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Original Paper

The effect of surface modification with ozone in waterless ($ScCO_2$) dyeing of polyester

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ABSTRACT

In this study, ozonation was carried out prior to dyeing 100% polyester fabrics through spray application (without the use of water) at various durations and with different gas flow rates. The ozonated fabrics were dyed with Dianix Dry XF2 Rubine (disperse dye) in conventional and supercritical carbon dioxide ($scCO_2$) medium. The main objective of the study is to investigate the colour efficiency of the ozone modified polyester fabric in waterless dyeing. Hydrophilicity, colour measurement, tensile strength, fastness tests, SEM analyses were performed on the samples. The results of dyeing the samples in a waterless medium after ozonation revealed higher colour strength (K/S) and better fastness test results with breaking strength values than conventionally dyed fabrics.

I. INTRODUCTION

Water is one of the most important resources for the continuation of life [1]. Global warming, population growth, and rapid increase in industrialization have led to excessive water consumption and water scarcity [2]. The textile industry is also one of the polluting sectors where the use of water and chemicals in finishing processes is high [3-7]. About 10-15% of dyes used in textile dyeing processes are discharged into wastewater [8]. Textile wastewater contains high COD/BOD levels, suspended solids, salts, dyes, and various chemicals [9]. The release of these chemicals into nature through wastewater leads to the degradation of the aquatic ecosystem and an increase in salinity in freshwater resources [8, 10]. As of 2018, about 80% of the wastewater worldwide is released into nature without treatment, and 20% is treated by developed countries [11].

As environmental awareness continues to grow globally and the need for sustainable use of natural resources becomes more important, the textile industry is actively researching and developing new environmentally friendly production methods with minimal environmental impact. Research on production methods with less environmental impact in the textile industry is progressing rapidly today [12, 13].

One of the studies carried out to reduce water consumption is waterless dyeing process in supercritical carbon dioxide medium. Waterless dyeing is also the method used to reduce water usage. Unlike the conventional dyeing process, carbon dioxide is used instead of water as a solvent in this method [14-16]. Carbon dioxide is preferred in waterless dyeing since it is nontoxic, non-flammable, recyclable, and has low critical temperature and pressure

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values [14, 17-24]. In this method, the most commonly dyed textile material is polyester fiber, which shows swelling in this medium [14]. Disperse dyestuffs that can dye polyester fiber can be dissolved in carbon dioxide, which is a non-polar solvent, without the need for any auxiliary chemicals. In addition, these dyes penetrate into the fiber better in the supercritical carbon dioxide medium, which has low viscosity and high diffusion coefficient at critical points [16, 18]. Dyeing processes in this medium take place in 4 stages: dissolution of dye, transfer of dye to fiber, absorption of dye by fiber, and diffusion of dyestuff to fiber [18].

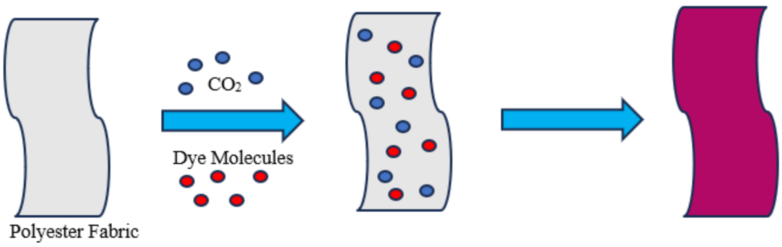


Figure 1. Diagram of dyeing process in supercritical carbon dioxide medium

Zheng et al. (2015) investigated the dyeing of polyester fabrics with C.I. Disperse Red 153 dyestuff in a supercritical carbon dioxide medium under different conditions, including temperatures (80, 110, 140 °C), pressure values (17, 23, 29 MPa), and times (20, 50, 80 min). Their findings revealed that an increase in temperature led to higher colour strength (K/S) values [25].

Xiong et al. (2017) focused on dyeing polyester fabrics using C.I. Disperse Yellow 82 dyestuff in supercritical carbon dioxide ($scCO_2$) medium. They identified the optimum conditions as 120 °C, 25 MPa, and 60 min, reporting enhanced durability of the fluorescent dyestuff in the $scCO_2$ medium [26].

Eren et al. (2019) explored the dyeing of polyester fabrics in a supercritical carbon dioxide medium with Disperse Blue 79 (C.I.11345) dyestuff, noting that fabrics maintained their strength and exhibited high fastness at 120 °C and 25 MPa [18].

