#### **REGULAR ARTICLE**



# Assessing the Washing Fastness and Environmental Impact of Various Reduction Clearing Processes on High-Performance Polyetherimide (PEI) Fabrics

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#### Abstract

PEI fibers are considered a high-performance type of fiber, known for their exceptional heat resistance and mechanical strength. These fibers, made of polyetherimide (PEI) polymer, have a completely amorphous structure, and can be dyed with disperse dyes, similar to how polyester fibers are dyed. This study focused on the reduction clearing process after dyeing PEI fibers with disperse dyes and examined various clearing agents used in both alkaline and acidic conditions, assessing how different concentrations, temperatures, and durations of reduction clearing processes affected the PEI samples. The main goal was to find the best reduction clearing parameters for disperse-dyed PEI samples, taking into account environmental impact, fabric durability, and color fastness. Analytical techniques like chemical oxygen demand (COD), biological oxygen demand (BOD<sub>5</sub>), total organic carbon (TOC), and carbon footprint analysis were used for evaluation. The study revealed that most disperse-dyed PEI fibers require a reduction clearing process, with few exceptions. It is important to note that clearing with sulfuric acid-based agents or in acidic conditions may slightly reduce the fiber's strength. On the other hand, the study identified that utilizing a solution containing 2 g/l sodium dithionite and 4 g/l NaOH for 30 min at 70 °C resulted in the least strength loss. Similarly, when considering the environmental impact, this same reduction clearing process was found to have the least detrimental effects based on COD, BOD<sub>5</sub>, TOC, and carbon footprint analyses. The data collection process was thoroughly documented throughout the study.

Keywords Reduction clearing · Polyetherimide · PEI · Dyeing · Sustainability · Environmental impact · Carbon footprint

# 1 Introduction

Notable for its extraordinary amalgamation of mechanical, thermal, and chemical characteristics, polyetherimide (PEI) stands out as an exceptional high-performance thermoplastic [1, 2]. The polymer's repeating unit is molecularly expressed as  $C_{37}H_{14}O_6N_2$ , with a molecular weight of 592 g/mol and glass transition temperature (Tg) at 215–219 °C [1, 3–7]. PEI fibers do not have an exact melting point due to its amorphous structure. PEI fibers have a moderate tensile strength ranging from 0.25 to 0.26 N/Tex and variable elongation

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<sup>1</sup> Textile Engineering Department, Faculty of Engineering, Pamukkale University, 20160 Denizli, Turkey at break values between 40 and 80% [3, 4, 8–10]. The versatility of PEI fibers is further underscored by several key attributes, including high thermal stability and mechanical strength, alongside resistance to chemicals, such as diluted acids, salts, and oxidants [3, 11]. Their intrinsic flame retardance and low emission of toxic fumes when exposed to fire are particularly noteworthy [10]. Additionally, these fibers offer dimensional stability, attributed to their low coefficients of thermal expansion and moisture absorption, superior electrical insulating capabilities, and resilience against ultraviolet light and ionizing radiation [12, 13]. Given these attributes, PEI fibers are invaluable for a range of demanding applications across various sectors, including aerospace, automotive, electronics, chemical processing, oil and gas, and areas requiring stringent fire safety measures, such as public transport and construction [5, 12, 14].

When it comes to color application on PEI fibers, they are generally considered easy to dye among high-performance fibers [8, 14, 15]. The primary coloring technique employs disperse dyes, commonly used for polyester and nylon [8, 15]. However, achieving optimal color saturation and stability requires dyeing under high temperature and pressure conditions.

Considering the rising application of high-performance fibers in innovative, functional textiles, a thorough study on the dyeing characteristics of PEI fibers will benefit both academia and industry.

Despite the widespread interest and applicability of PEI fibers, there is a surprising dearth of academic research on their dyeing processes. Existing information is often restricted to commercial brochures and patents, offering limited scientific insight. This scarcity of peer-reviewed studies on the subject necessitates a deeper academic inquiry [16].

Regrettably, academic investigations regarding PEI fiber dyeing are scarce, mostly limited to commercial materials and patents. One such patent by Kobayashi et al. explored dyeing conditions using C.I. Disperse Blue 56 at two different temperatures, 155 °C and 140 °C, using a concentration of 6% owf dyestuff and disperse dyeing auxiliaries (17% owf) for 60 min. The dyed PEI fibers were then subjected to a reduction clearing process using 2 g/l sodium carbonate and 2 g/l sodium hydrosulfide at 80 °C for 20 min [16]. The results of the study showed that the dyeing process carried out at 155 °C for 60 min yielded a higher K/S value of 15.8, while the dyeing process carried out at 140 °C for the same duration produced a lower K/S value of 4.8. The washing fastness was evaluated as 4-5 for both processes, while the light fastness was evaluated as 5 for the 155 °C process and 1 for the 140 °C process. Overall, the study suggests that PEI fibers can be successfully dyed using C.I. Disperse Blue 56, and that higher dyeing temperatures can result in better colorfastness properties [16].

Factors, such as dye concentration, temperature, duration, pH, and additives like surfactants and carriers, affect the dyeing outcome. Manufacturer Kuraray recommends dyeing procedure that includes specific conditions and ingredients (0.7–5.0% owf disperse dye, 1 g/l pH buffer, 5% owf UV absorber, 4% owf carrier, liquor ratio 1/70–20 at 130 °C and 60 min), with a warning on potential fiber damage from certain carriers [17]. In addition, although the use of carriers is recommended in cases where dark colors and high color fastness are required, it is stated that some carrier substances may be harmful to the fibers and may damage the mechanical and physical characteristics of the fibers.

The choice of reduction clearing agents and the specific conditions under which they are used are pivotal in the textile dyeing industry, particularly for enhancing washing fastness and eliminating residual or unbound dye on the fabric [18–21]. Among the numerous reducing substances employed in textile dyeing, sodium dithionite stands out as an effective agent for eliminating unbound dye and boosting the washing fastness level [19, 22–25]. However, it generates sulfur-based byproducts like sulfite, sulfate, and thiosulfate, which can be toxic and contribute to oxygen reduction in water bodies, thus escalating the chemical oxygen demand [25]. Additionally, these waste materials can be corrosive, leading to infrastructure damage in waste disposal systems. Sodium dithionite is also unstable in alkaline conditions and at elevated temperatures, which necessitates using an excess amount to counterbalance any loss due to oxidation [22, 24]. Moreover, transitioning from the acidic dyeing phase to highly alkaline conditions for clearing is followed by a neutralization step, adds complexity, time, and cost to the whole dyeing operation [24, 26].

Thiourea dioxide (TUDO) is another strong reducing agent that serves as an alternative to sodium dithionite. It has the advantage of being more stable but is generally more expensive [24, 26, 27]. There are also many alternative commercial reducing agents. Alkyl polyglycol ether and organic reducing agent are one of them and often used as leveling agents along with their reducing properties. They are generally less aggressive than sodium dithionite and thiourea dioxide and may be used for delicate fibers. Glycose derivatives are milder reducing agents and may be used in conjunction with other agents to fine-tune the reduction clearing process [21]. Synergistic mixtures of anionic dispersants and adols are complex mixtures. They can offer a balance of dye dispersion and reduction. They may improve the overall quality of the dyeing by ensuring uniform color and better fastness properties. Sulfonic acid derivatives are generally mild reducing agents and may be used for sensitive fibers or dyes [28]. They are often used when a mild reduction process is desirable to maintain the quality of the fiber. Although resin derivatives are less commonly used, these specialized agents can be employed for specific dyeing requirements and may offer unique advantages in terms of color yield or fastness.

