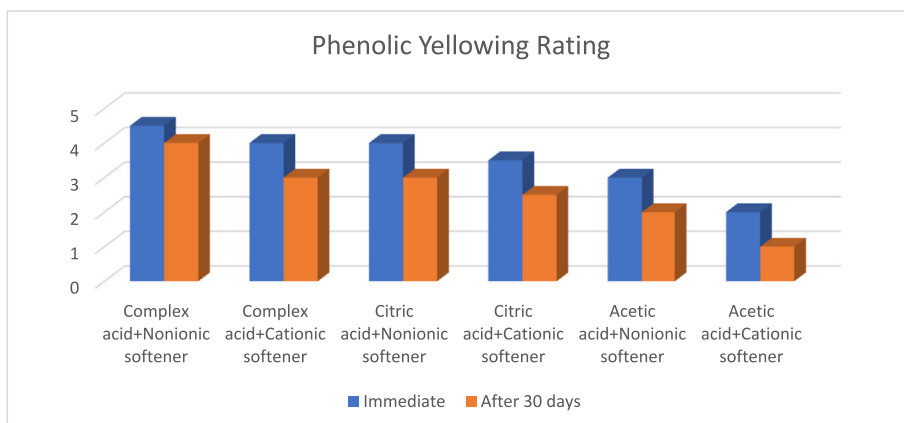


Figure 9. Phenolic yellowing analysis with 1.5 g/l Acid & 3 g/l Softener treatment.

**Table 1.** Bursting strength test.

Sample types	Bursting strength (kPa)
Complex acid & cationic softener	360.5
Citric acid & cationic softener	363.8
Acetic acid & cationic softener	365.9
Complex acid & nonionic softener	360.1
Citric acid & nonionic softener	363.4
Acetic acid & nonionic softener	365.2

**Table 2.** Lightfastness test.

Sample types	Lightfastness rating
Complex acid & cationic softener	3–4
Citric acid & cationic softener	3–4
Acetic acid & cationic softener	3–4
Complex acid & nonionic softener	3–4
Citric acid & nonionic softener	3–4
Acetic acid & nonionic softener	3–4

chemical reaction, here also higher pH accelerated the yellowing phenomenon. On the other hand, the nonionic softener does not have an active positive site, so it can not create a yellow complex by reacting with the BHT. Since the complex acid is an inorganic sulfuric acid complex that gives the best core neutralization and least yellowing, the phenolic yellowing testing was repeated once after the conditioning time was completed. Test results showed that all the samples had a slightly lower rating compared to the immediate one. Such phenomenon found severe for cationic softener in contrast to nonionic.

### 3.6. Bursting strength test

Bursting strength is a tensile testing phenomenon to test the capability of fabric to withstand rupture against applied pneumatic pressure. Under the subjected pressure load, the fabric begins to expand in all directions when the limit exceeds getting to be burst. Bursting strength tested for 1.5 g/l acid and 3 g/l for softener treated samples. In Table 1, the results depict that all the three acids have almost similar readings means the neutralization and finishing process does not affect the tensile strength phenomenon. All the tests were repeated five times and the average is taken as the result is calculated automatically by the machine.

### 3.7. Lightfastness test

The unsaturated structure of OBA absorbs ultraviolet and violet radiation at 340–370 nm and it re-emits the absorbed light (shorter wavelength) to visible blue light (longer wavelength) at 420–470 nm. This unsaturation is very sensitive to sunlight and losses its ability to work and leads to yellowing. Lightfastness is the photo-chemical process of any colorant to resist fading under specific light exposure. The samples were exposed for a specified time according to the standard method and results were assessed with the help of greyscale. In Table 2, a similar lightness rating was found for all three types of acid treatment (1.5 g/l). So, it can be concluded that neutralization & softening does not adversely impact the lightfastness rating of the samples.

## 4. Conclusion

Processing chemicals and finishing auxiliaries play a vital role in the phenolic yellowing phenomenon. In addition, atmospheric pollutant also has a major influence on such types of yellowing. Such types of yellowing are the synergistic outcome of these factors. Similarly, core pH and CIE whiteness index were also influenced by these variables. With the

increase of acid dosing, core pH consistently goes down. Non-volatile inorganic complex acid gives the best core neutralization while volatile organic acetic acid performs the least. Cationic softener greatly impacts the CIE whiteness index and phenolic yellowing for both cases of testing in contrast nonionic softener performs the best rating. FT-IR spectrum reveals the characteristic chemical functional groups for all sample types. Lightfastness rating & bursting strength are not greatly affected by these chemical treatments. Finally, we found that nonionic softener & commercial complex acid has good performance considering the variables.

## Declarations

### Author contribution statement

Md. Salauddin Sk: Conceived and designed the experiments; Performed the experiments; Wrote the paper and address reviewer comments.

Rony Mia: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

Bulbul Ahmed: Analyzed and interpreted the data; Wrote the paper.

Atiqur Rahman: Performed the experiments; Analyzed and interpreted the data.

Md. Mostafizur Rahman Palash: Performed the experiments.

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### Data availability statement

Data included in article/supp. material/referenced in article.

### Declaration of interests statement

The authors declare no conflict of interest.

### Additional information

No additional information is available for this paper.

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