Ozone gas has been utilized for post-washing and hydrophilization of hydrophobic synthetic fibers, such as polyester [27-29]. Studies have shown that ozone gas induces changes in both the fiber's surface and internal structure, transforming it into a hydrophilic structure with the formation of reactive regions and -CO and -COO molecules [28-31]. Surface modification with ozone can create controllable roughening, impacting surface wetting properties, a crucial requirement for manipulating superhydrophobic substrates [32, 33].

Gabardo et al. (2021) conducted a study in which they ozonated polyester fabrics for different durations (20, 30, and 45 minutes) and subsequently dyed them with C.I. Disperse Yellow 211 dyestuff [30]. The results demonstrated higher colour efficiency in ozonated fabrics, highlighting ozone gas as an alternative process to reduce the required dye amount. In their subsequent study, Gabardo et al. (2022) employed ozone-pretreated polyester fabrics for dyeing with C.I. Disperse Yellow 211 dyestuff [34]. Their findings indicated increased dye uptake in fabrics pre-treated with ozone, resulting in higher colour yield. This approach is expected to decrease textile wastewater treatment costs and reduce environmental impacts as dye consumption decreases.

As observed, surface modification studies using ozone are present in the literature. However, the distinctiveness of this study from the existing literature lies in the fact that polyester fabrics were ozonated using a spray without the use of water, and subsequently dyed in a waterless environment, followed by conducting tests and analyses.

II. EXPERIMENTAL METHOD / TEORETICAL METHOD

2.1. Materials

In the study, 100% polyester plain woven fabric (105 g/m2) was used. Dianix Dry XF2 Rubine (disperse dye) dyestuff was used for dyeing. Ozone gas was generated using the Opal PRODO2502-PRODOZON generator for the purpose of ozonating the fabric samples. To facilitate the ozone spraying procedure, a specialized ozone cabin, as described by Yigit et al. in their 2018 publication, was utilized [35]. Waterless dyeing processes were performed in Rapid Xiamen Model H-12 oil bath. The process diagram of the machine can be accessed from the publication [14]. Conventional dyeing was conducted using a Dispergator from Onan Kimya and acetic acid from Merck. Following conventional dyeing, the after-washing process involved the use of sodium hydrosulfite from Onan Kimya and sodium hydroxide from Merck.

2.2. Methods

2.2.1. Ozonation

The ozonation processes for the samples utilized an ozone generator with a capacity of 25 g/h. Ozone was generated using the corona discharge method within this generator as shown Figure 2. In this method, high voltage (electrical discharge) is applied to break down oxygen molecules, transforming them into oxygen atoms. These liberated oxygen atoms then combine with oxygen molecules, giving rise to the formation of triatomic oxygen molecules (O_3), as elucidated by Eren et al. in 2020 [15].

Polyester fabrics were placed on the printing template and ozonated in the device where the printing process can be carried out. Ozone gas is supplied to the system from the conical bottom part. Ozone gas entering the device affects the fabric between the templates. The application of ozone gas to the dry fabrics, referred to as gas flushing, was carried out for durations of both 15 and 45 minutes. These ozone gas treatments were administered at flow rates of 5 L/min and 10 L/min, following the procedure described by Gabardo and colleagues in 2021.

Figure 2. Ozone generator and printing device (1. Ozone Discharge Fan, 2. Screw,3. Top Cover, 4. Templates, 5. Conical Subtank, 6. Ozone Supply Inlet) [36]

2.2.2. Dyeing

Ozone-treated and untreated fabrics were subjected to dyeing with DyStar Dianix Dry XF2 Rubine dye at a 1% dye concentration. To facilitate a comparison between the two sets of samples, both conventional and waterless ($scCO_2$) medium dyeing methods were employed.

Waterless dyeing was performed using the Rapid Xiamen Model H-12 oil bath dyeing machine from DyeCOO in the Netherlands. The fabric was wrapped around beams and inserted into paint tubes with an inner volume of 290 ml, constructed from stainless steel. The dyestuff, prepared according to the specified dyestuff ratio, was introduced into the tubes as shown Figure 3. The amount of carbon dioxide (CO_2) required for each tube, based on the designated dyeing conditions, was calculated using data from the National Institute of Standards and Technology (NIST) Chemistry Web Book, as outlined by Yiğit et al. in 2021 [14]. The $scCO_2$ medium dyeing process was conducted at a temperature of 120°C and a pressure of 25 MPa for a duration of 90 minutes, without the need for post-washing.

