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Optimizing Ozone Gas Bleaching for Interlock Knitted Fabric:A Comprehensive Study on pH, Flow Rates, and Ozonation Durations

Semiha EREN [1] *, Merve ÖZTÜRK [1], Hüseyin Aksel EREN [1], Ezgi ERŞAN [2], Ahmed KARATAŞ [2]

[1] *Bursa Uludag University Faculty of Engineering and Architecture, Department of Textile Engineering, Görükle Campus, Özlüce, Nilüfer, Bursa*
[2] *Beneks Taahhüt Mühendislik Mümessillik Makina Sanayi Ve Ticaret Limited Şirketi, Bakır and Pirinç Industrial Site, Sardunya Street, Building No.5, Beylikdüzü, İstanbul*

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In this study, the bleaching of interlock knitted fabric using an innovative and environmentally friendly method involving ozone gas was explored. The bleaching processes were conducted under varying conditions: different pH levels (pH 7 to pH 12), ozone gas flow rates (2 L/min to 6 L/min), and ozonation durations (20, 40, and 60 minutes). Fabrics treated with ozonation were compared to those subjected to conventional bleaching methods. Assessments of the samples were performed for whiteness, bursting strength, color measurements, and fastness properties. Additionally, Chemical Oxygen Demand (COD) tests were conducted on wastewater from both conventional bleaching and ozonation processes. Optimal conditions were determined to be at pH 7, with a 6 L/min ozone flow rate, and a 60-minute duration. Under these conditions, the whiteness index reached 80 Stensby, and the yellowness index was 7.6. Furthermore, a reduction of approximately 90 % in COD values was observed, indicating a significant decrease in environmental impact. However, a decrease of about 30 % in burst strength was also noted. This study highlights the potential of ozone technology to replace traditional chemical-intensive methods, offering a more sustainable solution for the textile industry.

Keywords: cotton, bleaching, ozone, pH.

1. INTRODUCTION

Cotton fiber, belonging to the Gossypium genus in the Malvaceae family, is a widely used material in the textile industry (approximately 48 %) [1 – 4]. People prefer it due to its features such as absorbency, breathability, and softness [5]. To utilize a product made from cotton fiber, various finishing processes are necessary. The fiber contains various contaminants, such as oil and wax, which reduce efficiency in finishing processes [6]. Various pretreatment processes are employed to remove contaminants from the cotton fiber. Desizing and bleaching processes are most commonly applied, along with hydrophilization and mercerization processes [7, 8]. In bleaching processes, hydrogen peroxide (H_2O_2) bleaching is the most widely used method [9, 10]. H_2O_2 is inactive in neutral aqueous environments [11], requiring activation for the bleaching process to occur. The suitable activator for H_2O_2 is also alkaline [8]. In alkali-H_2O_2 systems, perhydroxyl anions ($HO_2\cdot$) constitute the main bleaching agent, whitening the fabric by reacting with these radicals [6]. Although hydrogen peroxide is the most environmentally suitable bleaching agent, it requires long reaction durations and high temperatures [12, 13]. High temperature and alkaline bleaching baths also lead to fiber loss [5]. While pretreatment processes applied to cotton fabrics are effective, water, energy, and chemical consumption are high in the production stages [5, 8, 11]. In addition to H_2O_2 and alkali, chemicals such as stabilizers, wetting agents, and ion traps are added to the bleaching bath. For the textile finishing sector, where water consumption is quite high, annual freshwater consumption in European countries is 600 million m^3 [14]. Besides this consumption, the auxiliary chemicals used during the processes also make it difficult to purify textile wastewater. Consequently, researchers and textile enterprises have adopted an innovative and clean production approach, seeking new methods for textile finishing processes. Cleaner production is a process strategy that addresses all stages of the product life cycle and is used to prevent or minimize human and environmental risks [15]. This strategy aims to minimize the consumption of resources and reduce the emission of pollutants [16]. In addition, in line with sustainable development goals, textile finishing enterprises should primarily aim to reduce water and energy consumption. Ozone applications also support the concept of clean production and sustainable development goals with their features such as being environmentally friendly, economical, saving water and energy use, not containing harmful chemicals, and not needing auxiliary chemicals [17].