Choosing the appropriate reducing agent is crucial, depending on the fiber type, dye, and desired properties of the dyed material. Making an incorrect selection or applying it improperly can have a negative impact on both the fabric's quality and fastness properties. Additionally, it is important to consider the reduction clearing conditions, including factors like time and temperature. It is essential to ensure that the clearing temperature doesn't exceed the glass transition temperature of the fiber to prevent the stripping of dye from within the fiber structure [28].

The majority of disperse dyes contain the azo group, -N = N-, which can be easily transformed into two amino groups (NH<sub>2</sub> and H<sub>2</sub>N) through treatment with a reducing agent. This property makes them well-suited for reduction clearing as the resulting amino residues are essentially colorless unless intentionally oxidized to produce entirely different products. In some of the brighter blue, pink, and red disperse dyes, there is another chromophore, anthraquinone, though it's more expensive and challenging to reduce. Interestingly, it remains intact during normal reductive clearing processes [19, 24].

This manuscript aims to bridge this knowledge gap by providing a comprehensive analysis of the reduction clearing processes employed in disperse-dyed PEI fabrics. Our research delves into the effectiveness of various reducing reduction clearing agents, their optimal operational parameters, and their impact on both the washing fastness and tensile properties of the dyed fibers. Akey component of this study is the environmental aspect, where wastewater metrics, such as Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD<sub>5</sub>), Total Organic Carbon (TOC), and Carbon footprint, are analyzed to understand the ecological implications of the reduction clearing processes after dyeing process.

# 2 Experimental Methods

#### 2.1 Materials

The present investigation utilized 330 dtex and 150 filament polyetherimide (PEI) fibers obtained from Kuraray Co. Ltd. to fabricate single jersey knitted fabrics. Reduction clearing agents having different chemical structures, such as sodium dithionite, thiourea dioxide (Tudo), glycose derivative, sulfonic acid derivative, resin derivative, synergistic mixture of anionic dispersants and adols, and alkyl polyglycol ether and organic reducing agent, were utilized in this study (Table 2). Reduction clearing agents were obtained from Orkim, Bozetto, and CHT. Additionally, an investigation was conducted to explore the effects of reduction clearing procedures on the dyeing of samples containing a variety of dyes, which differed in terms of color, molecular size, and classification of dyestuff. Table 1 provides a complete review of the dyestuffs that are employed.

 Table 2
 Reduction clearing agents and working conditions used to determine the ideal reduction clearing conditions for PEI fibers

	Reduction clearing agents	Working condi- tions
R1	Sodium dithionite	Alkali
R2	Thiourea dioxide (TUDO)	Alkali
R3	Alkyl polyglycol ether and organic reducing agent	Alkali
R4	Glycose derivative	Alkali
R5	Synergistic mixture of anionic dispersants and adols	Alkali
R6	Sulfonic acid derivative	Alkali
R7	Sulfonic acid derivative	Acidic
R8	Resin derivative	Acidic
Temperature (°C)	50, 60, 70, 80, 90	
Time (min)	15, 30	
Reducing agent concentration	2 g/l	
Alkali concentration	2 g/l sodium carbonate	
	4 g/l sodium hydroxide	
Acid concentration	2 g/l acetic acid	
	4 g/l acetic acid	
рН	3, 10, 13	

### 2.2 Scouring Process

Before dyeing process, knitted fabrics made from polyetherimide (PEI) fibers underwent a scouring procedure to remove possible impurities and oils embedded in the material. This cleaning step employed a solution including 1 g/l of Kieralon Jet B, a nonionic surfactant from BASF, and 1 g/l of sodium carbonate, heated to 60 °C and maintained for a 15-min period. Following this, the fabric was rigorously washed first with warm water and then with cold water, before being left to air-dry at room temperature.

Table 1	Utilized dvestuffs	
lable I	Utilized dyestuffs	

Company	Commercial name	Color Index (C.I.)	Class
Dystar	Dianix® Classic Rubine SE-FG	Disperse Red 73	Azo
Setaş	Red EC2G	Disperse Red 167	Azo
Setaş	Blue ECGS	Disperse Blue 79	Azo
Setaş	Yellow brown-2r	Disperse Orange 30	Azo
Setaş	Red ELF	Disperse Red 86	Anthraquinoid
CHT	Bemacron Blue EFBL 150	Disperse Blue 56	Anthraquinoid
CHT	Bemacron yellow E3GL	Disperse Yellow 54	Quinoline
Setaş	Yellow ELF	Disperse Yellow 42	Nitrodiphenylamine
LJ.SpecialitiesLtd	Lumacron Red CBN	Disperse Red 356	Benzodifuran

## 2.3 Dyeing Process

The coloration procedure was executed using an ATAC-IR HT lab-scale dyeing machine, operating at 130 °C for 60 min. The ratio of liquor to fabric was maintained at 1/20, with the addition of 4% owf dye, 4% carrier, and 1 g/l of anionic dispersion agents. The dispersion agent utilized in this study was Setamol BL, a product manufactured by BASF. Furthermore, the study incorporated Perigen EC, an ecofriendly carrier developed by Textilchemie Dr. Petry GmbH Dr. Petri. Perigen EC, an environmentally harmless dyeing accelerator, is suitable for dyeing polyester, triacetate, and their mixtures with wool and cotton. This dyeing accelerator is composed of aromatic esters and fatty alcohol polyglycol ether, and it is nonionic.

The pH of the dye bath was adjusted to pH 4.5 by carefully manipulating the quantities of acetic acid and sodium carbonate. After the dyeing process, the fabric was subjected to a series of rinsing steps using warm and cold overflow for a duration of 5 min. Following rinsing, the cloth was left to dry under ambient circumstances. It's worth mentioning that the dyeing specifications suggested by fiber producer Kuraray were followed, given the limited existing research on PEI fiber dyeing.

The study was structured into three separate phases, each using a variety of disperse dyes categorized by different properties, as specified in Table 1. In the initial phase, a specific disperse dye (C.I. Disperse Red 73) was selected to closely scrutinize the impact of reduction clearing agents. The second phase delved into how disperse dyes of varying colors, structures, and molecular weights affected the reduction clearing step. In the concluding phase, C.I. Disperse Red 73 was again employed as a benchmark to systematically assess other factors like fabric strength, various color fastness properties, etc., subsequent to the reduction clearing treatment.

### 2.4 Reduction Clearing Processes

#### 2.4.1 1st Step

In the initial phase of the research, a particular disperse dye known as C.I. Disperse Red 73, part of the azo dye family was selected for the coloration of PEI fiber fabrics. Following the dyeing process, various reduction clearing processes were applied to reduce excess dye and improve the colorfastness of the dyed fabrics. A total of eight different agents for reduction clearing were tested, each designed to work in either an acidic or alkaline setting. The selection of these agents was guided by their compatibility with PEI fibers and their efficacy in the clearing process. The working conditions and the manufacturer recommendations for each reduction clearing agent were considered to optimize the reduction clearing process. Therefore, an experimental design was developed to identify the most effective reduction clearing for PEI fibers (Table 2).

Various processing durations (15 and 30 min) and temperature ranges (50–90 °C) were experimented with, maintaining a constant reduction clearing agent concentration of 2 g/l. Furthermore, the study carefully examined pH levels suitable for each agent's working environment, covering pH 3, pH 10, and pH 13. Given that the only available information about PEI fiber's resilience in alkaline and acidic conditions comes from corporate literature, two different alkali and acid concentrations were explored. For alkaline settings, 2 g/l sodium carbonate or 4 g/l NaOH was tested, while 2 g/l or 4 g/l acetic acid was evaluated for acidic environments.

After examining changes in washing fastness subsequent to the initial reduction clearing steps, the best operating conditions were determined for each of the eight unique reduction clearing agents. The most effective processing temperatures were pinpointed for every agent under four separate conditions—specifically, at alkali/acid concentrations of 2 g/l and 4 g/l for durations of 15 and 30 min—to optimize performance.