In the case of conventional dyeing, the process was executed at a pH level ranging from 5 to 5.5 and a temperature of 120°C for 90 minutes. A liquor ratio of 1:15 was maintained, and 1 ml/l of a dispersing agent, along with 1 ml/l of acetic acid, was employed, as described by Eren et al. in 2019 [16]. Subsequent to conventional dyeing, the samples underwent post-washing, which involved the use of 2 g/L hydrosulfite and 3 g/L sodium hydroxide, carried out at 80°C for a duration of 20 minutes, following the procedure detailed by Gabardo et al. in 2021 [30].

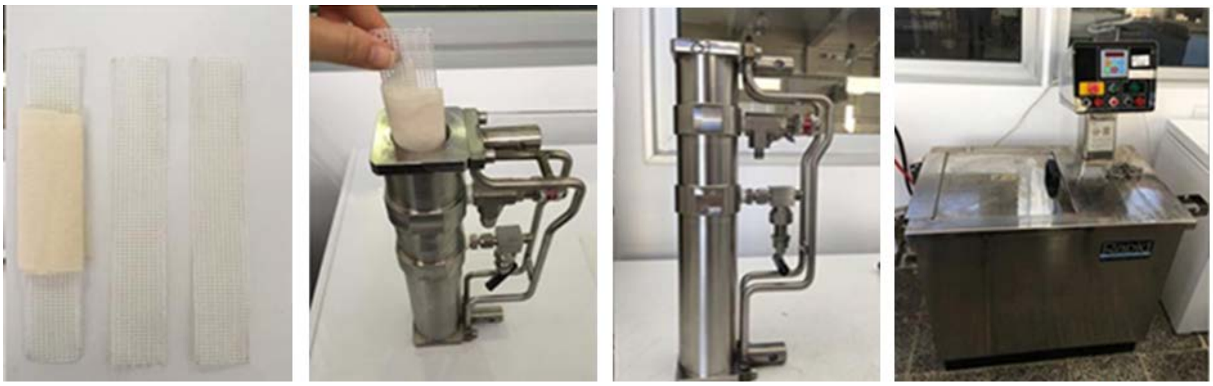


Figure 3. ScCO2 dyeing machine and tubes [16]

2.2.3. Hydrophilicity Test

Hydrophilicity tests of the samples were performed according to AATCC 79 test standard.

2.2.4. Color Measurement Tests

Colour measurements were made using Konica Minolta CM3600D model spectrophotometer device, on the samples obtained after dyeing in conventional and waterless ($ScCO_2$) medium.

2.2.5. Fastness Test

Colour Fastness to Washing Test: Samples were processed using 412 NB HT Model device according to ISO-105-C06 standard. After washing, the fabrics were measured using a Konica Minolta CM3600D model spectrophotometer. *Colour Fastness to Rubbing Test:* According to TS EN ISO 105-X12 standard, the wet and dry rubbing fastnesses of the samples were examined by using a James Heal Crockmeter. The rubbing fastness measurements of the samples were measured with a Konica Minolta CM3600D model spectrophotometer.

2.2.6. Tensile Strength Test

Tensile strength tests of fabrics were tested in SHIMADZU Model AG-X-Plus (Kyoto, Japan) test device with according to TS EN ISO 2062 standard.

2.2.7. SEM (Scanning Electron Microscope)

Images were taken at X2000 magnification according to the in-house method in Butekom Test Laboratory.

III.RESULT AND DISCUSSION

3.1. Hydrophilicity Test Results

Hydrophilicity test results of the samples are given in Figure 4. When the figure is examined, the hydrophilicity decreases when the untreated sample is dyed in supercritical carbon dioxide medium. When pre-treated with ozone, the decreasing hydrophilicity in the fabrics started to increase. In the supercritical carbon dioxide medium, the glass transition temperature (Tg) of polyester fibers decreases. Since this medium also has swelling properties on the fibers, it also causes polyester fibers to swell. Therefore, dye uptake of fabrics increases. However, in processes carried out in a supercritical carbon dioxide medium, oligomers accumulate on the fiber surface [37]. For this reason, the accumulation of oligomers on the surface affects the hydrophilicity.

