



The investigation of the effect of sodium chlorite and phosphonic acid catalysts on cotton bleaching process conditions

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Abstract

Traditional textile bleaching techniques need to be given another look in light of the environment and lifestyle of today. To achieve higher whiteness values while using less water and chemicals during the bleaching process, it is crucial for both the environment and the economy. The most effective disinfectant, chlorine dioxide (ClO_2), is produced by sodium chlorite (NaClO_2), which is a suitable oxidant for the job. During the COVID-19 pandemic, NaClO_2 gained popularity and became more widely available. The use of NaClO_2 as a bleaching agent offers many benefits, including a decrease in the number of washing steps and an increase in cotton strength. This reagent's ability to produce less weight loss in the fabric than other reagents is another benefit. Therefore, the present work was intended to improve the process conditions (different temperatures, concentrations, and times) of bleaching of cotton fabric by using NaClO_2 . A high whiteness index (W.I. = 88) was obtained by utilizing phosphonic acid (HEDP), and the ideal temperature and duration were found to be 30 min at 65 °C and 30 min at 85 °C. Moreover, the tensile strength, weight loss and morphologies of the samples were examined. Because sodium chlorite does not leave behind any alkaline residues, it has been found to do less harm to cotton fibers and use less water for rinsing.

Keywords: Sodiumchlorite, cottonfabric, bleaching, phosphonicacid

1. Introduction

The economy and environment of the textile industry are greatly impacted by the bleaching of cotton fabric [1,2]. Cotton fibers are naturally yellow or brown because of their structural makeup. Pigments that are naturally present may be the cause of this yellowish and brown discoloration [3]. Additionally, this color pollution may be brought on by environmental factors such as soil, climate, drought, frost, dust, and insects [4–6]. Before dyeing and finishing, which is one of the crucial steps, cotton fibers must typically undergo pretreatment to remove the natural pigments [7]. Bleaching is used to turn colored materials into the white fabric while causing the least amount of fiber deterioration possible. To achieve the desired whiteness, the bleaching chemicals either oxidize or decrease the coloring matter [8]. Washing off the treated colours and material yields satisfactory whiteness [1,9].

For a long time, textile and paper pulp have been bleached to a high white without losing strength using sodium chlorite (NaClO_2), which is a known commercial

chemical [10,11]. NaClO_2 is also easy to obtain and stores reasonably well. Additionally, it is a persistent free radical that acts as a one-electron oxidant in reactions with reducing substrates such as amines, sulfides, and phenols [12,13]. The activation of stable aqueous NaClO_2 solutions under alkaline conditions requires acidification. The rate at which sodium chlorite decomposes into chlorine dioxide (ClO_2), a potent oxidizing gas, increases with decreasing pH values and increasing bleach bath temperature (over 70 °C) [14,15]. The amount of NaClO_2 in the solution has a direct relationship to the rate of ClO_2 production. Maximum ClO_2 synthesis occurs between pH 2.5 and 3.0 [1]. Since the rate of ClO_2 generation almost doubles for each 0.4 pH drop at 85 °C, an acid like formic, acetic, or phosphoric acid is advised to alter pH values between 5.0 and 3.0 [16,17].

The ineffective storage of ClO_2 , the possibility of a gaseous explosion, and the fact that it is typically coupled with hypochlorite hinder the effective usage of

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ClO_2 even though it is a potent and affordable oxidant and disinfectant [18]. However, since the current pandemic, chlorine dioxide use has grown significantly and is still growing [19,20]. It was decided at this point that it would be advantageous to revisit the NaClO_2 bleaching procedures to dispel some myths, particularly in the textile industry, and to improve their usability and safety.

The present work aims to find the most appropriate and environmentally safe process conditions to bleach cotton using NaClO_2 , caring about the quality of the bleached fabric. In this study, in addition to other bleaching studies, some phosphonic acids were used and it was determined that the whiteness value of the bleached fabric increased significantly. In addition, it has been determined that the bleaching process can be performed without the use of wetting agent thanks to the phosphonic acid used, and thus the cost is reduced. As a result of the characterizations performed on the bleached fabric, it was determined that the morphology of the fabric was less damaged than the fabrics bleached by another bleaching method. In addition, this study is remarkable in terms of being environmentally friendly and obtaining cotton fabrics with good stretching properties by the bleaching process.

