



The Disperse Dyeing of Polyetherimide Fibers Using Orthogonal Array (Taguchi) Design

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Abstract

Polyetherimide (PEI) fibers are classified as high-performance fibers due to their notable characteristics, including chemical resistance, heat resistance, and flame resistance. However, the amorphous structure and intrinsic dyeability of polyetherimide (PEI) fibers set them apart from other high-performance fibers. The fibers possess considerable potential due to their remarkable performance features and are utilized in a wide range of industries, containing fashion and technical textiles, for varied purposes. The present work utilized the Orthogonal Array Taguchi method to systematically improve the dyeing process parameters for fabric samples made of polyetherimide (PEI). The use of a systematic methodology enabled effective experimentation and enabled the determination of the most favorable values of factors, while simultaneously reducing the utilization of resources. The study involved conducting rigorous experiments to evaluate the influence of several parameters on the color strength, tensile qualities, and washing fastness properties of the fabric samples. Furthermore, the utilization of the Taguchi approach facilitated the anticipation of these pivotal fabric properties by considering certain parameters. The objectives of this research encompass not only the progression of textile industry, but also the provision of beneficial perspectives for augmenting the caliber and efficacy of PEI fabrics in various applications.

Keywords Polyetherimide fiber · PEI · High-performance fibers · Coloration · Dyeing · Amorphous fibers · Disperse dyeing

1 Introduction

Within the ever-changing and complex realm of the textile industry, the endeavor to enhance the caliber and functionality of fabrics, while simultaneously exploring alternate sources of raw materials, poses a substantial aim. The dyeing process and dyeing conditions are of significant importance in defining the aesthetic appeal, longevity, and practicality of textile goods. Nevertheless, the attainment of adequate color for high-performance fibers poses significant challenges as a result of their unique material composition. As a result, in general, the most favored technique for dyeing these kinds of fibers is the dope-dyeing method, which entails incorporating colorants into the polymer solution. The amorphous structure and inherent dyeability of polyetherimide (PEI)

fibers distinguish them from other high-performance alternatives [1, 2]. These fibers exhibit significant promise due to their outstanding performance characteristics and wide-ranging uses in several industries, ranging from the fashion sector to technical textiles.

In contrast to other high-performance fibers that exhibit crystalline structures, PEI fibers possess an amorphous structure, which is due to their irregular chemical composition [3]. Furthermore, it should be noted that PEI fibers possess notable characteristics including chemical resistance, heat resistance, and flame resistance, rendering them very amenable to processing [2, 4]. On the other hand, high-performance fibers such as aramid, PEEK, and PPS exhibit reduced amorphous characteristics and instead display a greater degree of crystalline structures. The presence of this particular crystalline structure enhances the strength and durability of the material, making it more resistant to challenging environmental factors such as elevated temperatures, chemical exposure, and flames. However, it has a negative impact on the material's ability to interact with water (hydrophilicity) and its capacity to be dyed effectively. In addition, PEI fibers have exceptional thermal characteristics and

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demonstrate notable resilience to various chemicals. These materials demonstrate strong resistance to diluted acids and alkalis; however, their resistance to concentrated acids is somewhat less pronounced. The reaction of organic solvents can exhibit variability, and there may be some constraints associated with their exposure to aromatic solvents and vapors [5].

Polyetherimide (PEI) fibers are utilized in a range of technical textiles due to their notable chemical and heat-resistant characteristics. The aforementioned categories encompass protective, industrial, transportation, medicinal, and geotextiles, among others [1]. PEI fibers have the potential to be utilized either in isolation or in combination with other advanced or traditional fibers, hence facilitating the production of technical textiles. The determination of blend ratios is contingent upon the desired qualities of the final product. It is worth mentioning that PEI fibers are frequently utilized in conjunction with high-performance fibers that possess heat and flame-resistant capabilities, such as aramids, PBI, PBO, and polyimide, to augment their thermal characteristics [2].

The inherent adaptability of this material enables the production of many items that offer both protection and comfort, such as incorporating combinations with organic fibers such as wool. In addition, the amorphous nature of PEI fiber materials allows for easy coloration using traditional polyester dyeing procedures, resulting in a diverse range of colors [1].

However, the application of polyetherimide fibers within the textile industry, as well as research concerning these fibers, is predominantly focused on their role as reinforcement in composite structures [6–8], in porous membrane structures [9–11] (used in recycling textile wastewater [12–14], gas separation [15–17], oil/water separation [18, 19], etc.), in high-temperature thermal insulators [20] or in electronic sensors/applications [6, 21]. There is a scarcity of scientific literature that directly addresses their use as standalone textile products, particularly studies examining finishing processes or investigating the coloration of polyetherimide fibers. It was noted that the dyeing properties of these fibers were only mentioned in few patent studies [2, 4, 22]. In addition, technical bulletins from fiber-producing companies contain dyeing recommendations. The absence of scientific articles supporting the easy dyeability of this fiber, which is classified as a high-performance fiber, prompted our research into this topic. Therefore, this study aimed to investigate the dyeability of these fibers with disperse dyestuffs and explore the impact of dyeing parameters on process efficiency. An azo group disperse dye commonly used in industry was held constant across all dyeing processes, while dyeing parameters and levels were considered as variables. Statistical experimental design methods were employed to reduce the number of dyeing experiments required, minimize time and material wastage, and mitigate environmental impacts.

Ensuring optimal dyeing outcomes for PEI knitted fabrics is a paramount endeavor, directly influencing critical factors such as color strength, tensile properties, and washing fastness. The optimization of dyeing process parameters has garnered significant research interest, driven by the imperative to maximize resource efficiency, minimize environmental impact, and produce textiles with exceptional properties. In addition, the ability to predict dyeing outcomes holds substantial value for maintaining consistent product quality and minimizing production costs. The demand for methods and techniques that conserve raw materials, time, and energy is increasingly vital for the future, especially within industries bearing a significant environmental burden, such as the textile sector. In this context, the Taguchi experimental design method offers substantial savings by yielding optimal results through a significantly reduced number of experiments, particularly in scenarios involving multiple parameters and levels.

In recent years, optimization and experimental design techniques including the Orthogonal Array Taguchi method has gained widespread adoption across various industries and research domains worldwide, including the textile sector [23–27]. Over the last several years, a number of research have been observed an increase undertaken with the aim of optimizing and improving various facets of textile operations. Pervez et al. conducted a study that focused on the optimization and prediction of the cotton fabric dyeing process. The authors employed a hybrid method of Taguchi design and machine learning techniques to achieve their objectives [28]. In other study, Üstündağ et al. investigated the optimization of coating process parameters for denim fabrics. They utilized the Taguchi technique in conjunction with grey relational analysis [29]. The study conducted by Nassif in 2017 aimed to optimize the fabric characteristics that influence the air permeability of woven cotton fabrics [30]. The study conducted by Hossain et al. focused on the optimization of dyeing process parameters and the prediction of color strength for knitted fabrics made from a combination of viscose and Lycra. These researchers employed the Taguchi method for this purpose [31]. In addition, Das and Thakur conducted a study to examine the impact of various structural parameters of woven fabrics, including warp tension, fabric sett, backrest position, and pick insertion rate, on the shrinkage of woven fabrics in both the warp and weft directions [32]. The researchers employed the Taguchi technique to analyze the data [32–34]. In a study conducted by Saravanan et al., the authors investigated the scouring process of greige cotton fabrics. They employed a combination of cellulose, pectinase, and protease enzymes, and optimized several process parameters utilizing the Taguchi method [35].