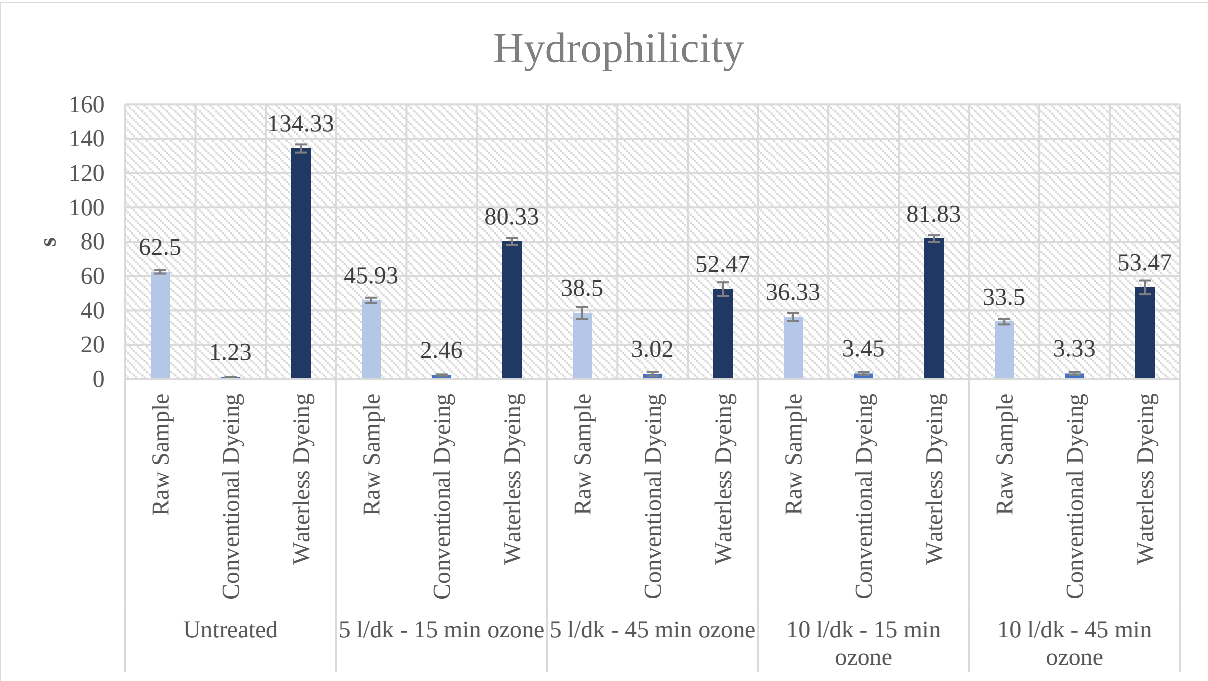


Figure 4. Hydrophilicity test results of the samples

It was observed that the hydrophilicity values increased after dyeing in $scCO_2$ medium for samples whose surface modification was made by ozonation. The reason for this is supported by the literature as ozone application increases the polar carboxyl ions, hydroxyl groups and amorphous region ratio in the fibers [30, 31, 36]. These groups increase the hydrophilicity of the samples. In addition, Lou et al. [38] conducted XRD analyses of raw and ozonated polyester fabrics after dyeing. According to the study, untreated raw PET fabric showed a crystalline ratio of 41.41%. Ozonated polyester fabric also showed a crystalline ratio of 37.87%. The crystallinity of the dyed PET fabric also decreased from 41.41% to 37.87% [38]. When the figure is examined, it is seen that the ozone gas flow rate increased, no significant change was observed in the hydrophilicity values of the samples, but the ozone treatment time is effective. When the ozone treatment time increased, the hydrophilicity values in the samples increased.

3.2. Colour Measurements

The samples were dyed after pre-treatment with ozonation. Table 3.1 shows the colour measurement results. The K/S values of the dyed fabrics in the $scCO_2$ medium were examined, and it was found that the colour efficiency was better than conventional dyeing, which is consistent with the literature [16]. The K/S values of the fabrics that underwent surface modification with ozone gas were higher than those without surface modification in both conventional dyeing and dyeing experiments in $scCO_2$ medium. Notably, the samples treated with ozone gas flow rate of 10 L/min for 45 minutes resulted in darker colours than the other samples, as shown in Figure 5 which displays images of the fabric samples. In surface treatments with ozone, the formation of carboxylate groups increases the fiber-dye interaction and allows more dye to penetrate the fiber [30]. Moreover, in the SEM images presented in Figure 7, more peeling and breakage were observed on the surface of fabric (e) treated for 45 minutes at a 10 L/min ozone gas flow rate.