2. Experimental

2.1. Equipments, materials and chemicals

With pots of 200 mL capacity constructed of AISI 316L stainless steel (thickness of 2 mm), the TM-Termal™ was utilized as a laboratory-size dyeing apparatus. The device has a 20-step, 36 programs, PT-100 temperature sensor, and 1–3 °C/min temperature control mechanism. 5 °C to 140 °C operational temperature range and heating rate. The Konica Minolta Spectrophotometer, CM-3600d, assessed the reflectance of the bleached samples' whiteness. meterLab Ag/AgCl electrode and analytical KCl were utilized with the pH meter 210 Standard. The Turkish company NUR Textile Co. provided the plain weave, 100 percent cotton. These were the fabric's specifications: 150 g/m² fabric weight, 30 weft yarns per centimeter, and 36 warp yarns per cm. Turoksi Chemical Co. supplied sodium chlorite as a 31% solution in water. Technical grade sodium tripolyphosphate (STPP), ethylenediaminetetraacetic acid (EDTA), amino trimethylene phosphoric acid (AMTP), polyacrylic acid (PAA), and 1-hydroxy ethylidene-1,1-diphosphonic acid (HEDP) were all provided by the Turkish domestic market. The following chemicals were acquired from Merck Chemical Company: acetic acid (CH_3COOH , 99%), sodium thiosulfate anhydrous ($\text{Na}_2\text{S}_2\text{O}_3$, 98.0%), citric acid ($\text{C}_6\text{H}_8\text{O}_7$, 99%), phosphoric acid (H_3PO_4 , 85%), formic

acid (CH_2O_2 , 96%), sulfuric acid (H_2SO_4 , 95.0–98.0%), and potassium iodide (KI, 99.0%).

2.2. Process conditions

For bleaching trials, fabrics were weighed out to around 10 g for each one immersed in a liquor solution. The main components of the liquors include acids that are used to modify the pH from 2.5 to 4.0, wetting agents, various stabilizers, and sodium chlorite. The mentioned amounts of chemicals given in the recipes were added to provide a material to liquor ratio of 1/10. Wetting agent (1 g/L), stabilizer (1 g/L), and NaClO_2 (0.850 g–5.225 g/L) were all used. The stabilizer agent STPP was employed. Acids including formic acid (FA), acetic acid (AA), phosphoric acid (PA), citric acid (CA), 1-hydroxy ethylidene-1,1-diphosphonic acid (HEDP), polyacrylic acid (PAA), and amino trimethylene phosphoric acid (AMTP) were used to change the pH of the solution. Both a pH regulator and a stabilizer are employed with HEDP. The completed mixture was put into 200 mL stainless steel HT tubes with fabric, and the tube lids were tightly sealed. The alphabetical order of a, b, c, d given in Fig. 1 is the order in which the reagents are added to the process. The heating program was run following the replacement of the tubes on the dyeing machine heel (Fig. 1). After the period, the tubes were taken out of the device and opened. First, warm water was used to rinse the bleached materials, and then tap water was used.

If the investigations are treated chronologically, the trials were conducted at various times (0.5, 1.0, 1.5, and 3.0 h), but the best time to access the other conditions were maintained constant. Trials were then undertaken at various temperatures (30, 50, 70, and 95 °C) while maintaining other parameters constant to identify the ideal temperature, which is identical to the ideal length setting. Following the establishment of the ideal times and temperatures, the ideal bleach concentration was established using a range of NaClO_2 concentrations (0.85, 1.70, 3.40, 4.25, and 5.25 g). Finally, experiments were carried out at various pH values (2.5, 3.0, 3.5, and 4.0) to find the ideal pH value, which has a significant impact on bleaching.

2.3. Preparation of reference fabrics

The reference fabric served as a witness throughout the investigation as it was used to compare research fabrics bleached with NaClO_2 to reference fabrics bleached with H_2O_2 . The witness fabric was bleached with 5g/L H_2O_2 . A solution of sodium hydroxide was used to modify the pH before bleaching, which was done at a pH of 10.5.