Ozone is an oxidizing substance that engages in numerous reactions with organic and inorganic substances, possessing a high oxidation potential of 2.07 eV [18, 19]. Being oxidative, it has emerged as an alternative to hydrogen peroxide in bleaching processes within the textile industry [20]. Factors influencing ozonation processes include ozone dose, duration, temperature, and pH. As the ozone dose and duration increase, the effectiveness of

* Corresponding author: S. Eren
E-mail: *semihaeren@uludag.edu.tr*

ozonation also increases. Ozone's solubility diminishes at higher temperatures, making it more effective in cold water. Ozone solubility is highly dependent on pH and temperature. While molecular ozone reacts in an acidic medium, hydroxyl radicals (OH·) react in an alkaline medium [21]. The oxidation potential of the hydroxyl radical is 2.80 eV, leading to faster oxidation in an alkaline medium. Prabaharan and Rao (2003) emphasized in their study that pH level is a crucial factor [22]. In their study, Perinçek et al. (2007) ozonated 100 % cotton woven fabrics at different pH values, revealing higher whiteness values in the pH range of 2 – 3 [18]. Prabaharan et al. (2001) observed in their study that as pH values increased, the degree of whiteness in fabrics decreased [23].

Interlock knitted cotton fabrics offer a superior combination of durability, comfort, and versatility, making them an ideal choice for a wide range of applications. Originating from rib knitting, interlock fabrics boast a smooth and stable structure that provides enhanced durability and a neat appearance [38]. Studies by Abhijit Majumdar et al. have highlighted the exceptional thermal properties and breathability of interlock fabrics, making them ideal for various uses [39]. Additionally, their flexibility in incorporating different yarn types and blends allows for customization to meet specific needs. Overall, interlock knitted fabrics emerge as the top choice for consumers seeking high-quality, comfortable, and long-lasting textiles across diverse settings. In their study, Chowdhary et al. examined 17 types of fabrics, including various combinations of Bamboo, Cotton, Rayon, Wool, Polyester, Spandex, Acrylic, and blends. These fabrics were represented in three knit structures: interlock, jersey, and pique. Across all studied knitted fabric types, the study found that the highest extension was consistently observed for fabrics composed of 100 % cotton [40].

In this study, interlock knitted fabric ozonated at different pH values, ozone dosages, and durations was bleached with ozone gas and compared with conventional bleaching. After bleaching, the optimum value was determined. Bursting strength and dyeing were performed using two different colors, and the COD values of the wastewater formed after bleaching were examined and evaluated.

2. MATERIALS AND METHODS

2.1. Materials

In this study, interlock fabric made from Ne 60 count single-ply yarn (Ne 60/1), with a mass per unit area of 170 g/m^2, was used. Conventional bleaching was used wetting agent (Merck), ion trap (Merck), stabilizer, hydrogen peroxide (Merck), and caustic (Merck). Ozone gas was generated using the Prodozon PRO DO25 model ozone generator with a capacity of 25 g/h. Ozonation processes were conducted on the Ataç BB01F sample dyeing machine, which was modified for ozonation. The modification involved adding a venturi injector to the solution circulation line. Acetic acid (Merck) and soda (Merck) were employed to adjust pH values. Dyeing processes utilized VINAZOL RED S3B and VINAZOL BRILLANT BLUE S-BRF reactive dyes with a laboratory-type sample dyeing machine. For COD testing, measurements were obtained using COD measurement kits (Merck).

2.2. Methods

In the study, interlock knitted fabric was bleached conventionally with the operating prescription given in Fig. 1 to be compared with fabrics bleached with ozone gas. 1 g/l wetting agent, 0.5 g/l ion immobilizier, 2 g/l caustic, 2.5 g/l hydrogen peroxide were used in the prescription. Ozone bleaching processes were carried out under the conditions given in Table 1 below.