#### 2.4.2 2nd Step

The second step of the study involved investigating the effects of different disperse dyes on the reduction clearing process. These dyes were selected based on variations in color, structure, and molecular weight. After dyeing the PEI fiber samples (at 130 °C for 60 min. at pH 4.5 with 4% owf dye and 4% carrier, LR: 1/30), the samples were subjected to treatment using eight distinct reduction clearing agents. The experimental procedures were carried out according to four predetermined circumstances, which involved treatment durations of 15 and 30 min, as well as alkali or acid concentrations of 2 g/l and 4 g/l. These settings were created during the initial phase of the investigation. Following that, an examination was conducted on the levels of washing fastness. The identification of the most effective reduction clearing agents for washing fastness across all dye groups, as well as the determination of the suggested processing parameters for their application, was based on the outputs of this second stage.

#### 2.4.3 3rd Step

In the final stage of the study, the dyeing of PEI fibers was performed using a single type of dyestuff, namely C.I. Disperse Red 73, which was also consistently used in the initial step. Following the dyeing, the colored PEI fiber materials underwent reduction clearing treatments, based on the best practices identified in the second stage, to pinpoint the most effective reduction clearing agents and processing parameters. After the reduction clearing procedures, various attributes, such as fastness levels, and tensile characteristics, were rigorously assessed. To gage the environmental impact of the experimental process, wastewater indicators like Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD<sub>5</sub>), and Total Organic Carbon (TOC) were also scrutinized.

#### 2.5 Evaluation of Tensile Properties of PEI Samples

In order to examine the influence of different reduction clearing processes on the strength of PEI samples, tensile strength test was carried out. The Tinius Olsen H10KT benchtop test apparatus was employed to conduct the experiments, following the guidelines outlined in the ASTM D 3822 standard for single fibers [29, 30].

#### 2.6 Evaluation of Fastness Properties

The PEI fiber fabric samples, which had been dyed using a disperse dyes, were tested for their resistance to washing using a home washing procedure outlined in the ISO 105:C06 (C06/B2S) Test Standard. The testing was conducted using a Rotawash machine at a temperature of 50 °C, with the inclusion of sodium perborate. The determination of both alkaline and acid perspiration fastnesses was conducted in accordance with the ISO 105-E04 standard, which measures color fastness to perspiration. In addition, dry and wet rub fastness tests were conducted in accordance with the ISO 105: X12 standard. The evaluation of water fastness and the sea water fastness tests were conducted in accordance with ISO 105-E01 and ISO 105-E02, respectively. The sublimation fastness tests were conducted according to the ISO 105:P01 procedure, utilizing the SDL Scorch Tester M247B apparatus at a temperature of 180 °C for a duration of 30 s.

Following a series of fastness tests, the extent of staining on multifiber fabrics was assessed using ISO gray scale ratings. This assessment was done in accordance with ISO 105-A03 Tests for color fastness Part A3 gray scale for assessing staining level of multifiber using DataColor SpectraFlash 600 Spectrophotometer. The colorimetric data were calculated using ISO/CIE 11664–1 (CIE 1964) supplementary standard colorimetric system with illuminant D65 and a 10° standard observer.

#### 2.7 Assessment of Chemical Oxygen Demand (COD)

The chemical oxygen demand (COD) of various reduction clearing wastewater samples was evaluated using the standard titrimetric approach, as outlined in the 19th edition of the APHA's "Standard Methods 5220 C: Closed Reflux, Titrimetric Method". In this process, 3 mL of each wastewater sample was introduced into COD test kits produced by Merck. These samples were then subjected to decomposition in a WTW CR 3200 thermoreactor for 2 h at a temperature of 148 °C. Following this period, the samples were cooled to room temperature. The COD levels were then quantified using a Merck Nova 60 photometer, with the results expressed in milligrams per liter (mg/L). The average COD values from these experiments are also reported in mg/L.

#### 2.8 Biological Oxygen Demand (BOD<sub>5</sub>) Analysis

To determine the biochemical oxygen demand (BOD) of both control and reduction clearing wastewater samples, a five-day testing process at 20 °C was employed, known as BOD<sub>5</sub>. This was conducted using the manometric method, with the aid of the OxiTop pressure sensor from WTW.

# 2.9 Total Organic Carbon (TOC) Analysis

The total organic carbon (TOC) content in the samples was analyzed utilizing a Shimadzu TOC-Vcph TOC analyzer (TNM-1/SSM-5000A model). For this analysis, control and reduction clearing wastewater samples were initially filtered through a 22  $\mu$ m filter and then diluted 20-fold with pure water. The average of three separate readings was recorded as the final TOC value for each sample.

#### 2.9.1 Environmental Impacts Evaluation

In order to evaluate the environmental effects of operations based on the specified process conditions, a unique piece of software known as the "Ruco-Logic-eCO<sub>2</sub> meter" was employed. In addition, the program not only allowed for the creation of carbon footprints but also made it possible to estimate fuel, energy, labor, water, chemical, and other costs during pre-production planning in a clearer and more precise way. Using "Ruco-Logic" eCO<sub>2</sub> meter software, calculations and projections were made in the third step of the study while maintaining constants for machine type, capacity (600 kg), liquor ratio (1:30), and the amount of product to be processed (500 kg) throughout the seven indicated process conditions.

Here, the liquor ratios are high because the reduction clearing procedures used in this investigation were limited to a laboratory setting. This means that a high water consumption will also translate into a high energy and fuel requirement for heating. On the other hand, substantially shorter liquor ratios are favored for minimizing reduction clearing procedures used in large-scale, real-time production. Because they would be irrational, the cost estimates of the procedures we carried out in the lab that were determined utilizing the aforementioned software were disregarded. To reduce the influence of high liquor ratios on environmental impacts, attention was directed into the calculations that provide the environmental footprint. Specifically, the comparison of the suggested reduction clearing conditions with each other was considered, rather than the obtained footprint values.

# **3** Results and Discussion

Reduction clearing agents are abbreviated as R1, R2, R3, R4, R5, R6, R7, and R8 as shown in Table 2 in the following parts of the study. R1-R6 reduction clearing agents work in alkaline environment. R6 is a sulfuric acid-derived reduction clearing agent that functions in alkaline settings, while R7 and R8 operate effectively in acidic conditions. For all processes, a consistent concentration of 2 g/l for the reducing wash agent was set for fair comparison. Two distinct pH levels were selected based on each reducing agent's functional environment. In experiments using alkaline-compatible agents (R1-R6), treatments with 2 g/l sodium carbonate were labeled as R1<sub>2C</sub>-R6<sub>2C</sub>, and those using 4 g/l sodium hydroxide were termed R14H-R64H. For the acidic-compatible agents R7 and R8, varying pH levels were achieved using either 2 g/l or 4 g/l acetic acid. Throughout the remainder of this study, treatments with 2 g/l acetic acid were abbreviated as R7<sub>2A</sub> and R8<sub>2A</sub>, while those with 4 g/l acetic acid were labeled as  $R7_{4A}$  and  $R8_{4A}$ .

### 3.1 First Step Results

These washing fastness results are thoroughly outlined in Table 3. They were assessed according to the ISO 105-A03 gray scale, which measures the level of staining on multifiber after undergoing reduction clearing processes with

various agents under different conditions of temperature. time, and pH. Significant staining was noted particularly on the polyamide component of the multifiber, which was consistently used throughout the study. Consequently, for simplicity and to minimize complexity, the staining values of polyamide were exclusively used for all comparative analyses in the research. The control samples, which were dyed with C.I. Disperse Red 73 but not subjected to reduction clearing, exhibited a washing fastness level of 1-2 on the gray scale for stained multifiber. The polyamide component showed the highest degree of staining in the multifiber; thus, it was selected as the focus for comparison to streamline the study and avoid potential complexities. This observation underscores the necessity of an effective reduction clearing process to enhance the washing fastness of PEI samples dyed with C.I. Disperse Red 73.