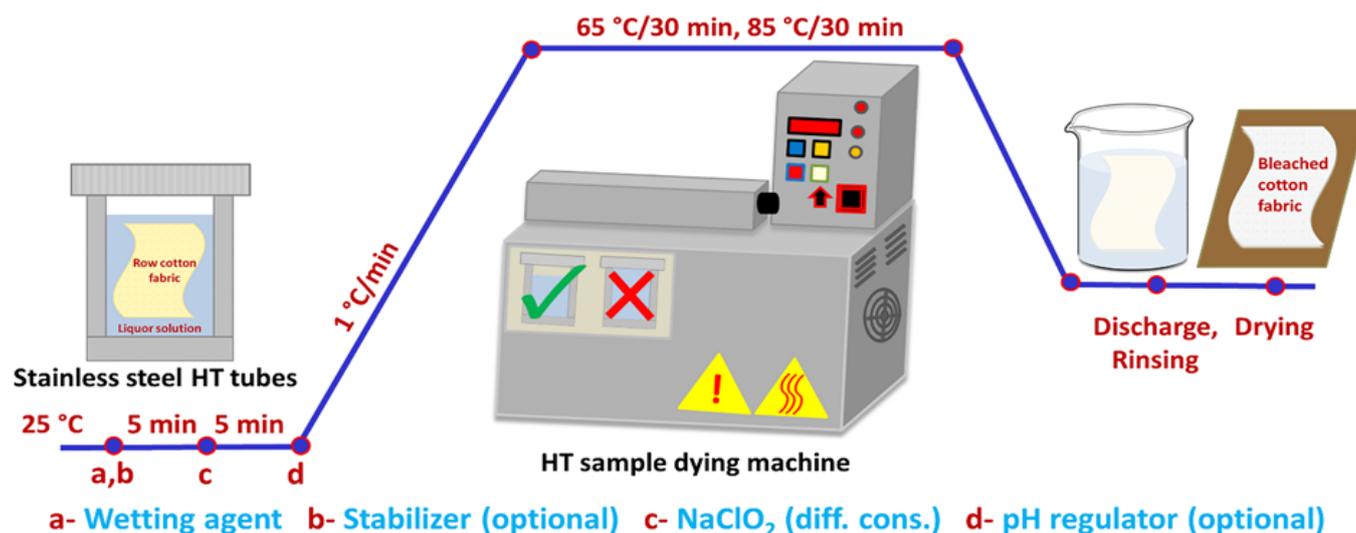


Figure 1. Schematic presentation of the cotton fabric bleaching process

The optimal bleaching time and temperature were found to be 2 h and 95 °C, respectively, and a nonionic STPP agent was utilized as the wetting agent.

2.4. Degree of whiteness

Necessary comparisons were made by carrying out the whiteness measurements of the experimental fabrics bleached with NaClO₂ and the reference fabrics bleached with H₂O₂. At 400 nm, the whiteness was measured. The degree of whiteness of bleached cotton samples, expressed as the whiteness index (W.I.), was measured using a Konica Minolta spectrophotometer (CM-3600d). The equation (ASTM Method E31373) was used to compute the W.I. in terms of the CIE Y (blue) and (green) reflectance components [21].

$$W.I. = \frac{4Z}{1.18} - 3Y \quad (1)$$

where Y and Z are the readings of the device.

2.5. Weight Loss

The following calculation was used to determine fabric weight decrease based on dry weight:

$$\text{Weight Loss (\%)} = \frac{W_1 - W_2}{W_1} \times 100 \quad (2)$$

where W₁ and W₂ represent the cotton fabric's dry weights before and after the bleaching process, respectively.

2.6. UV-visible absorption spectra of species

The spectrum of absorbance of NaClO₂ solutions with the addition of various quantities of acid was examined using a UV-Vis spectrophotometer (3100-PC) in the 240–500 nm wavelength range.

2.7. Tensile strength (RT)

Cotton samples' tensile strength was assessed using the strip method by ASTM procedure D 2256-66T [22].

2.8. Field Emission Scanning Electron Microscopy (FE-SEM) analysis of cotton samples

Cotton samples were analyzed using a Hitachi S-4700 scanning electron microscope (SEM) to learn more about the microstructure of the cotton samples and determine whether the residues had been successfully removed after bleaching. A sputter machine (Cressington 108 auto sputter coater) was used to coat cotton cloth. Using ImageJ software, the measurement of the diameter and size of at least 50 randomly selected cotton fibers were measured to ascertain the size, diameter, and size distribution of the cotton fibers.

2.9. Fourier transforms infrared (FT-IR) analysis

In order to determine if chemical bonding exists in raw and bleached cotton in the range of 4000–400 cm⁻¹ at a resolution of 4 cm⁻¹, Bruker Fourier transform infrared (FT-IR, Vertex-70) spectroscopy was utilized.

3. Results and discussions

According to earlier research, sodium chlorite breaks down into a strong oxidizing gas called ClO₂ and decomposes more quickly at low pH levels and higher temperatures [23,24]. Taking into account this information, the study's optimal timing and temperature were first established. The optimum temperature and time required for cotton fabrics to have a sufficient degree of whiteness were determined as a result of a series of bleaching experiments given in Table 1. As a result of the experiments performed at different temperatures and times using the same wetting agent

and acid conditioner, the ideal time and temperature were found to be 65 °C 30 min and 85 °C 30 min.

Examining the impact of the chemicals used to modify pH on cotton bleaching is one of the most crucial aspects of this study. The following substances are used to change the pH: AA, CA, PA, FA, AMTP, PAA, and HEDP. Utilizing sodium chlorite solutions of various concentrations and acids, information regarding the ideal concentration and acid was discovered to explore the bleaching impacts of NaClO₂ concentration and acidity controllers on cotton fabric bleaching. The experiment process was determined as a total of 60 min at pH = 3.0, 65 °C/30 min and 85 °C/30 min time/temperature.