In this study, the Taguchi method emerges as a potent tool for systematically optimizing dyeing process parameters

while simultaneously predicting resulting color strength, tensile properties, and washing fastness for dyed PEI knitted fabrics. Rooted in statistical principles and robust experimental design, the Taguchi method offers a structured approach to investigating multiple variables and their interactions, ultimately aiming for optimal outcomes.

This scientific article delves deep into the intricacies of optimizing dyeing process parameters for PEI knitted fabrics using the Taguchi method. Furthermore, it explores the feasibility of predicting key fabric properties based on the selected parameters. In doing so, this research contributes not only to the advancement of textile engineering but also offers invaluable insights to enhance the quality and performance of PEI knitted fabrics across diverse applications. Subsequent sections of this article will provide a comprehensive overview of the experimental methodology, data analysis, results, and discussions, ultimately shedding light on the potential of the Taguchi method to revolutionize the dyeing process of PEI knitted fabrics.

2 Experimental

2.1 Fabrics, Dyes, and Chemicals

In this current study, the dyeing experiments were conducted using single jersey polyetherimide (100%) knitted fabrics with yarn specifications of 300 denier and 150 filaments. Disperse Dye, C.I. Disperse Blue 148, (obtained from DyStar) was used for dyeing processes.

2.2 Fabric Pre-treatment

To prepare the polyetherimide (PEI) fiber-based knitted fabrics for dyeing, a pre-dyeing scouring process was implemented to remove any potential contaminants and oils from the fabric structure. This scouring process involved the use of a 1 g/L concentration of Kieralon Jet B, a nonionic surfactant provided by BASF, along with 1 g/L of sodium carbonate. The process was carried out at a temperature of 60 °C for a duration of 15 min. Following the scouring process, the fabrics were meticulously rinsed first with warm water and then with cold water. Finally, the fabrics were allowed to air dry at ambient room temperature.

2.3 Fabric Dyeing

PEI fabric samples, after undergoing a preliminary treatment to remove any potential contaminants such as dirt and oil, were subjected to the dyeing processes following the specified conditions outlined in the Taguchi $L_{25}(5^6)$ OA (orthogonal matrix) design. These dyeing processes were executed utilizing the ATAÇ LAB-DYE HT Infrared dyeing machine

through the exhaust method. For the dyeing process, CI Disperse Blue 148 (obtained from DyStar), an azo group dye with a medium molecular size, was selected as the disperse dyestuff. As part of the disperse dyeing process, Dr. Perigen EC (obtained from Petry company) served as the ecological carrier material, and Setamol BL (obtained from BASF) was used as the dispersion material at a concentration of 1 g/L.

2.4 Color Strength Measurement

The evaluation of the dyeing properties of PEI fiber fabrics considers K/S (color strength) values. These K/S values are determined by calculating the reflectance values at the appropriate wavelength of maximum absorbance (λ_{max}) for each dyed sample. This calculation is performed using a DataColor SpectraFlash 600 spectrophotometer, with illuminant D65 and a 10° standard observer. To ensure consistency, each fabric is assessed in four different areas, twice on each side, and the average value is computed. The Kubelka–Munk theory is used to calculate the K/S values for the samples, as shown in Eq. (1):

$$\frac{K}{S} = \frac{(1 - R)^2}{2R} \quad (1)$$

where K stands for the coefficient of light absorption, S is the coefficient for light scattering, and R denotes the reflectance level of the fabric that has been dyed.

2.5 Washing Fastness Evaluation

The washing fastness to home washing will be assessed using the Rotawash machine following the ISO 105:C06 Test Standard. This washing fastness test will be conducted on PEI fabrics at a temperature of 50 °C with the use of sodium perborate (C06/B2S). Following the washing fastness test, an assessment will be conducted to evaluate any alterations in the color of the dyed fabric samples that have been laundered. This assessment was done in accordance with ISO 105-A03 Tests for color fastness Part A3 grey scale for assessing staining level of multi-fiber using a 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.

2.6 Evaluation of Fiber Tensile Properties

To investigate the impact of varying parameters following the dyeing processes on the strength properties of the yarns within the fabric structure, a yarn strength test was conducted. This test involved extracting yarns from the knitted fabric structure, and the assessment was carried out in

accordance with the ASTM 3822 yarn strength standard. The testing equipment used for this purpose was a Tinius Olsen Tensile tester.

2.7 Design of Experiment: Orthogonal Array (Taguchi) Method

The Taguchi method is a statistical approach for optimizing complex systems and is widely used in engineering, manufacturing, marketing, and other fields to improve product quality and performance. It was developed by Japanese statistician and engineer Genichi Taguchi. It focuses on the efficient utilization of engineering principles rather than relying on complex statistical approaches. The fundamental framework of the Taguchi method for successful optimization comprises seven main steps [36]:

- (1) **Determining factors and interactions:** Identify the key factors that influence the process or system you are studying. In addition, consider any interactions between these factors that may affect the outcome.
- (2) **Determining the levels of each factor:** Decide on the specific levels or settings for each of the identified factors. These levels represent the range over which you will conduct experiments.
- (3) **Selecting the appropriate orthogonal matrix:** Choose an orthogonal matrix that aligns with the number of factors and levels. Orthogonal Arrays are utilized to systematically vary factors while minimizing the number of experiments needed.
- (4) **Transferring factors and interactions to orthogonal matrix columns:** Map the selected factors and their interactions to the columns of the orthogonal matrix. This step helps create a structured experiment plan.
- (5) **Performing experiments:** Execute the experiments based on the combinations of factor levels specified in the orthogonal matrix. These experiments are designed to gather data on how factors impact the system.
- (6) **Analyzing the data and determining the optimal levels:** Analyze the data collected from the experiments to assess the effects of each factor and interaction. The goal is to identify the optimal factor settings that lead to the desired outcome or performance.
- (7) **Conducting verification experiments:** After determining the optimal factor levels, conduct verification experiments to confirm that the chosen settings consistently produce the desired results. This step ensures the reliability of the optimization process.

By following these seven steps, the Taguchi method provides a structured and efficient approach to optimize processes and systems while minimizing the number of

experiments required. It is particularly valuable in the field of quality improvement and robust design [31, 36].

The Taguchi method goes beyond being just an experimental design technique; it is an exceptionally valuable tool, especially when aiming for high-quality system design. This method, to some extent, simplifies the consideration of interactions between various factors. In the Taguchi Experimental Design approach, the results obtained from experiments are assessed by transforming them into a signal-to-noise (S/N) ratio. Here, the signal component represents the desired and actual value that the system aims to measure, while the noise component reflects the impact of undesirable factors within the measured factor.