Table 3.1. Colour measurement values obtained as a result of Dianix Rubin Dry XF2 dyeing of ozonated and untreated polyester fabrics in conventional and waterless medium

Dyeing Method	Ozonation	L*	a*	b*	DE*	Sample K/S
Untreated Conventional Dyeing	-	34,54	46,85	-0,52	-	189,92
Conventional Dyeing	5 l/min 15 min	-2.351	-2.720	2.318	4.278	222,72
	5 l/min 45 min	-2.285	-2.053	1.734	3.528	223,99
	10 l/min 15 min	-2.252	-2.802	1.304	3.824	217,52
	10 l/min 45 min	-2.742	-2.846	-2.483	4.668	230,24
Dyeing in Supercritical Carbon Dioxide Medium	Untreated	-3.667	-4.670	3.854	7.079	244,75
	5 l/min 15 min	-1.349	0.676	1.176	1.913	284,98
	5 l/min 45 min	-1.622	0.905	1.926	2.676	294,76
	10 l/min 15 min	-1.769	0.210	1.757	2.502	295,31
	10 l/min 45 min	-1.732	0.912	1.685	2.582	298,19

*Reference sample: conventional dyeing of untreated polyester fabric K/S:189,92

In Figure 6, the K/S values of the untreated and pre-treated samples dyed in supercritical carbon dioxide medium are given. When Figure 6 is examined, the K/S values of the samples treated with ozone gas were higher than the untreated samples. In addition, K/S values increased when ozone gas flow rate and time increased.

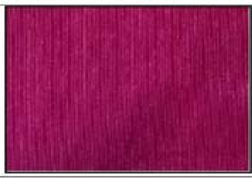
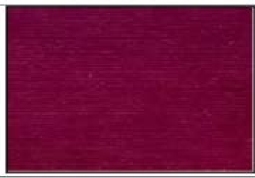
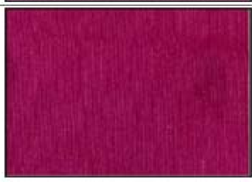
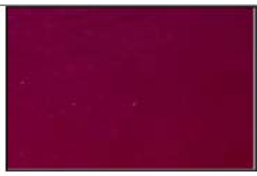
DYEING METHOD	Conventional Dyeing	Dyeing in supercritical carbon dioxide medium
Untreated		
5 l/min 15 min ozone pretreatment		
5 l/min 45 min ozone pretreatment		
10 l/min 15 min ozone pretreatment		
10 l/min 45 min ozone pretreatment		

Figure 5. Dyed fabric images of the samples

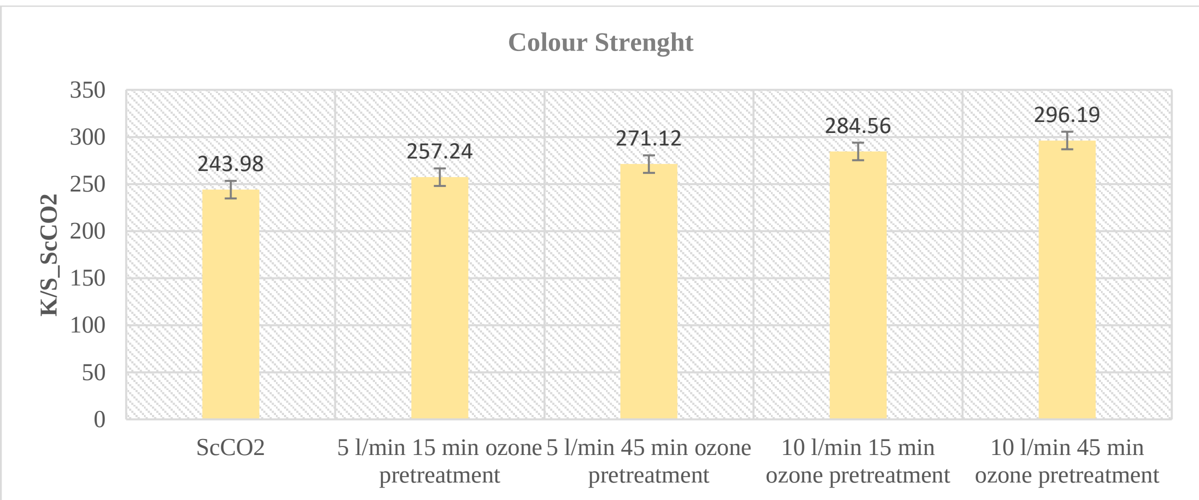


Figure 6. K/S values of pre-treated and untreated fabrics dyed in $ScCO_2$ medium: $K/SscCO_2$:243,98