The influence of acidity regulators and NaClO₂ concentration on the whiteness of cotton fabric is depicted in Fig. 2. In all acid tests, as shown in the figure (Fig. 2a), the amount of whiteness of the cotton fabric rises as the bleach concentration rises. It was discovered that the determined whiteness levels were on the equation with or higher than the reference sample following bleaching studies where the bleaching intensity ranged from 4.50 to 5.25. The most noteworthy acids among the used acid regulators were found to be FA and HEDP.

The whiteness rating of the bleaching experiments utilizing FA and HEDP at various NaClO₂ concentrations is contrasted with the whiteness rating of the reference fabric in Fig. 2b. The fabric's whiteness index was found to be low in the lower concentration tests (0.85, 1.70), to be reasonable in the slightly higher concentration test (3.40), and to be equal to or better in the experiments with concentrations of 4.25 and 5.25 for the reference fabric. The bleaching procedure utilizing an HEDP acid inhibitor at a concentration of 5.25 g/L NaClO₂ produces the best whiteness index (W.I. = 83).

One of the most crucial parameters, pH, was tuned at this point after the ideal temperature and time changes had been established in the initial phase of the study. The rate of sodium chlorite breakdown accelerates at low pH levels and high temperatures, according to earlier research [23]. To analyze the changes in the whiteness index of the fabrics, a variety of acid regulators (AA, CA, PA, FA, AMTP, PAA, and HEDP) were used during the bleaching trials at various pH levels (2.5, 3.0, 3.5, and 4.0) in Fig. 3a. As the pH level decreases, it can be observed in Fig. 3a that the whiteness value of the fabrics increases significantly. In all of the acid regulators utilized in the study, pH 2.5 and 3.0 produced the best whiteness results. This outcome is in line with research in the literature that looked at NaClO₂ and pH.

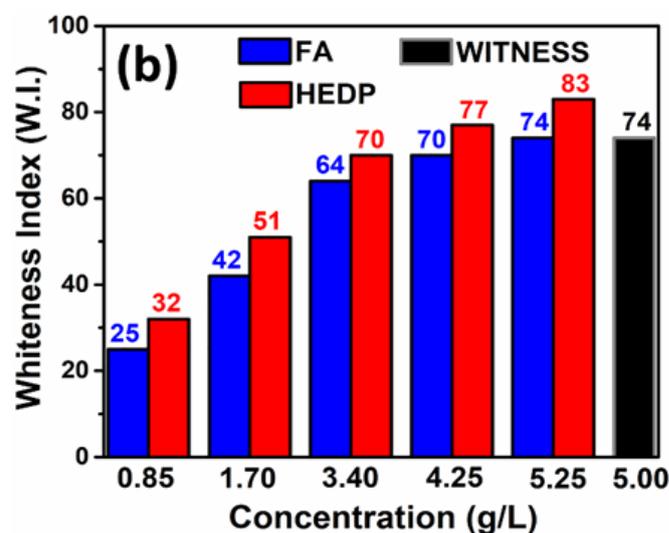
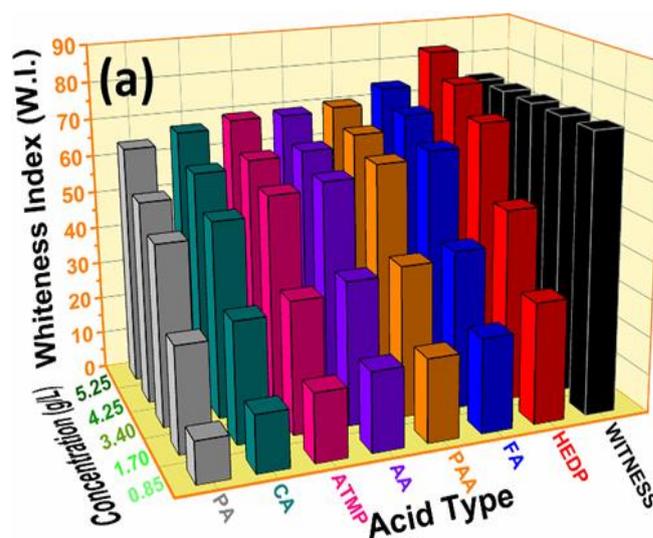


Figure 2. (a) The effect of sodium chloride amount and acid types on cotton bleaching and (b) the whiteness values obtained in the bleaching experiments with FA and HEDP compared with the whiteness value of the reference fabric

Table 1. Determination of optimum duration and temperature in cotton bleaching

Code	Temp. (°C)	Duration (min)	Whiteness Index(%)
1	30	30	44
2	30	60	50
3	30	90	55
4	30	180	58
5	50	30	61
6	50	60	64
7	50	90	71
8	50	180	74
9	65	30	63
10	70	30	65
11	70	60	72
12	70	90	75
13	70	180	80
14	85	30	67
15	95	30	69
16	95	60	78
17	95	90	80
18	95	180	82

NaClO₂: 5.25 (g/L) pH regulator: Formic acid, pH: 3.0, Stabilizer: STPP, Liquor Ratio: 1/10