The characteristics of the response that one hopes to achieve through these experiments play a crucial role in the computation of the signal-to-noise ratio. Consequently, S/N calculations are performed using three different approaches: “smaller is better” (aiming for the lowest value), “larger is better” (striving for the highest value), and “nominal is best” (targeting an average value). In whichever approach is selected for calculation, a larger S/N value signifies superior performance in the experimental design [36, 37].

Smaller is better,

$$S/N = -10 \log \left(\sum_{i=1}^n y_i^2 \right) \quad (2)$$

Larger is better,

$$S/N = -10 \log \left(\frac{1}{n} \sum_{i=1}^n 1/y_i^2 \right) \quad (3)$$

Nominal is best,

$$S/N = -10 \log \left(\frac{\bar{y}^2}{S^2} \right) \quad (4)$$

In equations, the symbols are explained as follows:

y_i represents the performance response. This symbolizes the performance value obtained from a trial or test.

i denotes the observation value. It represents a specific observation value from a trial or test.

n signifies the number of trial tests. It indicates how many trials or tests were conducted.

\bar{y} symbolizes the mean value. This represents the average value obtained by dividing the sum of observation values by the number of trials.

S^2 represents the variance of observation values. It is a term that measures how much observation values vary.

In addition, the effects of factors on the process are statistically examined using Analysis of Variance (ANOVA)

testing. This test is used to assess the contributions of factors to the process and, thus, determine factor combinations that will lead to optimal performance [38]. An important characteristic that distinguishes this design from other experimental designs is that factors can be evaluated independently of each other. This means that the impact of each factor on the process can be assessed independently of the other factors, increasing the flexibility of the design. [36, 39].

2.8 Experimental Design

In the present study, six control factors, namely dye concentration, dyeing time, dyeing temperature, carrier

concentration, liquor ratio, dyeing pH, and 5 levels for each parameter were chosen for Taguchi $L_{25} (5^6)$ OA (orthogonal matrix) design as shown in Table 1 and Fig. 1. The experimental design utilizing the Taguchi method was executed using MiniTab 18 software. Following the dyeing process, measurements were taken for color strength (K/S) values, fiber tensile properties (breaking force), and washing fastness values which served as the benchmark for identifying the optimal dyeing conditions. Consequently, the “larger is better” mode was deemed suitable for determining the optimum conditions in this experimental design.

Table 1 Experimental design parameters and levels of PEI disperse dyeing

Parameter	Code	Level					Unit
		1	2	3	4	5	
Temperature	A	100	110	120	130	140	°C
Time	B	15	30	60	90	120	Min
Dye owf	C	0,5	1	2	3	4	%
Carrier owf	D	0	1	2	4	6	%
pH	E	3	4	5	6	7	–
Liquor ratio	F	1:10	1:20	1:30	1:40	1:50	–

Experiment No	Temperature	Time	Dye concentration	Carrier concentration	pH	Liquor Ratio
	A	B	C	D	E	F
1	100	15	0.5	0	3	10
2	100	30	1	1	4	20
3	100	60	2	2	5	30
4	100	90	3	4	6	40
5	100	120	4	6	7	50
6	110	15	1	2	6	50
7	110	30	2	4	7	10
8	110	60	3	6	3	20
9	110	90	4	0	4	30
10	110	120	0.5	1	5	40
11	120	15	2	6	4	40
12	120	30	3	0	5	50
13	120	60	4	1	6	10
14	120	90	0.5	2	7	20
15	120	120	1	4	3	30
16	130	15	3	1	7	30
17	130	30	4	2	3	40
18	130	60	0.5	4	4	50
19	130	90	1	6	5	10
20	130	120	2	0	6	20
21	140	15	4	4	5	20
22	140	30	0.5	6	6	30
23	140	60	1	0	7	40
24	140	90	2	1	3	50
25	140	120	3	2	4	10

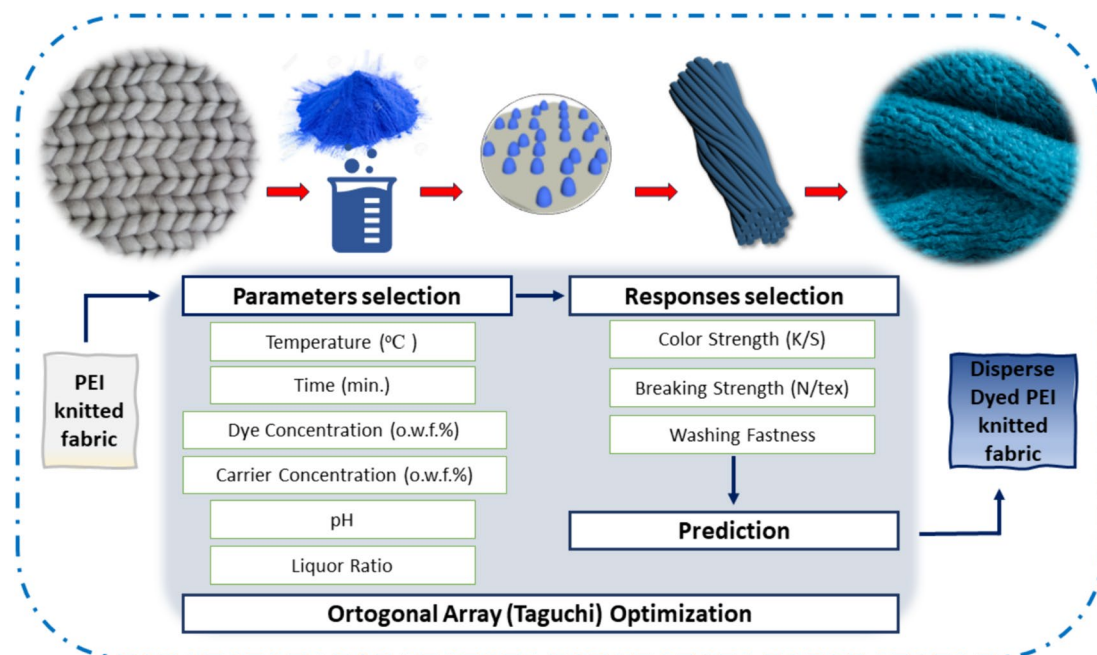


Fig. 1 Orthogonal Array (Taguchi) optimization of disperse dyeing conditions of PEI fabric samples

2.9 Determination of Optimum Levels for Each Factor

After obtaining K/S , washing fastness, and breaking force values as a result of the dyeing processes, S/N ratios are calculated for each experimental data. These ratios are then used to determine the main influence of each control factor. Afterward, MiniTab software is employed to create a response table and response graph for each control factor. The response table displays the average response value across different levels for each parameter [31]. To calculate the delta value of a factor in the response table, you can use the following formula:

$$\text{Delta value} = N_H - N_L \quad (5)$$

where N_H represents the highest S/N ratio value for a factor, and N_L represents the lowest S/N ratio value for the same factor. In accordance with Taguchi analysis principles, the factor with the highest delta value is considered the most influential or highest-ranking factor. Ultimately, the optimal parameters of the control factors are determined through rank analysis based on the information provided in the response table and response graph. This methodology allows for the identification of the most effective combinations of control factors to achieve the desired outcomes in the dyeing process.