Upon reviewing the washing fastness levels presented in Table 3, it was noted that the minimum score of 3 Gy scale rating was achieved following the reduction clearing treatments conducted with  $R4_{2C}$  at temperatures of 50 and 60 °C, for both 15- and 30-min durations. Washing fastness values of 4 and above were observed in the majority of the reduction clearing processes implemented at this stage.

As treatment time extends from 15 to 30 min, it is possible to say there is a slight increase in washing fastness values for most of the reducing agents. There is also slight improvement in washing fastness values with increased alkalinity, from 2 g/L of sodium carbonate to 4 g/L NaOH, especially in conjunction with agents like sodium dithionite, thiourea dioxide, and the sulfonic acid derivatives. Temperature is an effective parameter on almost all reduction clearing agents, and there is an improvement in washing fastness values with increasing temperature. Except for R8, excellent washing

 
 Table 3
 Washing fastness values of stained multifiber (polyamide results chosen for comparison), after reduction clearing processes of dispersedyed PEI fabrics

		Washi	ing fasti	ness val	ues <sup>a</sup>												
Time (min)	Temp. (°C)	2 g/l s	odium	carbona	ite			2 g/l a acid	cetic	4 g/l s	odium l	hydroxi	de			4 g/l a acid	cetic
		R1 <sub>2C</sub>	R2 <sub>2C</sub>	R3 <sub>2C</sub>	R4 <sub>2C</sub>	R5 <sub>2C</sub>	R6 <sub>2C</sub>	R7 <sub>2A</sub>	R8 <sub>2A</sub>	$R1_{4H}$	$R2_{4H}$	$R3_{4H}$	$\mathbf{R4}_{4\mathrm{H}}$	$R5_{4\mathrm{H}}$	$R6_{4H}$	R7 <sub>4A</sub>	$R8_{4A}$
15	50	4–5	4– <u>5</u>	3–4	3	3–4	4–5	<u>4–</u> 5	4	4–5	4– <u>5</u>	4	4	3-4	4– <u>5</u>	4_5	4
15	60	4– <u>5</u>	4– <u>5</u>	4	4	3–4	4– <u>5</u>	4– <u>5</u>	4	4– <u>5</u>	4– <u>5</u>	4– <u>5</u>	<u>4–</u> 5	<u>4–</u> 5	4– <u>5</u>	4– <u>5</u>	4
15	70	4– <u>5</u>	5	4	4	3– <u>4</u>	4– <u>5</u>	4– <u>5</u>	<u>4–</u> 5	4– <u>5</u>	5	4– <u>5</u>	4– <u>5</u>	4– <u>5</u>	4– <u>5</u>	5	4
15	80	5	5	5	4–5	4	5	5	<u>4–</u> 5	5	5	5	5	5	5	5	4
15	90	5	5	5	5	5	5	5	<u>4–</u> 5	5	5	5	5	5	5	5	4–5
30	50	4–5	4– <u>5</u>	3–4	3	4	4–5	4–5	4	4–5	4– <u>5</u>	<u>4–</u> 5	4	3–4	4– <u>5</u>	4–5	4
30	60	4– <u>5</u>	4– <u>5</u>	3– <u>4</u>	3	4	4– <u>5</u>	4– <u>5</u>	<u>4–</u> 5	4– <u>5</u>	4– <u>5</u>	4–5	4–5	<u>4–</u> 5	5	4– <u>5</u>	<u>4–</u> 5
30	70	5	5	<u>4–</u> 5	3–4	4–5	5	4– <u>5</u>	4–5	5	5	4– <u>5</u>	5	4–5	5	5	<u>4–</u> 5
30	80	5	5	5	5	5	5	5	4–5	5	5	5	5	5	5	5	4–5
30	90	5	5	5	5	5	5	5	4	5	5	5	5	5	5	5	4

<sup>a</sup>The lower line in the evaluation means a sensitivity of 0.25 points. For example, 4/5 is better than 4/5 and less than 5 Gy scale rating

fastness values with 5 Gy scale rating were recorded for all reduction clearing agents in 80 °C and 90 °C processes. It was observed that the washing fastness value remained constant between 4 and 4.25 in the processes carried out with R8 in varying temperature, time, and acid concentrations.

In order to facilitate the second part of the investigation, four distinct process conditions were identified for each type of reducing agent, while considering the relevant variables as outlined in Table 4. The aforementioned determined conditions will serve as a point of reference in the context of two-step assessments.

# 3.2 Second Step Results: Investigation of the Impact of Different Types of Dyes on the Reduction Clearing Processes

The dyeing process of textiles, especially with disperse dyes, is greatly influenced by several factors related to the dyes utilized. Molecular size, color, and structure of the dye play significant roles in determining how effectively the dye can penetrate the fibers and how stable and vibrant the resulting color will be. So, in the second stage of the investigation, it was delved into the impact of diverse disperse dyes on the reduction clearing processes.

Table 5 displays dyes of varied colors, molecular weights, and structures, and dyed PEI fabric appearances that were obtained by dyeing at 130 °C for 60 min. The dyeing was conducted at a pH of 4.5, utilizing 4% owf dye, 4% carrier, and an LR of 1/30. The resulting color shades and the K/S

 Table 4
 Recommended conditions for reduction clearing processes of disperse-dyed PEI fabrics as a result of initial stage

1 2		e	
		nmended reducti sses at the end of	0
R1: Sodium dithionite	$R1_{2C}$	15 min., 80 °C	30 min., 70 °C
	$R1_{4H}$	15 min., 80 °C	30 min., 70 °C
R2: Thiourea dioxide	$R2_{2C}$	15 min., 70 °C	30 min., 70 °C
(TUDO)	$R2_{4H}$	15 min., 70 °C	30 min., 70 °C
R3: Alkyl polyglycol ether	R3 <sub>2C</sub>	15 min., 80 °C	30 min., 80 °C
and organic reducing agent	$R3_{4H}$	15 min., 80 °C	30 min., 80 °C
R4: Glycose derivative	R4 <sub>2C</sub>	15 min., 90 °C	30 min., 80 °C
	$R4_{4H}$	15 min., 80 °C	30 min., 80 °C
R5: Synergistic mixture of	R5 <sub>2C</sub>	15 min., 90 °C	30 min., 80 °C
anionic dispersants and adols	$R5_{4H}$	15 min., 80 °C	30 min., 80 °C
R6: Sulfonic acid derivative	R6 <sub>2C</sub>	15 min., 80 °C	30 min., 70 °C
	$R6_{4H}$	15 min., 80 °C	30 min., 60 °C
R7: Sulfonic acid derivative	R7 <sub>2A</sub>	15 min., 80 °C	30 min., 80 °C
	$R7_{4A}$	15 min., 70 °C	30 min., 70 °C
R8: Resin derivative	R8 <sub>2A</sub>	15 min., 90 °C	30 min., 80 °C
	$R8_{4A}$	15 min., 90 °C	30 min., 80 °C

values from these dyeing activities are detailed in Table 5. Post the dyeing of the PEI fiber specimens using the disperse dyes mentioned in Table 5, these specimens underwent reduction clearing treatments with eight distinct reducing clearing agents as delineated in the preceding procedural step (Table 4).

Generally, dyes with lower molecular weights exhibit a higher dyeing rate, whereas those with higher molecular weights display a slower rate [18, 31]. Smaller dye molecules have greater ease in penetrating fibers compared to their larger counterparts. This characteristic is particularly significant for polyester and other synthetic fibers with tightly knit polymer structures. Larger molecules may offer enhanced washing fastness and light fastness scores due to the stronger van der Waals forces they exert within the fiber structure. Conversely, smaller molecules can be more prone to washing out, especially if their potential bonding is not robust [18, 19].