3.4. Fastness Test Results

Washing and rubbing fastness tests were carried out in accordance with ISO standards. Table 3.2 presents the washing and rubbing fastness test results of the samples. The dyeing experiments conducted in $scCO_2$ medium demonstrated higher fastness results compared to the conventional dyeing process. During dyeing, $scCO_2$ acts as a swelling agent on synthetic fibers, causing the polymer to swell [18]. This effect enhances the diffusion of dyestuffs in the fiber, enabling deep penetration.

Consequently, dyeing experiments conducted in supercritical carbon dioxide medium resulted in better fastness results. In addition, it is thought that the groups formed on the fabric and the increase in the amorphous zone ratio in ozone surface modification affect both fastness and colour measurement results.

Table 3.2. Washing and rubbing fastness test results of the samples

		Washing Fastness						Rubbing Fastness	
		Wool	Acrylic	Polyester	Polyamide	Cotton	Acetate	Wet	Dry
Conventional Dyeing	Untreated	3	4	3/4	3/4	4	4	3	3/4
	5 L/min – 15 min ozone pretreatment	4	4/5	4/5	4/5	4/5	4	4	4/5
	5 L/min – 45 min ozone pretreatment	4	4/5	4/5	4/5	4/5	4	4/5	4/5
	10 L/min – 15 min ozone pretreatment	4	4/5	4/5	4/5	4/5	4	4	4/5
	10 L/min – 45 min ozone pretreatment	4	4/5	4/5	4/5	4/5	4/5	5	4/5
Supercritical carbon dioxide Dyeing	Untreated	4/5	4/5	4/5	4/5	4/5	4/5	5	4/5
	5 L/min – 15 min ozone pretreatment	4/5	4/5	4/5	4/5	4/5	4/5	5	4/5
	5 L/min – 45 min ozone pretreatment	4	4/5	4	4	4/5	4	5	4
	10 L/min – 15 min ozone pretreatment	4/5	4/5	4/5	4/5	4/5	4/5	5	4/5
	10 L/min – 45 min ozone pretreatment	4/5	4/5	4/5	4/5	4/5	4	5	5

3.5. SEM

Figure 7 shows SEM images of the samples, with the best surface images captured at 2000x magnification. Upon examining the SEM images, the surface of the samples not pretreated with ozone gas appeared smoother, while peeling and cracks were observed on the fiber in the pretreated samples.