2.10 Analysis of Variance (ANOVA)

Data yielded from the Taguchi experimental setup will undergo analysis to pinpoint errors in the experiment and perform significance tests. Moreover, ANOVA will be employed to gauge the relative influence of each control variable on the outcome. A few key terms and calculations frequently associated with ANOVA are elaborated below [31, 37]:

Sum of squares for a factor (SS_F): Eq. (6) enables the calculation of SS_F :

$$SS_F = \frac{mn}{L} \sum_{k=1}^L (\bar{y}_k - \bar{y})^2 \quad (6)$$

It takes into account m as the total experiments conducted, n for the number of repetitions for each experiment, L for factor levels, \bar{y}_k as the mean outcomes for a specific factor at k^{th} level, and \bar{y} for the overall average across all experiments [31].

Total square sum (SS_T): Eq. (7) is used here, where y_i denotes individual experimental results.

$$SS_T = \sum_{j=1}^m \left(\sum_{i=1}^n y_i^2 \right)_j - mn(\bar{y})^2 \quad (7)$$

Error in sum of squares (SS_{Error}): Eq. (8) shows SS_{Error} calculated as SS_T minus SS_F

$$SS_{Error} = SS_T - SS_F \tag{8}$$

Degree of freedom (DOF):

$$DOF_F = L - 1 \tag{9}$$

Error degrees of freedom (DOF_{Error}): Eq. (10) defines DOF_{Error} as m multiplied by $(n-1)$.

$$DOF_{Error} = m \times (n - 1) \tag{10}$$

Variance (V_F): Eq. (11) calculates V_F as SS_F divided by DOF_F , multiplied by 100.

$$V_F = SS_F / DOF_F \times 100 \tag{11}$$

Error variance (V_{Er}): Eq. (12) provides V_{Er} as SS_{Error} divided by DOF_{Error} .

$$V_{Er} = SS_{Error} / DOF_{Error} \tag{12}$$

2.11 Control Experiment

The final stage in the Taguchi method includes a control experiment, crucial for validating the additive model derived from the outcomes [31, 37]. The total signal-to-noise (S/N) ratio’s predicted value under ideal conditions is determined by employing an additive model, as illustrated in Eq. (13).

$$S/N = n_m + \sum_{i=1}^n (n_i - n_m) \tag{13}$$

where n_m is the mean of total S/N ratio, i is the number of factors, and n_i denotes the S/N ratio for optimum factor level.

This facilitates predicting optimal conditions to refine the process, which can subsequently be verified through a control experiment. In addition, the resultant value will fall within a confidence interval (CI). Equation (14) defines how the CI is expressed under a $1 - \alpha$ confidence level.

$$CI = \left| \left(N_{\frac{\alpha}{2}} \times \frac{S}{\sqrt{m_e}} \right) \right| \tag{14}$$

where N is the total experiment number, S is the standard deviation, m_e is the ratio of total experiment number and the degree of freedom in the equation for calculating the predicted value.

The ultimate objective of these complex calculations is to establish individual mathematical models for color strength (K/S), washing fastness, and breaking force. This is achieved by forming a linear relationship between operational parameters, executed using Minitab 18 software.

2.12 Mathematical Model Development

Linear relationships have been established for K/S , breaking force, and washing fastness, correlating these results with the operational variables. This analysis was performed using Minitab 18 software and is displayed in Table 11.

3 Results and Discussion

3.1 Taguchi Optimization of the Dyeing Processes

3.1.1 Analysis of Experimental Results

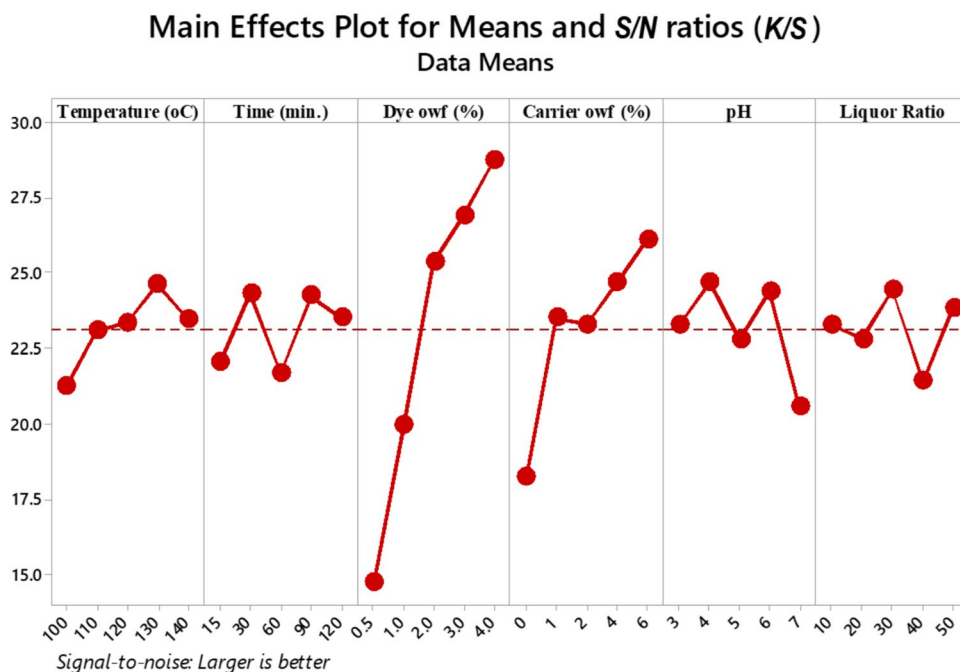
A comprehensive analysis is essential for optimizing the disperse dyeing processes for PEI fabric samples, focusing on key performance indicators such as color strength, breaking force as tensile strength, and washing fastness. This research aims to explore the complex interplay between dyeing parameters and each aspect of the dyeing results in depth. The objective is to systematically identify the optimal settings for each parameter, achieved by closely examining the nuanced interactions among various dyeing variables.

Figure 2 clearly demonstrates a discernible and significant enhancement in K/S when the dyeing temperature is raised from 100 to 140 °C. This phenomenon can be explained by the fact that higher temperatures promote better dispersion of disperse dye particles, leading to improved color uniformity and intensity [40, 41]. Also, at elevated temperatures, the structure of synthetic fibers relaxes, opening up more sites for the dye to enter. This can result in deeper and more uniform coloration. What is more, higher temperatures can speed up the molecular movement of the dyes. This increased kinetic energy allows for faster diffusion of dye molecules into the fiber structure, enhancing the dyeing process [41, 42].