Azo dyes represent the most prominent category, constituting over half of the total commercially available disperse dyes globally. The azo group is an inherently potent chromophore, boasting high tinting strength when compared to the relatively weak anthraquinone chromogens. Typically, azo dyes are two to three times stronger in tinting strength. Their simplicity and ease of production make them a preferred choice, in addition to their competitive pricing. Azo dyes encompass a wide spectrum of colors, ranging from yellow to blue-green hues [18]. The ability of a dye to form chemical bonds with fibers depends on its molecular structure. For instance, azo dyes may engage in different interactions with fibers compared to anthraquinoid dyes. Besides, anthraquinoid dyes have significantly contributed to the violet and blue shade range, delivering vibrant dyeing results and generally excellent lightfastness without dye stability issues during the dyeing process [18, 20, 32, 33].

The reduction clearing process aims to eliminate unfixed dye particles from the fiber's surface. The chemical structure of dyes plays a crucial role in determining their reactivity and solubility during this process. Some dye structures may be more susceptible to reduction and wash-off, while others may exhibit greater resistance [18, 34].

When the values provided in Table 6 are summarized, it is evident that, in the reduction clearing processes carried out after the dyeing procedures with various disperse dyes having different chemical structures and molecular weights (as determined in the initial step), commercially acceptable washing fastness values (4 and above) were generally achieved under all reduction clearing agents and process conditions. It is worth noting that even in cases where no specific reduction clearing process was applied, particularly for samples dyed with C.I. Disperse Blue 56, C.I. Disperse Orange 30, C.I. Disperse Yellow 42, and C.I. Disperse Red 356, good to excellent washing fastness values of 4, 4, 4–5,

Color Index	Dye Class	Chemical Structure of Disperse Dye	Molecular Weight (g/mol)	Color Strength (K/S)	Dyed Fabric Appearance	Color Index	Dye Class	Chemical Structure of Disperse Dye	Molecular Weight (g/mol)	Color Strength (K/S)	Dyed Fabric Appearance
Disperse Red 73ª	Azo		348	28.2		Disperse Orange 30 <sup>f</sup>	Azo	°,	450	23.4	
Disperse Blue 79 <sup>b</sup>	Azo		625	29.7		Disperse Red 167 <sup>g</sup>	Azo		520	27.8	
Disperse Red 86°	Anthraquinoide		423	19.4		Disperse Blue 56 <sup>h</sup>	Anthrquinoide		305	22.1	
Disperse Yellow 54 <sup>d</sup>	Quinoline		289	24.2		Disperse Yellow 42 <sup>i</sup>	Nitrodiphenylamine		369	14.4	
Disperse Red 356°	Benzodifuranes		412	7.7			I	Disperse Red 73 <sup>a</sup> (35) Disperse Blue 79 <sup>b</sup> (36) Disperse Red 86 <sup>e</sup> (32) Disperse Yellow 54 <sup>d</sup> (37) Disperse Red 356 <sup>e</sup> (32)	Disp Disp	e Orange 30 erse Red 16 berse Blue 5 rse Yellow	7 <sup>g</sup> (40) 6 <sup>h</sup> (41)

Table 5 Disperse dyes used in the second stage of the study and their respective dyed PEI fabric appearances

and 5, respectively, were obtained as a result of thorough rinsing.

Although there are many parameters that enable the fiber to adhere to the dye structure, the molecular size of the dye has a great impact among these parameters. As can be understood from the studies on polyester in the literature, as the molecular size of the dyestuff enhances and the substantivity increases, better washing fastness values are obtained after dyeing and reduction clearing wash [35].

While the washing fastness outcomes from the reduction clearing of dyed PEI fabrics were largely satisfactory, there was no evident correlation observed. Nonetheless, it was highlighted that the samples dyed with C.I. Disperse Blue 79, which has the largest molecular azo group, almost uniformly displayed superior washing fastness after undergoing reduction clearing. On the other hand, it was found that the samples dyed with C.I. Disperse Yellow 54, belonging to the quinoline group with the smallest molecular scale, showed comparatively lower washing fastness after reduction clearing treatments than fabrics dyed with other colorants.

Specific conditions, such as the type and amount of additive, duration, and temperature may also influence the final color appearance of samples after reduction clearing processes. Specifically, when reduction clearing procedures are carried out at temperatures near the point where fibers in the fabric structure turn into glass transition  $(T_g)$  or the temperature at which they are dyed, it can result in the removal of the dye from the fiber structure [14]. For instance, reduction clearing processes conducted at temperatures near or above the glass transition temperature of polylactic acid fibers have led to significant dye loss and a decrease in strength [22, 23]. Nevertheless, the sensitivity of fibers to alkaline or acidic conditions can also result in the loss of dye and strength in the fiber structure and worse washing fastness evaluation [18].

The washing fastness values presented in Table 6 were used to conduct more effective examinations in the subsequent processing step. Gray scale staining evaluations were performed using a DataColor spectrophotometer, and multifiber contamination values were calculated numerically. Additionally, the average washing fastness value for each reduction clearing process in this step was determined by averaging the washing fastness values across nine different disperse dyestuff groups.

Table 6 clearly demonstrates that the reduction clearing processes resulted in very high washing fastness values, the majority of which exceeded the commercially acceptable threshold. Consequently, to conduct a more precise

Dyea + Keauction cleared with Keauction clearing	Keduction clearing		Used disp	Used disperse dyes (C.I. Disperse)	(nemder of							
below process type	time (min)	temp. (°C)	Red 73	Red 167	Blue 56	Orange 30	Blue 79	Red 86	Yellow 42	Yellow 54	Red 356	Average <sup>a</sup>
			Washing f	Washing fastness values <sup>b</sup>								
R1 <sub>2C</sub>	15	80	5	4–5	5	5	5	5	5	4-5	5	4.89
R1 <sub>4H</sub>	15	80	5	5	5	5	5	5	5	4-5	5	4.97
R2 <sub>2C</sub>	15	70	5	5	45	5	5	5	5	3-4	5	4.78
R2 <sub>4H</sub>	15	70	5	4-5	4-5	5	5	5	5	4-5	5	4.89
$ m R3_{2C}$	15	80	5	4-5	4-5	4-5	5	4-5	5	4-5	5	4.75
$ m R3_{4H}$	15	80	5	4-5	4-5	4-5	5	4-5	5	4-5	5	4.83
$ m R4_{2C}$	15	06	5	4-5	4	4-5	5	4	5	4-5	5	4.61
$ m R4_{4H}$	15	80	5	4-5	4-5	4-5	5	4-5	5	4-5	5	4.78
R5 <sub>2C</sub>	15	06	5	4-5	4	4-5	5	4-5	5	3-4	5	4.64
R5 <sub>4H</sub>	15	80	5	4-5	4-5	5	5	4-5	5	4-5	5	4.83
R6 <sub>2C</sub>	15	80	5	5	4-5	5	5	4-5	5	4	5	4.81
R6 <sub>4H</sub>	15	80	5	5	4-5	5	5	4-5	5	4-5	5	4.89
$R7_{2A}$	15	80	5	5	4	5	5	4-5	4-5	3-4	5	4.69
${ m R7}_{ m 4A}$	15	70	5	5	4	5	5	4-5	5	3-4	5	4.64
$R8_{2A}$	15	06	4-5	4-5	4-5	5	5	4-5	5	4-5	5	4.75
$ m R8_{4A}$	15	06	4-5	4-5	4-5	5	5	4-5	5	4	5	4.69
R1 <sub>2C</sub>	30	70	5	5	5	5	5	5	5	4	5	4.89
R1 <sub>4H</sub>	30	70	5	5	5	5	5	5	5	4-5	5	4.97
R2 <sub>2C</sub>	30	70	5	5	5	5	5	5	5	4	5	4.89
$ m R2_{4H}$	30	70	5	5	5	5	5	5	5	5	5	5.00
R3 <sub>2C</sub>	30	80	5	4-5	5	4-5	4-5	4-5	5	4–5	5	4.83
R3 <sub>4H</sub>	30	80	5	4-5	5	4-5	5	4-5	5	5	5	4.92
$ m R4_{2C}$	30	80	5	4-5	4-5	4-5	4-5	4-5	5	4-5	5	4.64
$ m R4_{4H}$	30	80	5	4-5	4-5	4-5	5	4-5	5	4-5	5	4.83
R5 <sub>2C</sub>	30	80	5	4-5	4-5	5	5	5	5	4-5	5	4.92
R5 <sub>4H</sub>	30	80	5	4-5	4-5	5	5	5	5	5	5	4.94
R6 <sub>2C</sub>	30	70	5	5	5	5	5	5	5	4	5	4.89
R6 <sub>4H</sub>	30	60	5	5	5	5	5	5	5	5	5	5.00
$R7_{2A}$	30	80	5	5	5	5	5	4-5	5	4-5	5	4.86
$R7_{4A}$	30	70	5	5	5	5	5	4-5	5	4-5	5	4.89
R8 <sub>2A</sub>	30	80	4-5	5	5	5	5	5	5	4-5	5	4.89
0	00	00		1								