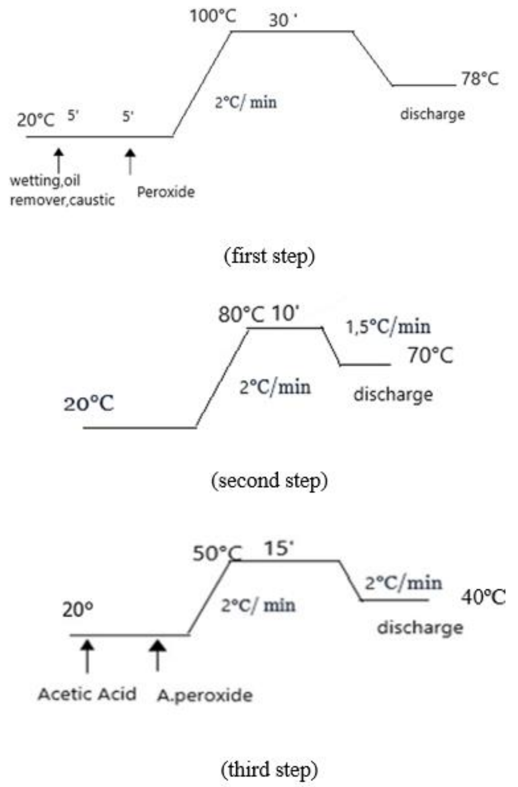


Fig. 1. Bleaching process steps

Table 1. Ozone working parameters

pH	Ozone gas flow rate, L/min	Durations, min
7	2	20, 40 and 60
	6	20, 40 and 60
12	2	20, 40 and 60
	6	20, 40 and 60

As a result of the tests, the optimum condition was determined and dyeing, COD, and bursting strength tests were carried out. For the dyeing processes, light and dark tone dyeings were obtained by working at 60 °C for 60 minutes with 1 % and 5 % dye concentrations.

After bleaching and dyeing, color measurements were made on a Konica Minolta CM3600D model spectrophotometer, and temperature and pH measurements were made on a WTW PH 3210 model pH meter. For COD measurements of conventional bleaching water and ozonation waters, the test was carried out for 2 hours at 148°C using Merck COD measurement kits and a WTW CR 2200 model thermoreactor. The test result was measured with a Spectroquant Pharo 300 model UV-visible spectrophotometer. This measurement was made according to the standard titrimetric method (Standard Methods 5220 C: Closed Reflux, Titrimetric Method, APHA, 19th ed., American Public Health Association, 1995). Washing fastness is based on TS EN ISO 105-C06, rubbing fastness is based on TS EN ISO 105-X12, and bursting resistance is based on ISO 13938-1 standard. 412 NB HT model test device was used for washing fastness, the James Heal Crockmeter test device was used for rubbing fastness, and the SDL Atlas Brand M229P PnuBurst model test device was used for bursting strength.

3. RESULTS AND DISCUSSION

3.1. Whiteness and yellowness results

Pre-treatment processes are performed to prepare the fabric for dyeing, and bleaching is one of these processes applied to enhance the whiteness of fabrics by breaking down color-imparting substances in cotton. Hydrogen peroxide is commonly used in conventional bleaching processes [24]. Ozonation processes, an innovative method with high oxidation potential, can now replace traditional bleaching. Literature reports indicate that ozone increases the whiteness of cotton fabrics [5, 7, 18, 25]. Fig. 2 illustrates the whiteness degrees of ozonated cotton knitted fabric. Examining the results in Fig. 2 reveals that the value closest to the degree of whiteness in conventional bleaching (79.1 Stensby) was obtained after 60 minutes of ozonation (80 Stensby) at pH 7. Additionally, as the ozone dose and duration increased, whiteness degrees increased in all processes. To investigate the effect of pH values on whiteness in ozonation, pH 7 and pH 12 values were selected. The ozone molecule exhibits two different reactions under acidic and alkaline conditions. In an acidic medium, a direct reaction occurs with molecular ozone, while in an alkaline medium, an indirect reaction occurs with free radicals reacting [20]. At low pH values, molecular ozone causes selective reactions targeting chromophoric bonds in colored products [18]. Perinçek et al. (2007) noted in their study that there was no significant difference in whiteness as a result of ozonation in acidic and neutral mediums. Therefore, pH 7 was chosen in this study for its economic and environmentally friendly aspect, avoiding the use of chemicals in a neutral medium. The higher whiteness results at pH 7 compared to pH 12 in Fig. 2 support this observation.

Bleaching cannot eliminate yellowness in the fiber [26], primarily due to the presence of colored pigments called flavan in the fiber [27]. Even after cleaning, natural fibers and fabrics made from them may still exhibit yellowing. Additionally, materials tend to turn yellow when exposed to light and dirt [28]. Therefore, the yellowness degrees of fabrics ozonated at two different pH values were examined and compared with conventionally bleached fabric. Yellowness results are presented in Fig. 3, showing an exact opposite trend to the whiteness values: as the whiteness values increased, the yellowness values decreased.