The relationship between dyeing time and the K/S value is notably non-linear, as illustrated in Fig. 2. An increase in K/S is observed when the dyeing time is extended from 15 to 90 min. However, the trend slightly alters when the dyeing duration is further prolonged to 120 min. The data also reveal a clear relationship between dye concentration and color strength. As the dye concentration increases, the dye concentration in the bath rises correspondingly. This increase in dye concentration enhances dye absorption by the fibers, consequently leading to higher K/S values (Fig. 2). The relationship is directly proportional, highlighting the importance of optimizing dye concentration for desired color strength.

In Fig. 2, a clear correlation emerges as higher carrier concentrations are observed alongside elevated K/S values. To shed light on this relationship, it is crucial to explore how carriers impact the thermal energy required for achieving

Fig. 2 Response graph for S/N ratio and means of K/S depending on temperature, time, dye concentration, carrier concentration, pH, and liquor ratio



sufficient free volume for the dyeing process. First, carriers serve as plasticizers, effectively reducing the glass transition temperature (T_g) of the fiber. This reduction, in turn, significantly influences the diffusion of dyes into the fiber matrix. Second, the plasticization effect induced by carriers brings about a range of alterations in the fiber's characteristics. Some of these alterations may lead to fiber swelling, while others may not exhibit such a response. It is worth emphasizing that fiber swelling is not necessarily indicative of improved dye diffusion into the fibers. Instead, this phenomenon can be attributed to the carrier's role in loosening the fiber structure, facilitating the penetration of dyestuffs into the fibers [42–44]. Consequently, this heightened accessibility to dye sites accelerates the rates of dye exhaustion and fixation, ultimately resulting in the observed higher K/S values.

It is clear from the data that the influence of dyeing pH and dyeing time on color strength do not follow a linear pattern. K/S values exhibit increase at pH levels of 3, 4. However, a significant decrease in K/S values is observed at pH 7 (Fig. 2). This shift can be explained by the fact that acidic conditions are recommended for PEI fabric dyeing [2].

As the liquor ratio expands from 1:10 to 1:50, the average K/S values generally decrease (Fig. 2). This reduction can be attributed to the dilution effect. When the liquor ratio is increased while maintaining constant dye concentration (owf), carrier concentration (owf), and pH, the dye concentration per unit volume in the dye bath (g/L) decreases. This diluted dye solution in unit volume of the dye bath ultimately may result in less dye absorbance by the fibers and, therefore, less dye diffusion into the fibers, which is manifested

by a decrease in K/S values [42]. Consequently, pinpointing the optimal liquor ratio becomes crucial for achieving the desired color intensity without excessive dilution. In this context, a liquor ratio of 1:30 was found to yield the highest S/N , confirming its efficacy for this particular dyeing process.

These observations provide valuable insights for optimizing the parameters and levels in PEI dyeing to get the desired K/S values and boost the efficiency and efficacy of the dyeing process. This can be accomplished by achieving the desired K/S values.

Figure 3 reveals that elevating the temperature from 100 to 140 °C adversely affects the breaking force of the fiber, indicating a decline in its tensile strength. A delta value of 4.257, as shown in Table 3, signifies that this decrease is significant. Thus, it appears that lower temperatures are more conducive for maintaining the fiber's tensile strength (higher breaking force values) during the disperse dyeing process. While higher temperatures can indeed enhance the dyeing process by improving dye diffusion rates, they also risk damaging the fiber structure if they are excessively high or sustained for extended periods [40–42]. As a result, finding the optimal dyeing temperature is a balancing act, essential for achieving the most effective dyeing outcome while preserving the fiber's mechanical properties.

The delta values of ANOVA result also prove that the most effective factors on breaking force are carrier and temperature. The other parameters such as dyeing pH, dyeing time, dye concentration, and dyeing liquor ratio possess non-linear relationship, and they are not much effective on the breaking force.

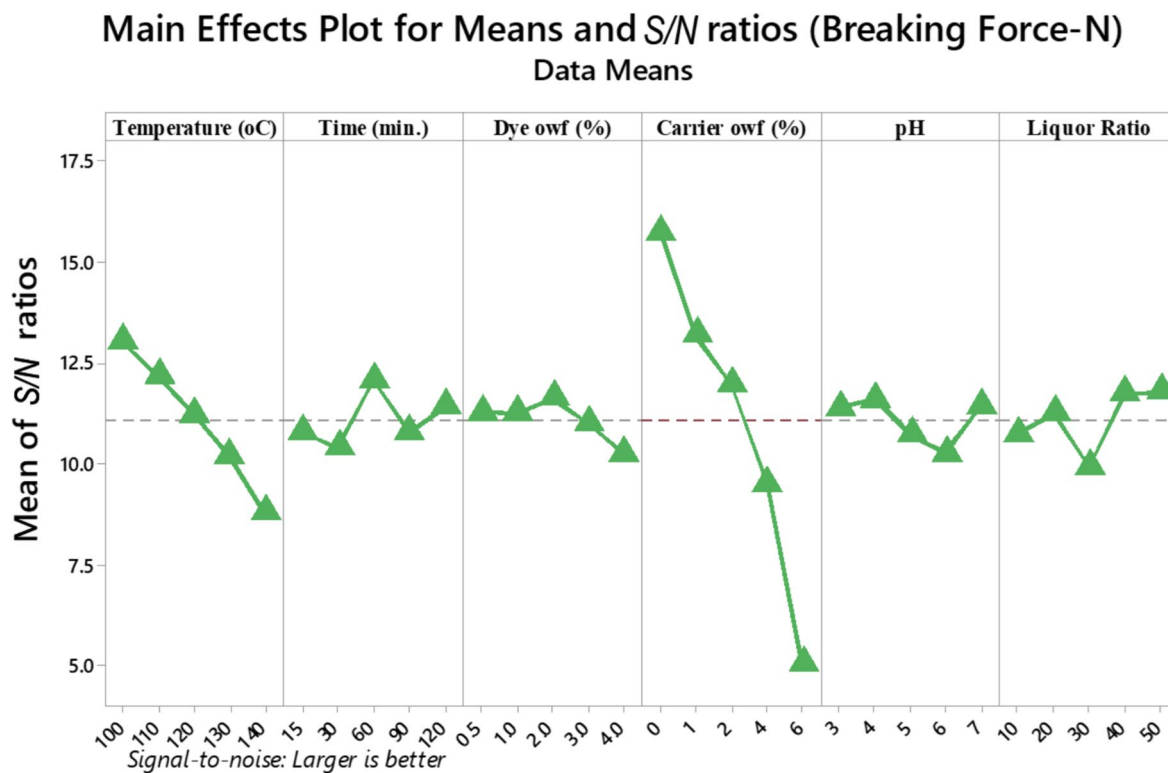


Fig. 3 Response graph for means and S/N ratio of breaking force depending on temperature, time, dye concentration, carrier concentration, pH, and liquor ratio

The influence of carrier concentration on breaking force is notably pronounced, as can be seen in Fig. 3. There is a clear downward trajectory in breaking force as the carrier concentration rises from level 1 (0.5% owf) to level 5 (6% owf). This decline is further corroborated by the S/N ratios, which plummet from 15.72 at level 1 to 5.014 at level 5 (6% owf). Both Fig. 3 and Table 3 make it evident that carrier concentration stands as the most impactful parameter affecting the breaking force of the dyed PEI samples in line with the literature [43, 44]. This suggests that attention must be paid to optimizing carrier concentration in the dyeing process to maintain the mechanical integrity of the fiber.