Dyed + Reduction cleared with Reduction clearing Reduction clearing	Reduction clearing	Reduction clearing	Used disp	Used disperse dyes (C.I. Disperse)	Disperse)							
below process type	time (min)	temp. (°C)	Red 73	Red 167	Blue 56	Orange 30	Blue 79	Red 86	Red 73         Red 167         Blue 56         Orange 30         Blue 79         Red 86         Yellow 42         Yellow 54         Red 356	Yellow 54	Red 356	Average <sup>a</sup>
			Washing f	Washing fastness values <sup>b</sup>								
Control <sup>c</sup>			1-2 2-3	2–3	4	4	3-4	3-4	3-4 3-4 4-5 2-3		5 3.44	3.44
<sup>a</sup> The average washing fastness values of dyed PEI samples with ten different disperse dyes measured with Data color spectrophotometer <sup>b</sup> The lower line in the evaluation means a sensitivity of 0.25 points. For example, $4/5$ is better than $4/5$ and less than 5 Gy scale rating <sup>c</sup> Control: Washing fastness values of stained multifiber (Polyamide 6) for dyed PEI samples without reduction clearing applications, only dyed samples	ss values of dyed Pl titon means a sensit alues of stained mu	EI samples with ten ivity of 0.25 points. Iltifiber (Polyamide	different For exam 6) for dye	disperse dyes ple, 4/Σ is be d PEI sample	s measured v stter than 4/5 es without re	with Data color 5 and less than eduction cleari	<ul> <li>spectropho</li> <li>5 Gy scale 1</li> <li>ng applicati</li> </ul>	tometer ating ons, only dy	ed samples			

Table 6 (continued)

analysis of the results in the next processing step, recommended process conditions were determined based on achieving an average washing fastness value of 4.9 or higher (measurements were performed using a DataColor spectrophotometer).

According to this criterion, the following process conditions were recommended for the third step processes:

- R1(Sodium dithionite) with 4 g/l NaOH at 80 °C for 15 min
- R1(Sodium dithionite) with 4 g/l NaOH at 70 °C for 30 min
- R2 (Thiourea dioxide, TUDO) with 4 g/l NaOH at 70 °C for 30 min
- R5 (Synergistic mixture of anionic dispersants and adols) with 2 g/l NaCOOH at 80 °C for 30 min
- R5 (Synergistic mixture of anionic dispersants and adols) with 4 g/l NaOH at 80 °C for 30 min
- R6 (Sulfonic acid derivative) with 4 g/l NaOH at 60 °C for 30 min
- R8 (Resin derivative) with 4 g/l acetic acid at 80 °C for 30 min

# 3.3 Third Step Results

In the final stage of the study, which marks the third and concluding step, a series of rigorous fastness tests and strength test were conducted under the carefully selected reducing clearing conditions. Furthermore, this phase aimed to identify optimal reduction clearing processes by incorporating various environmental impact assessments and carbon footprint calculations, thus contributing to the assessment of the role of reduction clearing processes in environmentally sustainable production practices.

# 3.3.1 Fastness Properties

During this stage of the research, PEI samples dyed with C.I. Disperse Red 73 were employed for the reduction clearing processes, mirroring the approach adopted in the initial step. All the examined fastness properties (perspiration, water, seawater, washing, and rub fastness) except for sublimation fastness, met commercially accepted standards (Table 7). Surprisingly, even sublimation fastness, unlike many synthetic fibers, exhibited values close to the good range (ranging between 3–4 and 4–5 Gy scale ratings).

Given the proximity of the fastness values, gray scale multifiber contamination assessments were conducted using a DataColor spectrophotometer to enhance sensitivity and identify the most favorable reduction clearing process conditions with respect to fastness properties in this phase. The average of the measured values for all fastness attributes is presented in the table for a more straightforward comparison.

			Fastness evaluations <sup>a</sup>	luations <sup>a</sup>							
Dyed + reduction cleared with below process type	Reduction clear- ing time (min)	Reduction clear- Reduction clear- ing time (min) ing temp. (°C)	Alkali per- spiration	Acidic per- spiration	Water	Sea water	Washing	Sublimation	Wet rub <sup>b</sup> fastness	Dry rub <sup>b</sup> fastness	Average <sup>c</sup>
R1 <sub>4H</sub>	80	15	5	5	5	5	5	3-4	5	5	4.84
$R1_{4H}$	70	30	5	5	5	5	5	3-4	5	5	4.84
$R2_{4H}$	70	30	5	5	5	5	5	3-4	5	5	4.84
R5 <sub>2C</sub>	80	30	<u>4</u> -5	4-5	4	4-5	5	3-4	5	5	4.41
$R5_{4H}$	80	30	5	5	4-5	4-5	5	4-5	5	5	4.84
$ m R6_{4H}$	60	30	5	5	5	5	5	<u>4</u> -5	5	5	4.91
${ m R8}_{ m 4A}$	80	30	4-5	4-5	4-5	4-5	4-5	3-4	5	5	4.44
Control <sup>d</sup>			2 <u>-3</u>	e,	2–3	2-3	1-2	c.	5	5	3.25

To assess and contrast reduction clearing methods, the average fastness rating was determined by computing the mean of all the fastness values associated with each reduction clearing proce-

samples without reduction clearing applications, only dyed samples

Control: Washing fastness values of stained multifiber (Polyamide 6) dyed PEI

Based on these calculated values, the highest overall fastness average score (4.91 Gy scale rating) was achieved with the  $R6_{4H}$  treatment at 60 °C for a duration of 30 min.