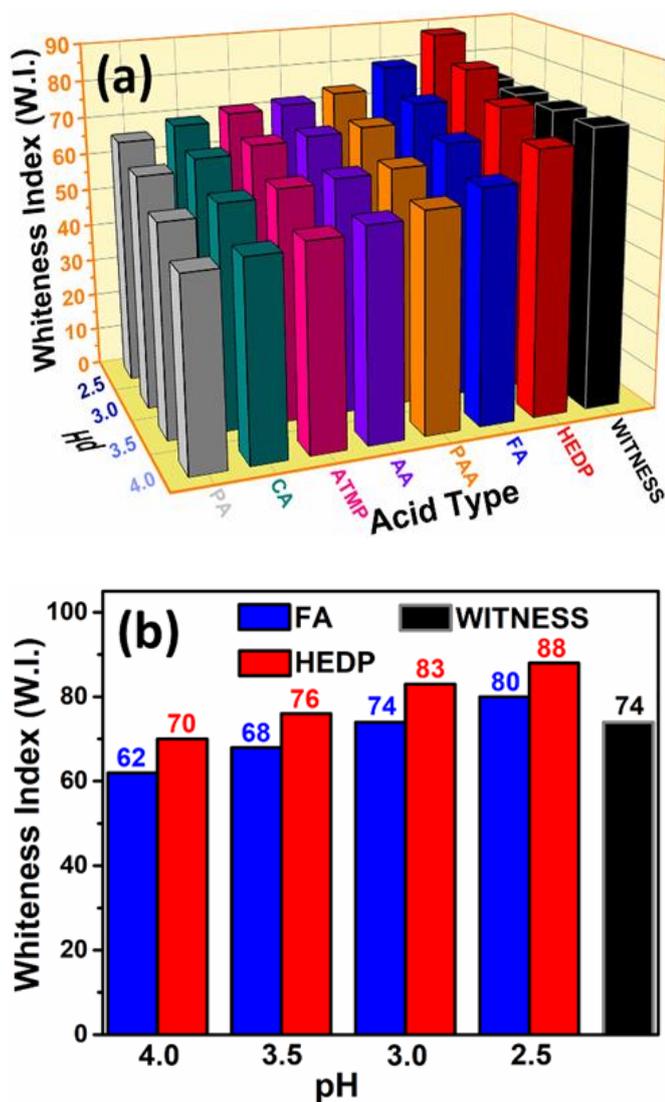


Figure 3. (a) The effect of pH on bleaching and (b) Comparison of the bleaching processes performed at different pH values by means of FA and HEDP with the reference fabric

The greatest whiteness index amongst acid controllers is provided by the HEDP acid, which can be better compared using Fig. 3b. It performs higher than the whiteness rating of the witness textile sample even when the pH level is 3.5.

The FTIR spectra of the unbleached fabric and the fabric bleached with sodium chlorite are shown in Fig. 4. The weak band at 1240 cm^{-1} is assigned to pectin's C-O side chain vibration [25]. However, this peak is not seen in the FT-IR spectrum of the bleached cotton, indicating that pectin was removed as a result of the bleaching process. At 1155 cm^{-1} , a comparable condition is also observed. This finding suggests that the bleaching process deforms the anti-symmetric C-O-C bond [26,27].

The raw and bleached cotton peak measurements of 1027 and 885 cm^{-1} and 1024 and 882 cm^{-1} , respectively, show that pectin and hemicelluloses are present in the cotton fiber [28]. The O-H frequency is also responsible for the broad peaks at 3328 and 3325 cm^{-1} in both spectra,

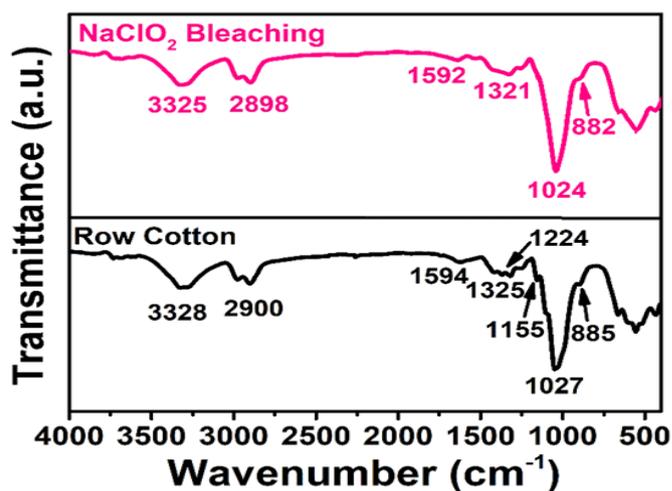


Figure 4. FT-IR spectra of raw cotton fabric and NaClO₂ bleached cotton fabric

whereas the C-H stretch bands are responsible for the peaks at 2900 and 2898 cm^{-1} [29].