In the realm of experimental design, the results of all tested washing fastness tests consistently demonstrate exceptional performance. The lowest washing fastness value, recorded at 4.07, according to ISO 105-A03 grey scale ratings for the staining level of polyamide in multi-fiber, was observed in conditions where no carrier was employed. As aforementioned, the colorimetric evaluation was calculated using ISO/CIE 11664-1 (CIE 1964) supplementary standard colorimetric system using the DataColor SpectraFlash 600 Spectrophotometer with illuminant D65 and a 10° standard observer. A staining level of 4.07 is indicative of good washing fastness, signifying that even the lowest value meets commercial standards.

Based on the findings presented in Fig. 4, it can be observed that elevated temperatures, particularly at levels 4 and 5, lead to superior washing fastness. The signal-to-noise ratio reaches its maximum at level 5, with a value of 13.26. This observation suggests that increasing the dyeing temperature has a beneficial effect on the wash fastness. The highest temperature setting investigated yields the most favorable outcomes in terms of washing fastness.

The analysis of the signal-to-noise ratios suggests the presence of a non-linear correlation between the variable of time and the attribute of washing fastness. The significance of washing fastness is not substantial. In addition, it is evident from the analysis of variance (ANOVA) that the delta value is merely 0.2. However, there is a little rise observed during the 120-min dyeing procedure. The highest signal-to-noise ratio (S/N ratio) of 13.25 is seen when the recording volume is set at 5.

The impact of dye concentration on the wash fastness is seen to be negative, as increasing the concentration of dye during the dyeing process leads to a slight decrease in wash fastness. The signal-to-noise ratios (S/N ratios) pertaining to the dye concentration values suggest that increased dye concentrations (namely levels 2, 3, and 4) lead to less wash fastness, whereas the S/N ratio is maximum at level 1.

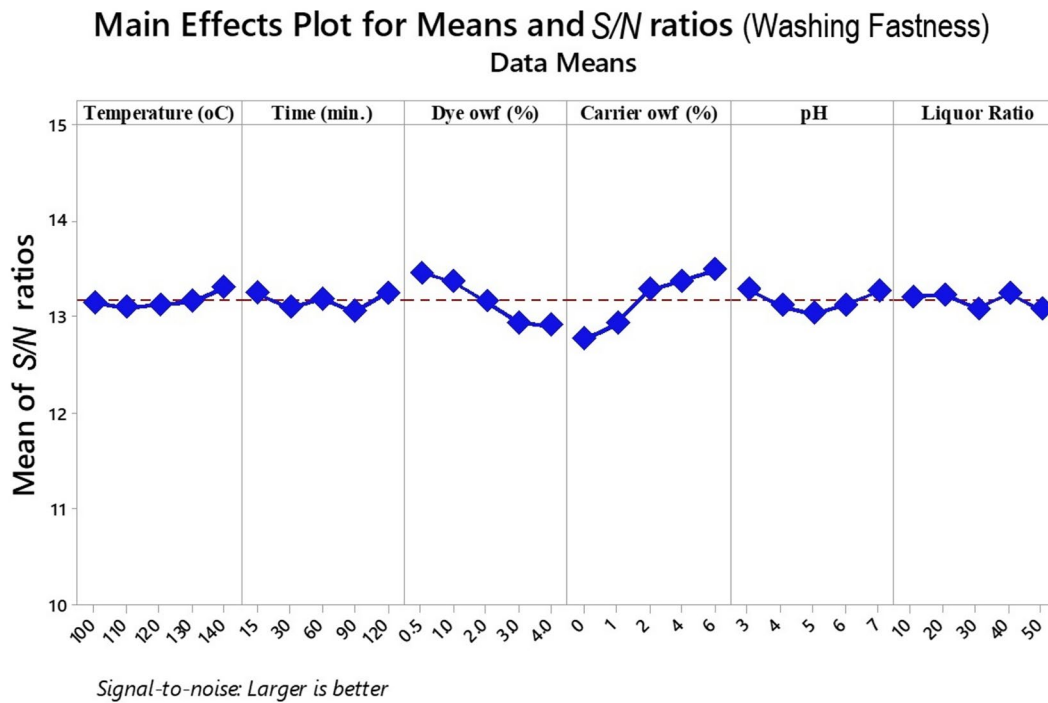


Fig. 4 Response graph for S/N ratio of washing fastness depending on temperature, time, dye concentration, carrier concentration, pH, and liquor ratio

Table 2 Signal-to-noise ratios table for *K/S* (larger is better)

Level	Temperature (°C)	Time (min)	Dye concentration (% owf)	Carrier concentration (% owf)	pH	Liquor ratio
1	21.26	22.05	14.75	18.21	23.26	23.28
2	23.06	24.31	19.95	23.49	24.70	22.81
3	23.35	21.66	25.38	23.30	22.79	24.42
4	24.61	24.23	26.92	24.67	24.40	21.41
5	23.46	23.49	28.73	26.08	20.59	23.82
Delta	3.35	2.65	13.98	7.87	4.11	3.02
Rank	4	6	1	2	3	5

Table 3 Signal-to-noise ratios table for breaking force (larger is better)

Level	Temperature (°C)	Time (min)	Dye concentration (% owf)	Carrier concentration (% owf)	pH	Liquor ratio
1	13.031	10.776	11.282	15.72	11.371	10.733
2	12.156	10.379	11.239	13.171	11.604	11.237
3	11.227	12.072	11.609	11.969	10.706	9.909
4	10.188	10.756	10.995	9.503	10.259	11.726
5	8.774	11.393	10.252	5.014	11.436	11.771
Delta	4.257	1.694	1.356	10.707	1.345	1.862
Rank	2	4	5	1	6	3

Figure 4 clearly demonstrates a noticeable trend with respect to the carrier concentration parameter. As the carrier concentration increases from level 1 [0% owf (no carrier)] to level 5 (6% owf), there is a consistent improvement on washing fastness. Increasing the carrier concentration during the dyeing process has a positive influence on wash fastness. The highest carrier concentration (level 5) results in the best wash fastness.

The *S/N* ratios for pH values and liquor ratio values suggest that variations in pH and liquor ratio within the studied range have a relatively minor impact on wash fastness. There is no strong trend in the *S/N* ratios with changing pH levels and liquor ratio levels (Fig. 4). Level 4 liquor ratio results in the highest wash fastness, but the differences are not substantial.

3.1.2 Analysis of Optimum Conditions

Determining the optimum conditions for dyeing PEI fibers, color strength *K/S*, tensile strength, and washing fastness results were examined by Taguchi analysis. According to Table 2 and response graph in Fig. 2, the optimal dyeing process parameters and their corresponding level values for *K/S* obtained are dyeing temperature 130, dyeing time 30 min, dye concentration 4% owf, carrier concentration 6% owf, pH 4 and liquor ratio 1:30 as shown in Table 2 in bold.