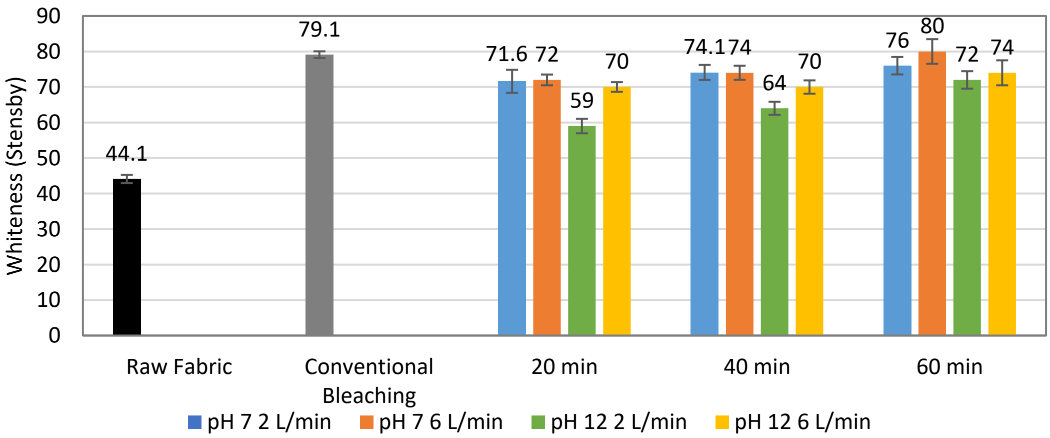


Fig. 2. Whiteness degrees of fabrics ozonated at pH 7 and pH 12

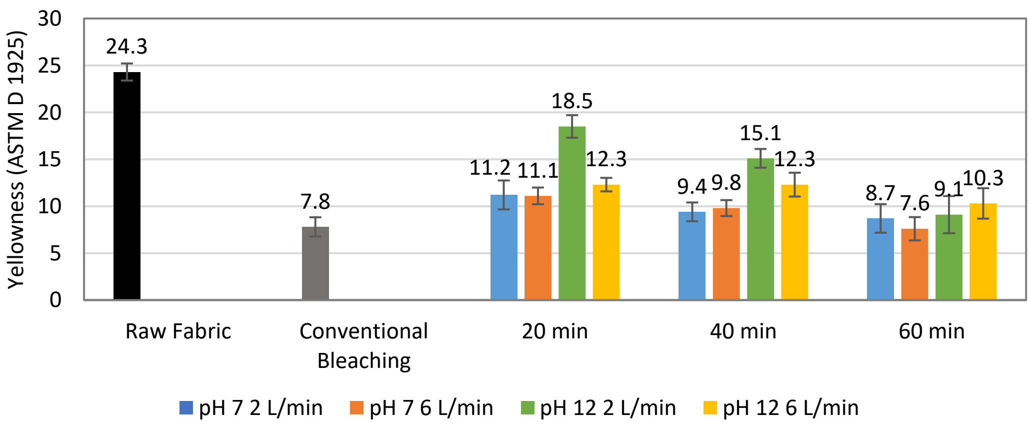


Fig. 3. Yellowness degrees of fabrics ozonated at pH 7 and pH 12

3.2. COD test results

COD measures the oxygen value of organic matter through a strong chemical oxidizer in a water sample and stands as the most widely used test method in wastewater characterizations [24, 29, 30]. As environmental impact in textile finishing wet processes gains significance today, the COD of conventional and ozone bleaching waters was determined. COD kits capable of measuring in the range of 500 – 10000 mg/L were employed for measurements. In ozone bleaching, the pH 7 – 6 L/min – 60 min process, which yields the highest degree of whiteness, was selected. Examining Figure 4 reveals that the COD value of the conventional bleaching bath is significantly higher than that of the ozone bath. A reduction of approximately 91 % in COD values was observed due to the ozonation process being conducted in a neutral environment without the use of auxiliary chemicals. This finding aligns with the literature [8, 10]. Furthermore, the high temperature used in the conventional bleaching process leads to the separation of impurities on the cotton, rendering the bleaching bath more polluting. In contrast, the use of low temperatures in ozone bleaching and the spontaneous conversion of ozone into oxygen contribute to obtaining low COD values [10].