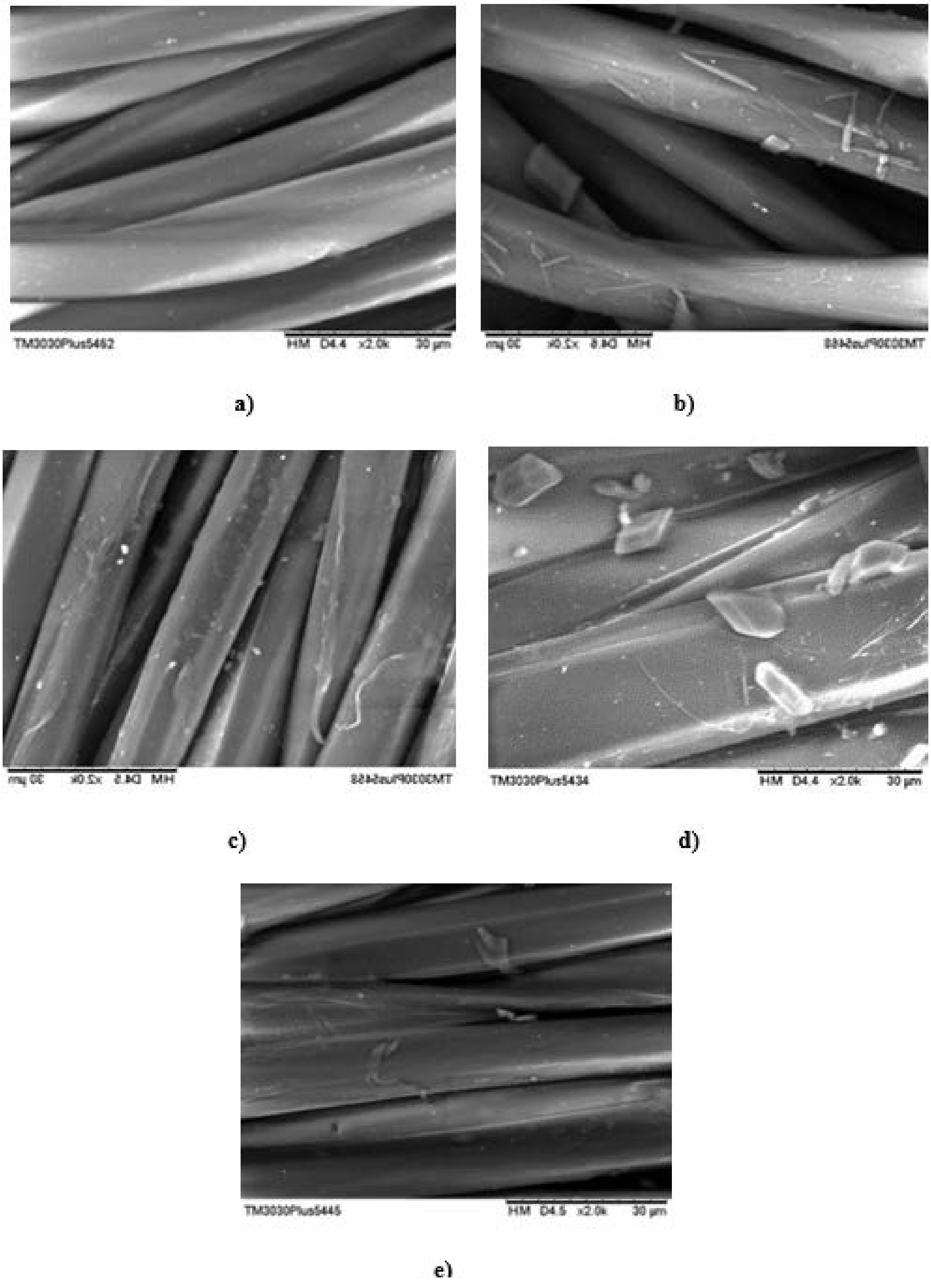


Figure 7. SEM images of raw and dyed fabrics; a) Sem image of raw fabric, b) SEM image of conventional dyed fabric, c) SEM image of fabric dyed in ScCO_2 medium, d) fabric dyed in scCO_2 medium, pre-treated with ozone for 45 minutes at a flow rate of 5 L/min ozone gas, e) fabric dyed in scCO_2 medium, pre-treated with ozone for 45 minutes at a flow rate of 10 L/min ozone gas

3.3. Tensile Strength Test Results

The tensile strength results are graphically presented in Figure 8, with three tests conducted for each sample in the study. The findings revealed noteworthy insights into the fabric strength under various dyeing conditions. It was observed that the strength of fabrics dyed through the conventional process without pretreatment experienced a decrease when compared to raw fabrics. However, fabrics dyed in $scCO_2$ exhibited higher strength values in contrast to conventionally dyed fabrics. When comparing pre-treated fabrics, a more substantial strength loss was evident in conventional dyeing, whereas the strength of samples dyed in $scCO_2$ was generally less affected. Interestingly, the raw fabrics displayed a decrease in strength as the gas flow rate and ozonation time increased.

When the tensile strength of fabrics pre-treated for 15 minutes at 5 L/min and 10 L/min gas flow rates after dyeing in supercritical carbon dioxide medium was examined, there was a decrease in strength when the gas flow rate increased. The same situation is observed at both gas flow rates at a processing time of 45 minutes. A 10.8% decrease in the strength of pre-treated fabrics dyed in a $ScCO_2$ medium at a gas flow rate of 5 L/min was observed when the processing time increased, and in addition, a decrease of 10.1% was observed when the processing time increased at a gas flow rate of 10 L/min.

Significantly, the strength values of waterless-dyed fabrics surpassed those of conventionally dyed fabrics. Additionally, pre-treatment with ozone was found to decrease the strength values of fabrics dyed in $scCO_2$. This effect was further supported by SEM images in Figure 7, which demonstrated that samples treated at a 10 L/min ozone gas flow rate (e) exhibited lower strength values due to more surface deformation when compared to those treated with a 5 L/min ozone gas flow rate.