According to response in Table 3 and response graph in Fig. 3, the optimal dyeing process parameters and their corresponding level values for breaking force obtained are

dyeing temperature 100, dyeing time 60 min, dye concentration 2% owf, carrier concentration 0% owf (no carrier), pH 4, and liquor ratio 1:50 as shown in Table 3 in bold. Furthermore, the substantial delta value of 10.707 for the carrier concentration parameter indicates a significant variation in breaking force values across different carrier concentration levels, emphasizing the strong influence of dye concentration on this response variable. The rank value of 1 for carrier concentration confirms that, among the parameters studied, carrier concentration is the most influential factor in determining breaking force when higher breaking force values are favored. In summary, the data strongly suggests that lower carrier concentrations tend to result in significantly higher breaking force values since larger breaking force values are preferred. This observation can be a crucial factor in optimizing the dyeing process to achieve the desired breaking force in PEI fabric. Furthermore, dyeing temperature is the second significant parameter for the larger breaking force values. The delta value of 4.257 indicates a significant variation in breaking force across different temperature levels. The other parameters pH, dye concentration, liquor ratio, and dyeing time possess nearly close effects on the breaking force.

According to response in Table 4 in bold and response graph in Fig. 4, the optimal dyeing process parameters, and their corresponding level values for washing fastness obtained are dyeing temperature 140 °C, dyeing time 120 min, dye concentration 0.5% owf, carrier concentration 6% owf, pH 3, and liquor ratio 1:40 as shown

Table 4 Signal-to-noise ratios table for washing fastness (larger is better)

Level	Temperature (°C)	Time (min)	Dye concentration (% owf)	Carrier concentration (% owf)	pH	Liquor ratio
1	13.15	13.24	13.45	12.76	13.28	13.2
2	13.1	13.09	13.37	12.93	13.11	13.22
3	13.12	13.19	13.16	13.28	13.03	13.09
4	13.16	13.05	12.94	13.37	13.13	13.24
5	13.37	13.25	12.91	13.48	13.26	13.07
Delta	0.2	0.2	0.55	0.72	0.25	0.17
Rank	5	4	2	1	3	6

Table 5 Optimal parameters level and their corresponding values for dyeing process

	Unit	<i>K/S</i>		Breaking force (N)		Washing fastness	
		Level	Value	Level	Value	Level	Value
Temperature	(°C)	4	130	1	100	5	140
Time	(min)	2	30	3	60	5	120
Dye concentration	(% owf)	5	4	3	2	1	0,5
Carrier concentration	(% owf)	5	6	1	0	5	6
pH	–	2	4	2	–	1	3
Liquor ratio	–	3	30	5	50	4	40

Table 6 ANOVA table for *S/N* ratio (*K/S*)

Factors	DOF	Seq SS	<i>F</i> value	<i>p</i> value	Contribution
Temperature	4	256.2	53.54	0.0000000000000000*	2.73%
Time	4	128.8	26.92	0.0000000000000036*	1.37%
Dye (% owf)	4	7184.5	1501.29	0.0000000000000000*	76.45%
Carrier (% owf)	4	1116.8	233.37	0.0000000000000000*	11.88%
pH	4	450.5	94.13	0.0000000000000000*	4.79%
LR	4	140.7	29.41	0.0000000000000004*	1.50%
Error	100	119.6	–	–	1.27%
Total	124	9397.3	–	–	100.00%

Seq SS sequential sum of square, *DOF* degree of freedom

*Significant at $p \leq 0.05$, and *NS* not significant at $p > 0.05$

Table 7 ANOVA table for *S/N* ratio (breaking force)

Factors	DOF	Seq SS	<i>F</i> value	<i>p</i> value	Contribution
Temperature	4	39.937	189.4	0.00000000*	12.28%
Time	4	2.4800	11.76	0.00000007*	0.76%
Dye (%owf)	4	12.782	60.62	0.00000000*	3.93%
Carrier (%owf)	4	259.36	1230	0.00000000*	79.73%
pH	4	1.5840	7.510	0.00002472*	0.49%
LR	4	3.9000	18.49	0.00000000*	1.20%
Error	100	5.2710			1.62%
Total	124	325.31			100.00%

Seq SS sequential sum of square, *DOF* degree of freedom

*Significant at $p \leq 0.05$, and *NS* not significant at $p > 0.05$

in Table 5 in bold. In summary, the analysis of the *S/N* ratios for wash fastness suggests that temperature, time, dye concentration, carrier concentration, and pH have positive impacts on achieving larger (better) wash fastness values within their respective ranges. Among these parameters, higher levels (e.g., level 5 for temperature, time, and carrier concentration; level 1 for dye concentration and pH) tend to result in better wash fastness. Liquor ratio shows relatively minor differences across levels, and

its impact on wash fastness appears to be limited within the studied range.

3.1.3 Analysis of Variance (ANOVA)

ANOVA for *S/N* ratio as illustrated in Tables 6, 7, 8 is applied to evaluate which factors significantly affect the process responses. According to ANOVA table for *S/N* ratio (*K/S*) in Table 6, all the controllable factors (dyeing temperature, dyeing time, dye concentration, carrier concentration, pH, and liquor ratio) are found to be significant (p value < 0.05) for fabric *K/S* at 95% confidence level. Dye concentration (% owf) has the highest contribution (76.45%), indicating it has the most substantial influence on *K/S* values. Carrier concentration and dyeing pH also have significant contributions (11.88% and 4.79%, respectively). Dyeing temperature, dyeing time, and liquor ratio have relatively smaller but still significant contributions for *K/S* (2.73%, 1.37%, and 1.50%, respectively). For all parameters, the p values are extremely small (close to zero), indicating that all parameters significantly influence the *S/N* ratio according to *K/S* values. This analysis provides valuable insights into the factors affecting *K/S* values and can guide the optimization of dyeing processes to achieve desired outcomes in terms of color strength.

Table 8 ANOVA table for *S/N* ratio (washing fastness)

Factors	DOF	Seq SS	<i>F</i> value	<i>p</i> value	Contribution
Temperature	4	0.1566	18.63	0.000000000181019*	3.05%
Time	4	0.2032	24.17	0.0000000000000525*	3.96%
Dye (% owf)	4	1.6194	192.6	0.0000000000000000*	31.54%
Carrier (% owf)	4	2.5036	297.76	0.0000000000000000*	48.76%
pH	4	0.286	34.01	0.0000000000000000*	5.57%
LR	4	0.1558	18.54	0.0000000000000525*	3.04%
Error	100	0.2102	–	–	4.09%
Total	124	5.1348	–	–	100.00%

Seq SS sequential sum of square, *DOF* degree of freedom

*Significant at $p \leq 0.05$, and *NS* not significant at $p > 0.05$

According to ANOVA table for *S/N* ratio (breaking force) in Table 7, all the controllable factors, dyeing temperature, dyeing time, dye concentration, carrier concentration, pH, and liquor ratio, are found to be significant (p value < 0.05) for breaking force at 95% confidence level. Further, the contribution of different factors to the breaking force measurements is presented as following order: carrier concentration (79.73%) > dyeing temperature (12.28%) > dye concentration (3.93%) > liquor ratio (1.20%) > dyeing time (0.76%) > dyeing pH (0.76%). Although, carrier concentration (% owf) has the highest contribution, indicating it has the most substantial influence on breaking force values, all the factors are selected to calculate approximately the *S/N* ratio for optimum factor combination because all the factors are found significant. All the factors are found significant and, hence, are selected to calculate approximately the *S/N* ratio for optimum factor combination.