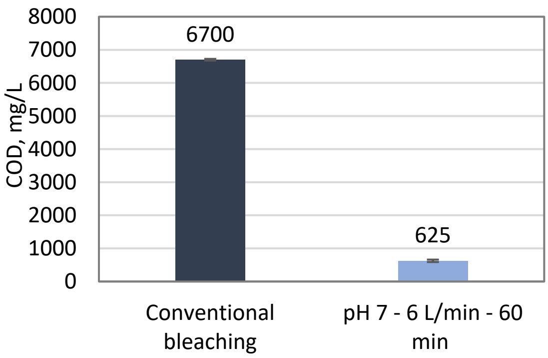


Fig. 4. COD values of conventional and ozone bleaching baths

3.3. Burst strength test results

The test method employed to assess the strength of textile materials influenced by force from various directions simultaneously is referred to as burst strength [31]. This test measures the pressure needed to burst the fabric [32]. The burst strength test was conducted to determine the strength loss of the raw fabric after conventional and ozone bleaching. Results of the burst strength test for the samples are presented in Fig. 5. Upon examination of Fig. 5, the sized raw fabric exhibits the highest strength value, while the fabric bleached with ozone gas shows the lowest strength value. This discrepancy is attributed to the oxidative properties of ozone [17]. The presence of NaOH in the H_2O_2 solution leads to homolytic fission, forming hydroxyl ions, and the resulting hydroxyl radicals transform cellulose into oxycellulose [33]. A decrease in the strength characteristics of fabrics containing oxycellulose is also observed. Öztürk and Eren (2010) stated in their study that ozone decomposes rapidly at high pH values (pH 7 and above) and generates hydroxyl radicals with high oxidation potential [20]. The resulting hydroxyl radicals quickly break double bonds, causing damage to the chains on cellulose. This leads to a decrease in strength values [5, 36, 37]. Fabric images after bursting strength are given in Table 2.

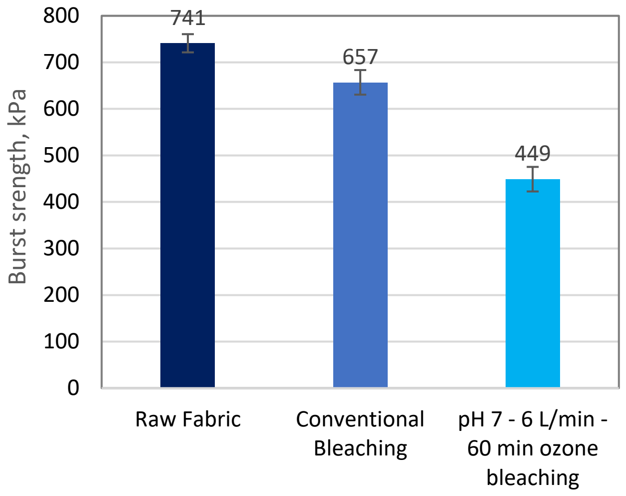
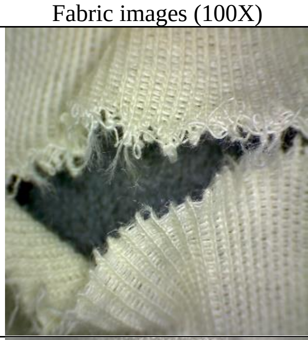


Fig. 5. Burst strength test results of conventional and ozone-bleached fabrics

Table 2. Fabric sample images after burst strength test

Sample names	Fabric images (100X)
Raw fabric	
Conventional bleaching	
Ozone bleaching	

3.4. Color measurement and fastness test results

Color is a crucial factor in today's modern world, significantly influencing the commercial success of a product [34]. Therefore, ozone-bleached fabrics must be dyed to enhance their marketability. In this study, ozone-bleached fabric and conventionally bleached fabric were dyed at two different dyeing concentrations under pH 7 – 6 L/min – 60 min conditions, selected as the optimal working range. Dye concentrations of 1 % and 5 % were employed to achieve different shades. Images of dyed fabric are given in Table 3.