Fig. 5a and Fig. 5b show, respectively, the structure of the reference bleaching with H₂O₂ and the test sample bleached with NaClO₂. The reference cloth bleached with H₂O₂ and the untreated fabric utilized in the experimental research come from the same raw fabric roll. According to Fig. 5a, the structure of H₂O₂ bleached cloth is made up of fibers of comparable sizes. Inset in Fig. 5a depicts the periodicity of the fibers' measured diameters. 142 nm was the average diameter determined. The red dashed circle in Fig. 5a highlights the area where the fiber ripped following bleaching with H₂O₂. The surface of the fiber has deformations, as shown in the image. Furthermore, as shown by the red arrow, it is apparent that there are contaminants or chemical residues from bleaching agents on or between the fibers.

The cotton fabric in Fig. 5b has been bleached with NaClO₂ at pH = 3.0 with the help of the HEDP, pH regulator. The smooth fibers in the figure are greater in diameter than the fibers in Fig. 5a, and they are also not distorted. An inset of Fig. 5b displays the frequencies of the fiber sizes that were measured. Its 213 nm average diameter was determined. When comparing the two bleaching methods, it can be seen that the bleached with NaClO₂ results in less fiber deformation and leaves no bleaching residue on the cloth. These findings agree with previous research [10].

Finding out how much weight is lost in the fabric after bleaching is one of the key aspects of fabric bleaching. The weight of the fiber cotton is affected by various NaClO₂ concentrations and acidity regulators, as shown in Fig. 6a. As can be observed in the image, fabrics bleached with FA and HEDP have the highest decreased weight among acid regulators. As can be seen in Fig. 6a,

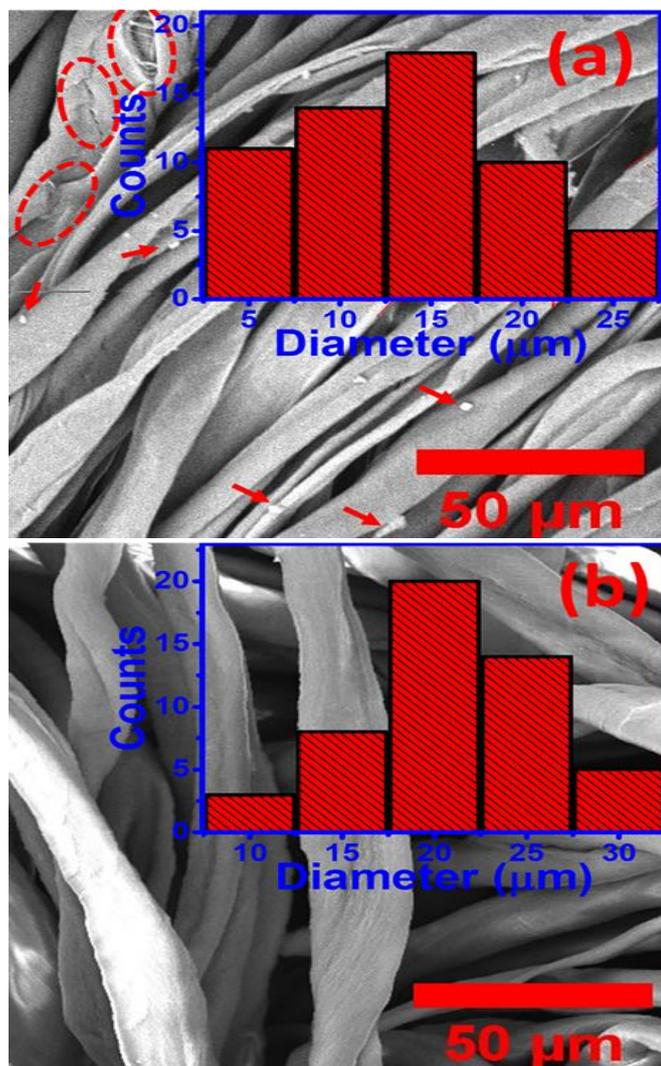


Figure 5. SEM images of (a) a reference fabric sample bleached with H_2O_2 and (b) an experimental cotton fabric sample bleached with $NaClO_2$ (top appendices in each figure show the frequency of the calculated diameters of the fibers)

acid controllers in terms of the bleaching trials' whiteness indices.