In summary, the research concluded that dyeing in an $scCO_2$ waterless medium yielded high strength values compared to conventional dyeing methods, offering valuable insights into the impact of different dyeing processes on fabric strength.

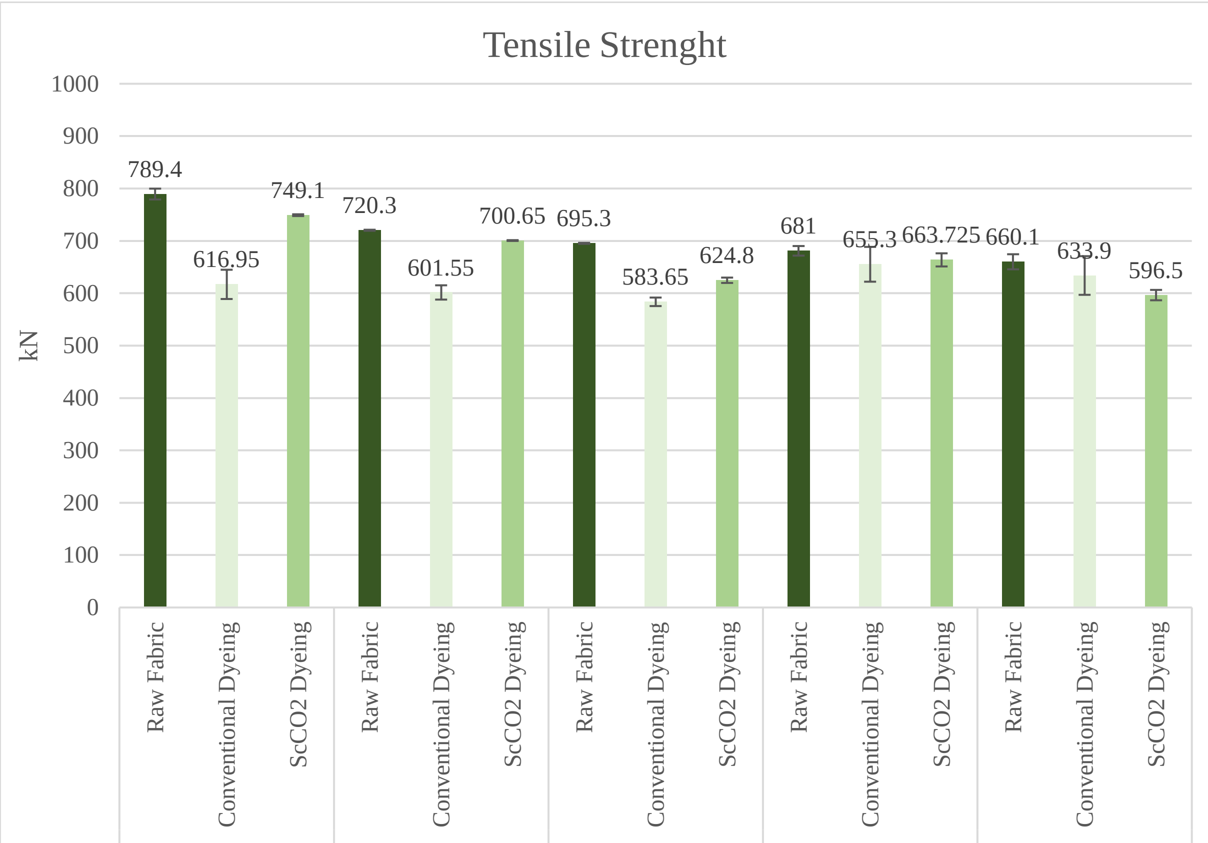


Figure 8. Tensile strength values of the fabrics

IV. CONCLUSIONS

Environmental policies addressing the increasing impact of global warming primarily focus on water-related issues. Consequently, there is growing interest in waterless operations. The most innovative aspect of this study is that both surface modification and dyeing processes were conducted without water. According to the test results:

1. Ozone gas changed the surface and structure of polyester fiber.
2. When the ozone gas flow rate increases, the hydrophilicity values also increase as the amorphous region ratio increases.
3. Since the polymer swells in the supercritical carbon dioxide medium and the amorphous region ratio increases, the fabrics are dyed darker.
4. It has been observed that the strength values decrease as the ozonation time increases.

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