Table 3. Images of dyed fabrics

Colors	Red	Blue
Light tone (1 %)		
Standard (conventional bleaching – dyeing)		
Sample (ozone bleaching – dyeing)		
Dark tone (5 %)		
Standard (conventional bleaching – dyeing)		
Sample (ozone bleaching – dyeing)		

Color measurement values for the fabric bleached with ozone after dyeing were compared to the fabric dyed through conventional bleaching, and the results are presented in Table 4 . Upon examining Table 4, it is observed that the color of the ozonated fabric is darker in the light tone of both colors. In dark tone red, DE was lower than 1, indicating an acceptable value. The ozonated fabric in a dark blue color appeared darker. Overall, the color of the ozonated fabric can generally be similar to that of conventionally bleached and dyed fabric while using less dyestuff. This supports environmental friendliness and reduces economic costs by minimizing the use of dyestuff.

The resistance of products to the factors they encounter during production or use is called fastness [34]. Gray scales are used to evaluate the degree to which the dyed fabric soils an adjacent companion fabric. The color change in the dyed fabric and the degree of dye bleeding onto the accompanying fabric are determined by the gray scale. The grey scale means 1-little, 2-medium, 3-good, 4-fairly good, 5-very good [35]. Within the scope of the study, the washing and rubbing fastness results of the dyed fabrics are given in Table 5. When Table 5 is examined, acceptable values for both washing and rubbing fastness results were obtained.

4. CONCLUSIONS

The whitening efficiency of knitted cotton fabric at different pH values was explored using ozone technology, recognized for its innovative and environmentally friendly approach to textile pre-treatment. This study demonstrated that ozonation produced results comparable to those obtained from traditional bleaching baths containing hydrogen peroxide. Specifically, the highest whiteness was achieved within 60 minutes in a neutral medium.

The COD test results indicated that the ozonation bath is significantly more environmentally friendly than conventional methods. Overall, ozonated fabric provided satisfactory dyeing results, with an additional observation that the colors of the ozonated fabric appeared darker, suggesting a potential reduction in dyestuff usage. Furthermore, good wet fastness was observed after dyeing.

Table 4. Color comparison of ozonated fabrics after dyeing

	Color	DL	Da	Db	DE	K/S_sample	K/S_standard
Light tone (1 %)	Red	2.266	-3.235	-0.686	4.008	33.66	41.52
	Blue	3.605	-0.767	1.654	4.039	52.3	66.35
Dark tone (5 %)	Red	-0.557	0.077	0.387	0.682	125.34	118.52
	Blue	1.132	-0.083	-0.52	1.249	209.19	228.98
**The reference samples are conventionally bleached and dyed (blue and red) fabrics. K/S_sample: Kubelka-Munk scattering coefficient of the sample, K/S_standard,: Kubelka-Munk scattering coefficient of the standard							

Table 5. Washing and rubbing fastness results of the samples

		Washing fastness						Rubbing fastness	
		Wool	Acrylic	Polyester	Polyamide	Cotton	Acetate	Wet	Dry
Light tone	Red_con.	5	5	5	5	4	4 – 5	4	4 – 5
	Red_samp.	5	5	5	5	4 – 5	4 – 5	4 – 5	4 – 5
	Blue_con.	5	5	5	5	4	4	4	4 – 5
	Blue_samp.	5	5	5	5	5	4 – 5	4 – 5	5
Dark tone	Red_con.	4 – 5	4 – 5	5	5	4	4	4	4 – 5
	Red_samp.	4 – 5	5	5	5	4 – 5	4 – 5	4 – 5	4 – 5
	Blue_con.	4 – 5	4 – 5	5	5	4	4	4	4 – 5
	Blue_samp.	5	5	5	5	4 – 5	5	4 – 5	5

In summary, ozone (O_3) has effectively replaced hydrogen peroxide (H_2O_2) for fabric bleaching. The ozonation method achieved conventional whiteness values at room temperature without the need for high temperatures or chemicals typically required in traditional bleaching baths. The optimal process conditions identified in this study were pH 7, 6 L/min ozone flow rate, and a 60-minute duration. Dyeing was conducted at 60 °C for 60 minutes under these conditions.

This research highlights the novelty of using ozone gas in textile bleaching, presenting a sustainable and eco-friendly alternative to conventional chemical methods, thus contributing significantly to the field of textile engineering and environmental sustainability.

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