Fig. 6b is included so that you may more clearly understand how these two acid controllers and various $NaClO_2$ concentrations affect weight loss. The bleached fabric loses weight as the sodium chlorite concentration rises, as shown in the figure. In conclusion, the bleaching and elimination of contaminants in the bleached cotton fabric is the cause of the fabric's increased weight loss with a rise in the whiteness index. The most startling finding is that even though the sodium chlorite

Table 2. Effect of $NaClO_2$ concentration on the physical properties and whiteness index of the bleached cotton fabric

Code	$NaClO_2$ (g/L)	Whiteness Index (%)	Tenacity (kg f)	Elongation (%)
1	0.85	32	59.2	30.3
2	1.70	51	58.5	31.7
3	3.40	70	57.6	32.1
4	4.25	77	56.4	32.5
5	5.25	83	54.5	33.1
Witness	5.00 H_2O_2	74	51.2	34.3
Raw Cotton	—	—	68	21.3

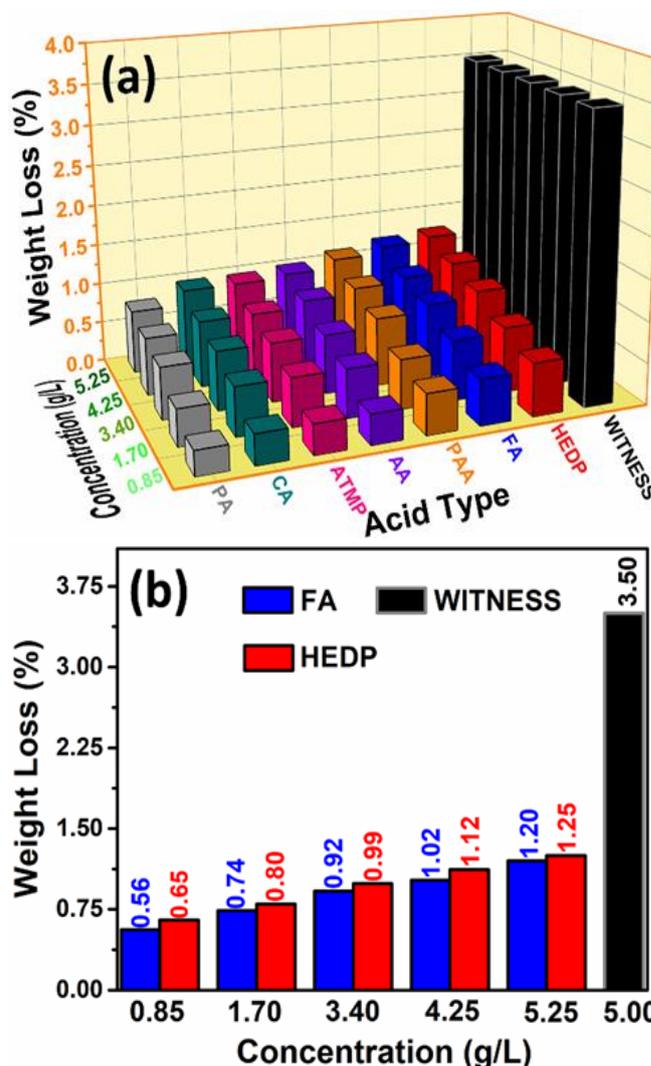


Figure 6. (a) The effect of $NaClO_2$ concentration on fabric weight and (b) Comparison of the percent loss in fabric weight as a result of bleaching processes performed at different $NaClO_2$ concentrations with the reference fabric

bleaching used in our investigation produced whiter results than the reference sample's whiteness, there was still a weight loss of about a third. This could imply that bleaching with $NaClO_2$ is cost-effective for techniques that do not result in a significant loss in fabric weight. The mechanical characteristics of bleached cotton fabrics, including tenacity and elongation% at the break, are shown in Table 2 and Table 3. Effect of pH and $NaClO_2$ concentration on the physical properties and whiteness index of the bleached cotton fabric. The table shows that as the concentration of $NaClO_2$ rises, the tenacity of the bleached fabric gradually declines.

Table 3. Effect of pH on the physical properties and whiteness index of the bleached cotton fabric

Code	pH	Whiteness Index (%)	Tenacity (kg f)	Elongation (%)
1	2.5	88	53	34
2	3.0	83	54.5	33.1
3	3.5	76	56.6	32.1
4	4.0	70	57.4	32.8
Witness	10.5	74	51.2	34.3
Raw Cotton	—	—	68	21.3

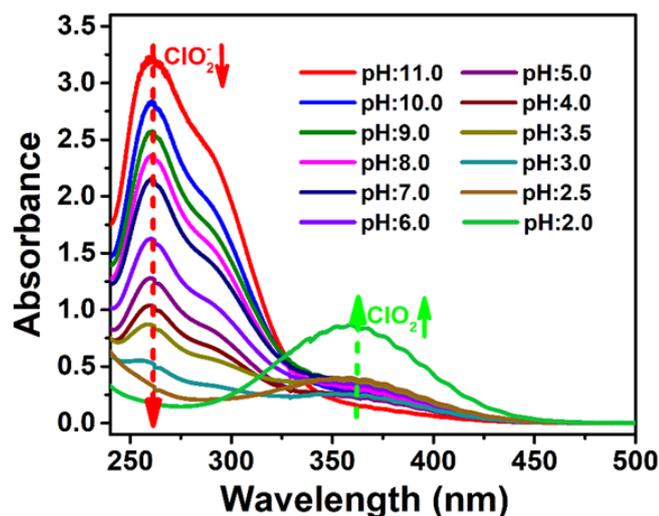


Figure 7. UV-visible absorption spectra of ClO_2^- and ClO_2 species in NaClO_2 solution under different pH irradiation (NaClO_2 concentration 0.8 g/L)