#### 3.3.2 Tensile Strength Properties

In textile finishing, the use of auxiliary chemicals and specific process conditions significantly impacts the tensile strength of fabrics. Table 8 presents tensile strength data for polyetherimide (PEI) fibers dyed with C.I. Disperse Red 73, post-reduction clearing treatments. The study includes a control group of fibers that were dyed but not subjected to reduction clearing, to gage the impact of such processes on fabric strength. After conducting nine measurements per reduction clearing treatment, a noticeable decline in tensile strength was observed in the PEI fibers compared to the control group. The minimal loss of strength was noted when using the R1<sub>4H</sub> reduction clearing process, which includes 4 g/l of NaOH at a temperature of 70 °C for 30 min. Conversely, the most significant strength reduction occurred with  $R6_{4H}$  and  $R8_{4A}$  reduction clearing treatments. R6 is a sulfuric acid derivative and operates as a reducing agent in alkaline conditions, while R8 is a resin-based reducing agent effective in acidic conditions. The pronounced strength degradation with these agents is likely because acid treatments on PEI fibers lead to strength diminution, as documented in existing research. Here, treatments with concentrated 10% acid solutions of nitric acid (HNO<sub>3</sub>), acetic acid (CH<sub>3</sub>COOH), hydrochloric acid (HCl), and sulfuric acid  $(H_2SO_4)$  resulted in strength losses of 8%, 7%, 14%, and 15% for the PEI fibers, respectively. Also, past research indicates that the resilience of PEI fibers is affected when exposed to varying alkaline solutions. Specifically, a 10% concentration of NaOH has been found to slightly reduce the fibers' strength by approximately 7% [17, 19].

## 3.3.3 Environmental Analyses of Reduction Clearing Processes

The environmental impact of wet processing in the textile sector is garnering heightened scrutiny. Reduction clearing processes, specifically, pose significant environmental challenges, encompassing the type of reducing agents employed, as well as the consumption of water, energy, and additional chemicals required in their use. Consequently, when optimizing reduction clearing conditions for PEI fibers, it's crucial to weigh the environmental repercussions of these processes alongside considerations of colorfastness and the physical attributes of the fabric. This dual focus ensures that the chosen procedures are not only effective for the textile's properties but also sustainable and less detrimental to the environment [21]. It is aimed to assess the eco-footprint of different reduction clearing agents and reduction clearing processes,

Dyed+reduction cleared with below process type	Reduction clearing time (min)	Reduction clearing temp. (°C)	Initial modulus (N/tex)	Breaking Force (N)	Breaking tenac- ity (N/tex)	Elongation (%)
R1 <sub>4H</sub>	80	15	0.97	2.69	0.08	10.32
R1 <sub>4H</sub>	70	30	0.88	2.83	0.09	10.32
$R2_{4H}$	70	30	0.65	2.70	0.08	12.24
R5 <sub>2C</sub>	80	30	0.62	2.70	0.08	12.88
R5 <sub>4H</sub>	80	30	0.63	2.39	0.07	10.55
R6 <sub>4H</sub>	60	30	0.58	2.18	0.07	10.30
R8 <sub>4A</sub>	80	30	0.45	2.13	0.06	11.48
Control <sup>a</sup>			0.96	2.84	0.09	10.08

Table 8 Effect of recommended reduction clearing agents and processing conditions on PEI fabric strength properties

<sup>a</sup>Control: Washing fastness values of stained adjacent multifiber (Polyamide 6) dyed PEI samples without reduction clearing applications, only dyed samples

the key environmental analyses according to water quality parameters [Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD<sub>5</sub>), and Total Organic Carbon (TOC)] was applied (Table 9). In addition to these, carbon footprints were calculated that may occur during the reduction clearing process.

The agents selected for study are those commonly used in commercial applications, with some marketed as ecofriendly due to their biodegradability. However, as the study's experiments were conducted using laboratory equipment, which differs from industrial settings in terms of liquor ratios and procedural steps, direct comparisons of the COD, BOD<sub>5</sub>, and TOC values with those from full-scale production may not be accurate. Thus, the environmental impact assessments derived will be instrumental in contrasting the various reduction clearing processes examined. The ultimate aim is to employ reduction clearing for disperse-dyed PEI fibers in a manner that preserves the fibers' structure, texture, and colorfastness, while also ensuring the process is environmentally sustainable.

Table 9 displays the application of five distinct reducing agents, labeled R1, R2, R5, R6, and R8, under various processing conditions, with the concentration of each reducing agent maintained at 2 g/l. The chemical oxygen demand (COD) values, which reflect the oxygen quantity needed to oxidize organic substances in the wastewater, reveal that higher COD figures suggest a greater environmental impact. Specifically, the data shows that the  $R8_{4A}$ agent has a substantially larger environmental footprint than the other reduction clearing agents assessed.

BOD, or biological oxygen demand, indicates the quantity of oxygen needed by bacteria to decompose organic materials. Elevated BOD levels typically point to increased contamination; however, the data indicates low to nonexistent BOD values, implying the potential resistance of these materials to biodegradation. TOC, or total organic carbon, measures the carbon content within organic compounds and is frequently utilized as a broad marker of water quality. From the data, it is clear that no single reducing agent is entirely benign from an environmental perspective. Options like thiourea dioxide may look favorable in terms of TOC but still carry a higher COD value. On the other hand, glycose derivatives represent the highest environmental risk based on both COD and TOC.

Given these considerations, the selection of a reducing agent should balance effective dye reduction with minimal environmental impact. This might involve considering not just the immediate metrics but also the lifecycle analysis of

Table 9 Environmental analyzes according to water quality parameters of wastewater samples taken after recommended reduction clearing processes

Dyed + reduction cleared with below process type	Reduction clearing time (min)	Reduction clearing temp. (°C)	COD (mg/l)	BOD <sub>5</sub> (mg/l)	TOC (mg/l)
R1 <sub>4H</sub>	80	15	264	50	49.60
R1 <sub>4H</sub>	70	30	288	50	48.29
$R2_{4H}$	70	30	368	100	44.68
R5 <sub>2C</sub>	80	30	312	100	259.4
$R5_{4H}$	80	30	928	150	314.4
$R6_{4H}$	60	30	248	50	351.3
R8 <sub>4A</sub>	80	30	3808	250	1521

the chemicals involved, including their production, transportation, and disposal costs to the environment.

Carbon footprint: The carbon footprint serves as a measure reflecting the impact of all greenhouse gas emissions attributable to a product, service, or procedure across its life span [36, 37]. The most accurate method for determining the carbon footprint involves conducting a life cycle assessment (LCA) or employing input–output analysis techniques [38, 39]. These methods adhere to the standards set forth by ISO 14040 and ISO 14043, which focus on LCA protocols [39, 40]. Additionally, the carbon footprint calculations for businesses and products follow the guidelines of ISO 14064 (parts 1 through 3), ISO 14067, and PAS 2050, which specify the methodology for assessment [37, 39, 41–43]. The computation of the carbon footprint includes cumulative primary emissions from significant processes over the product's entire life cycle [37].

During the third phase of the research, projections and estimations were conducted using specialized software, which held constants such as the quantity of material processed (500 kg), as well as the type and capacity of the machine, across seven different proposed processing conditions (Fig. 1 and Table 10). The software's capability to calculate environmental impact was utilized due to the variance in liquor ratios between laboratory settings and actual industrial applications. The laboratory's higher liquor ratios (1/30) lead to inflated values for carbon, natural gas, and electricity footprints because of the increased need for heating, chemicals, and water, unlike in actual industrial scenarios where lower ratios (1/7-1/10) in short liquor are standard. Consequently, the footprint data presented in Fig. 1 should be employed primarily to evaluate and compare the environmental efficiency of the processing conditions against one another.

A closer inspection of the provided Fig. 1 and Table 10 reveals distinctions between alkaline and acidic reduction clearing processes. Alkaline reduction clearing typically include subsequent rinsing and neutralization steps, whereas acidic reduction clearing treatments are followed only by rinsing. These differences significantly influence water and energy usage. Consequently, the acidic reduction clearing process (R8: resin derivative) registers the smallest carbon footprint. However, when assessing the environmental load through COD, BOD<sub>5</sub>, and TOC measures for R8, it emerges as the condition with the highest environmental impact. It is important to consider that these assessments are based on waste liquor resulting from reduction clearing treatments performed in a laboratory setting.