The situation is different when expressed as a percentage of elongation. Similarly, bleaching at low pH causes a decrease in the fabric's strength while increasing the whiteness index, percent elongation, and percent loss of weight of treated materials. The previous image discussed how bleach concentration increased as the weight of bleached materials increased.

As a result of the elimination of non-cellulosic components from cotton, it may be inferred that the weight loss, strength, and elongation have all increased, decreased, and are all increasing. A conclusion that can be drawn from the data is that the bleaching concentration and pH that should be utilized in the bleaching process are 5.25 g at pH = 3.0, which results in a desirable whiteness index and greatly protects the mechanical characteristics of the cotton fabric.

Considering the economic climate, even a 4.25 g concentration of bleach may be sufficient, according to the data gathered. However, the bleach concentration producing the best whiteness value is 5.25 g. Additionally, it has been found that bleach at this concentration provides a sufficient whiteness index and considerably safeguards the mechanical qualities of cotton fabric. The successful removal of these components may result in an increase in the whiteness index and a decrease in fabric weight since non-cellulosic substances in cotton fabric's natural structure darken the fabric's color.

Fig. 7 displays the absorbance spectrum of NaClO_2 solutions in various HEDP acid concentrations. This illustration depicts the absorption peaks of and at wavelengths of 260 nm and 360 nm, respectively [30]. As was already established, a rise in the amount of H^+ has a beneficial impact on the ability to oxidize. The fact that sodium chlorite can decompose into poisonous and highly corrosive ClO_2 gas is also known [31]. Because of this, experiments were conducted to identify the ideal

pH level by taking measurements at various pH levels. Once the UV-vis spectra were studied, it was found that at high pH values, there was no discernible change in the concentration of NaClO_2 and that NaClO_2 got used as the environment turned acidic, or as the pH value declined. It becomes an unfavorable gas as the pH value decreases (2 and below) [32]. It was decided to conduct cotton bleaching experiments between pH: 4.0 and 2.5 as a consequence of the data gathered.

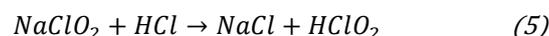
When water-soluble sodium chlorite is dissolved;



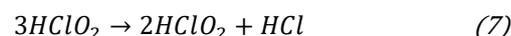
A straightforward solution is found using Eq. 3. Agster claims that bleaching is provided by acid chlorite, not sodium chlorite or its ions. According to the equations below (Eq. 4), chlorite acid bleaches by releasing active oxygen.



When bleaching, chlorine acid (HClO_2) rapidly breaks down into chlorine dioxide (ClO_2), the species that is active (Eq. 5), whose generation rate is related to the solution's sodium chlorite content.



They can produce the reactions described in Eq. 6 and Eq. 7, as well as the chlorous acid emitted in the acidic environment, generating a reaction as given in Eq. 5.



4. Conclusion

In this study, first, the optimum temperature and time parameters were determined. The optimum temperature and time were determined as 65 °C for 30 min and 85 °C for 30 min. In addition, the effect of NaClO_2 concentration on whiteness was investigated, and it was determined that the whiteness values obtained as a result of the bleaching study performed using 4.25 and 5.25 g/L NaClO_2 were better than the whiteness value of the reference fabric. Moreover, the effect of pH on bleaching was investigated, and it was observed that as the acidity of the medium increased, the whiteness increased in direct proportion. However, when working at very low pH values, undesirable ClO_2 gas output has

been observed. In addition to these, the effect of different acid types on bleaching was investigated, and the highest whiteness value was reached when HEDP was used (W.I. = 88). HEDP has reduced the use of chemicals, both because it acts as a wetting agent such as STPP, and because it enables fabrics to exhibit high whiteness even at low NaClO₂ concentrations. As a result of the calculations, it was seen that the weight loss in fabrics bleached with NaClO₂ was very small compared to the weight loss in the reference fabric. These results show that the bleaching process within the scope of this study is very economical. Moreover, it was observed that the bleaching process used in this study did little damage to cotton fibers and there was no alkali residue in bleached fabrics, so less water was needed to rinse the product and remove sodium. In addition, in this study, NaClO₂, which is the source of ClO₂, which has recently attracted a lot of attention as a disinfectant, was used. These results show that the bleaching process within the scope of this study is a very environmentally friendly process.

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