The findings from this comprehensive investigation into reduction clearing procedures for high-performance PEI fibers dyed with disperse dyes provide significant insights into the balancing act required between operational efficiency, fabric quality, and environmental sustainability. In this context, Table 11 and Fig. 2 detail the total environmental impact of the assessed reduction clearing methods, presenting each process's contribution as a percentage in the conducted analyses. According to these resources, the identification of sodium dithionite at 70 °C for a duration of 30 min as the optimal process highlights the potential for reducing environmental impact through the careful selection of conditions and chemicals. Given the close similarity in fastness values among the various reduction clearing processes, the environmental impact played a pivotal role in determining the optimal reductive clearing conditions. This study not only pinpoints the optimal conditions for reduction clearing but also establishes a precedent for future research and industry practices by underscoring the importance of environmental considerations in the textile manufacturing process. A key takeaway from this study is the essential role of process optimization in achieving environmental sustainability without compromising fabric quality. This insight is particularly relevant in light of the global textile industry's ongoing efforts to reduce its carbon footprint and water use, suggesting a pathway toward more sustainable manufacturing practices.

# 4 Conclusions

The study aimed to identify the most effective reduction clearing procedures for high-performance PEI fibers dyed with disperse dyes. It was conducted in three consecutive phases, with each phase building upon the optimal results achieved in the previous one. Consequently, the recommended reduction clearing methods were compared, considering fabric attributes and the environmental implications of the processes. This comprehensive evaluation aimed to determine the ideal reduction clearing conditions for dyeing PEI fiber structures.

Initial reduction clearing steps were conducted at temperatures ranging from 50 to 90 °C, for durations of 15 to 30 min. These steps occurred under varying acidic and alkaline conditions, tailored to the pH requirements of the reducing wash agent. The minimal washing fastness rating observed in the first phase of reduction clearing was 3, typically noted in processes conducted at 50 and 60 °C. It was noted that most procedures at this phase demonstrated commercially viable washing fastness ratings of 4 or higher.

During the second stage, the reduction clearing of PEI fabrics dyed using various disperse dyes, characterized by different dye structures, molecular sizes, and color attributes, was analyzed. In this phase, the minimum washing fastness rating observed was between 3 and 4, specifically in PEI fabrics dyed with C.I. Disperse Yellow 54. For all other processes, washing fastness ratings deemed commercially satisfactory, at 4 or higher, were achieved.

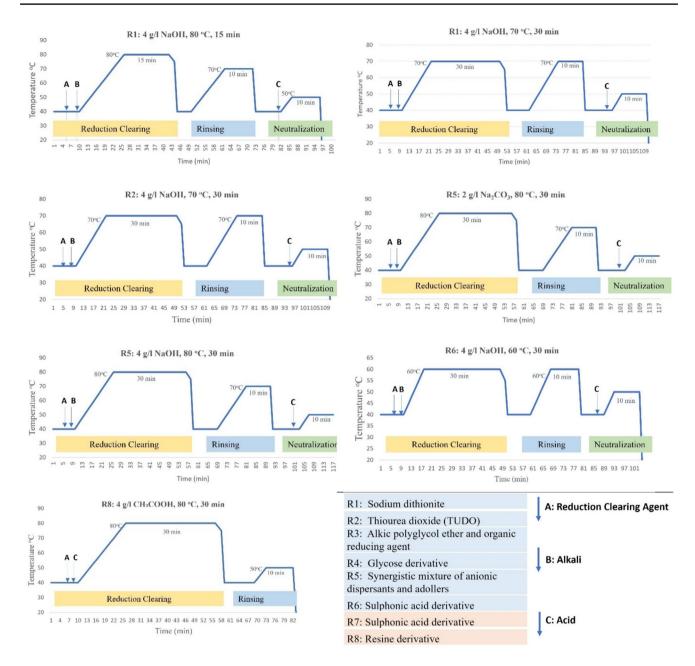


Fig. 1 Process charts used in calculating the production-related carbon footprint of reduction clearing processes with Ruco-Logic® eCO<sub>2</sub> meter software

In the final stage, leveraging the optimal outcomes from the first two steps of the reduction clearing processes, the study focused on evaluating the fastness, breaking strength, and environmental impacts of these reductive washing procedures. In summary, the majority of the tested reduction clearing processes in the study demonstrated both satisfactory color fastness and environmentally friendly outcomes for PEI fibers.

Upon closer examination, it was evident that the reduction clearing process involving sodium dithionite at 70 °C for a duration of 30 min emerged as the optimal choice in terms

of color fastness assessments, strength values, and overall environmental impact.

This process is critical, not only for its operational efficacy but also for its environmental footprint, which includes the selection of specific reducing agents and the associated resource consumption—water, energy, and auxiliary chemicals. In optimizing the reduction clearing conditions for PEI fibers, the manuscript argues for a balanced approach that accounts for both environmental impacts and the maintenance of key fabric qualities, such as colorfastness and integrity. This comprehensive perspective is vital to ensure

Table 10 Comparison of environmental impacts as a result of the process using 30.6 kWh energy at a ratio of 1/30 liquor for 500 kg of dispersedyed PEI fabric under different recommended reduction clearing conditions

Dyed + Reduction cleared with below process type	Reduction clearing time (min)	Reduction clearing temp. (°C)	Carbon Footprint <sup>NGC</sup> (CO <sub>2</sub> /kg)	Carbon Footprint <sup>EC</sup> (CO <sub>2</sub> /kg)	Carbon Footprint <sup>T</sup> (CO <sub>2</sub> /kg)
R1 <sub>4H</sub>	80	15	683	80	763
R1 <sub>4H</sub>	70	30	598	89	687
$R2_{4H}$	70	30	598	89	687
R5 <sub>2C</sub>	80	30	683	95	778
R5 <sub>4H</sub>	80	30	683	95	778
R6 <sub>4H</sub>	60	30	513	81	594
R8 <sub>4A</sub>	80	30	427	70	497

NGC Carbon Footprint from natural gas consumption, EC Carbon Footprint from electricity consumption, T Total Carbon Footprint

	COD (%)	BOD <sub>5</sub> (%)	TOC (%)	Carbon Foot- print (%)	Total environ- mental impact (%)
R1 <sub>4H</sub> , 80 C, 15 min	4.25	6.67	1.91	15.95	7.19
R1 <sub>4H</sub> , 70 C, 30 min	4.63	6.67	1.86	14.36	6.88
R2 <sub>4H</sub> , 70 C, 30 min	5.92	13.33	1.72	14.36	8.83
R5 <sub>2C</sub> , 80 C, 30 min	5.02	13.33	10.37	16.26	11.2
R5 <sub>4H</sub> , 80 C, 30 min	14.93	20.00	12.10	16.26	15.8
R6 <sub>4H,</sub> 60 C, 30 min	3.99	6.67	13.52	12.42	9.15
R84A, 80 C, 30 min	61.26	33.33	58.53	10.39	40.9
Total	100	100	100	100	100

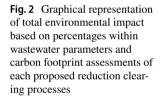
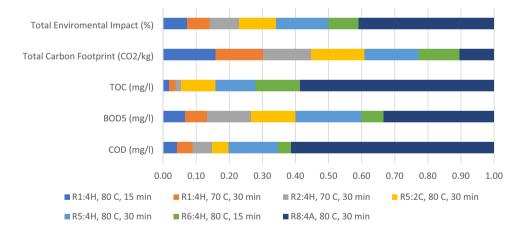


Table 11Total environmentalimpact based on percentageswithin wastewater parametersand carbon footprintassessments of each proposedreduction clearing processes



that the industry adopts reduction clearing practices that are not only technically efficient but also advance the cause of sustainability and environmental responsibility.

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**Data Availability** All data that support the findings of this study are included within the article.

### Declarations

Conflict of Interest There is no potential conflict to declare.

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