The ANOVA table for *S/N* ratio (washing fastness) in Table 8 demonstrates that all the studied parameters, including dyeing temperature, dyeing time, dye concentration (% owf), carrier concentration (% owf), dyeing pH, and liquor ratio, have a significant impact (p value < 0.05) on the *S/N* ratio according to washing fastness values at 95% confidence level. Each parameter contributes differently to the overall variability in the *S/N* ratio: carrier concentration (48.76%) > dye concentration (31.54%) > dyeing pH (5.57%) > dyeing time (3.96%) > dyeing temperature (3.05%) > liquor ratio (3.04%). In summary, dye concentration and carrier concentration are the most significant parameters for all results even though all parameters are significant.

The key component influencing washing fastness and breaking force, as well as the second most significant factor affecting color strength (*K/S*), is the concentration of carriers. However, the concentration of dye plays a crucial role in determining the intensity of color and is the second most significant factor in assessing washing fastness.

The model summary indicates that the developed models for *K/S*, breaking force, and wash fastness are highly effective in explaining and predicting the *S/N* ratios based on the studied parameters (Table 9). Both ANOVA and regression analysis will usually report measures of “goodness of fit” for the model to the data and will often provide indicators of whether the model assumptions have been met (residuals, etc.). In summary, while ANOVA tells you if there is a difference in means and which factors are responsible for variability in data, regression analysis will tell you how each predictor variable impacts the dependent variable, often with an associated prediction equation. These models exhibit high R-squared values indicating strong relationships between the parameters and the *S/N* ratios. The R-squared values for *K/S*, breaking force, and wash fastness in the ANOVA model are extremely high (98.73%, 98.38%, and 95.91%, respectively).

Table 9 Model summaries of developed models for disperse dyeing PEI fabrics

Model summary (ANOVA)	S	R-sq	R-sq(adj)	R-sq(pred)
<i>K/S</i>	1.0938	98.73%	98.42%	98.01%
Breaking force	0.2296	98.38%	97.99%	97.47%
Wash fastness	0.0458	95.91%	94.92%	93.60%
Model summary (regression analysis)				
<i>K/S</i>	2.97679	88.87%	88.31%	87.41%
Breaking force	0.4617	92.27%	91.87%	91.27%
Wash fastness	0.1046	74.83%	73.56%	71.72%

These values indicate a very strong relationship between the studied parameters and each of the dependent variables, suggesting that the model explains almost all the variability in the data. The R-squared values in the regression models are slightly lower, especially for wash fastness (74.83%). However, they are still strong for *K/S* (88.87%) and breaking force (92.27%). This means that while the regression model still explains a high degree of variance, it may not capture all the nuances that the ANOVA model does, particularly for wash fastness.

ANOVA model seems to be a better fit for the data, given the higher R-squared values and lower residual standard deviations. However, each approach provides unique insights. While ANOVA tells which factors significantly affect the dependent variable and explains most of the variability in the outcomes; regression provides a more detailed understanding of how each predictor variable influences the dependent variable, which is essential for making predictions and optimizations.

3.1.4 Confirmation of Taguchi Design of Experiment

A confirmation experiment is the final step after conducting an optimization study. This experiment aims to validate the optimal settings of the factors (variables) obtained from the optimization study. The optimal settings for *K/S*, breaking force, and washing fastness were tested in a real-world scenario to ensure that they indeed provide the best performance or output as predicted. The actual experimental results and the prediction outcomes from the developed Taguchi model were then compared with the optimum experimental outcomes as displayed in Table 10.

3.1.5 Prediction by Taguchi Mathematical Model

For all 25 trials, Eqs. (15) and (16) were employed to forecast the color strength (*K/S*), tensile properties (breaking force), and washing fastness of PEI fabric. We assessed the accuracy of the predictions made by the Taguchi-based

Table 10 The actual and predicted properties of disperse dyed PEI fabric samples by the Taguchi mathematical model

Exp	Color strength (K/S)			Breaking force (N)			Washing fastness		
	Experimental result	Predicted	Absolute error (%)	Experimental result	Predicted	Absolute error (%)	Experimental result	Predicted	Absolute error (%)
1 ^a	34.01	33.90	− 0.32	0.727	0.721	− 0.83	4.41	4.39	− 0.46
2 ^b	11.66	11.61	− 0.43	7.613	7.584	− 0.38	4.21	4.17	− 0.96
3 ^c	13.45	13.76	− 1.45	1.903	1.897	− 0.15	5.00	5.00 ^d	0

^aExperiment K/S = 130 °C, 30 min., dye 4% owf, carrier 6% owf, pH 4, liquor ratio 1:30

^bExperiment breaking force = 100 °C, 60 min., dye 2% owf, carrier 0% owf, pH 4, liquor ratio 1:50

^cExperiment washing fastness = 140 °C, 120 min., dye 0.5% owf, carrier 6% owf, pH 3, liquor ratio 1:40

^dPredicted washing fastness value was 5.18 but maximum evaluation is 5.00 according to AATCC fastness standards

mathematical model through statistical metrics such as the coefficient of determination (R^2) and mean absolute error percentage (MAE). These metrics were specifically used to compare the estimated and observed K/S values for the fabric:

$$R^2 = 1 - \frac{\sum_{i=1}^n (y_{pi} - y_{ei})^2}{\sum_{i=1}^n (y_{ei} - y_m)^2} \quad (15)$$

$$\text{MAE} = \frac{1}{n} \left\{ \left(\frac{y_{ei} - y_{pi}}{y_{ei}} \right) \times 100 \right\} \quad (16)$$

In this context, y_{ei} represents the observed experimental data, y_{pi} denotes the estimated outcomes, y_m is the average of the empirical values, and n is the count of experiments performed.

After conducting the Taguchi analyses, one of the significant observations was that the residuals, or the differences

between observed and predicted values, exhibited a normal distribution for various fabric properties as K/S (color strength), breaking force, and washing fastness. This was confirmed through an analysis of normal probability plots which is developed through a model where plots of the residuals generally fall on a straight line (Fig. 5), meaning that errors are distributed normally.

The correlation between experimental and predicted fabric properties is also graphically depicted in Fig. 6. Figure 6a shows that the R-squared value was measured at 0.987 when comparing actual and estimated fabric color strength, signifying that the Taguchi mathematical model accounts for 98.7% of the total variability in PEI fabric color strength. Similarly, the R-squared values for breaking force and washing fastness were found to be 0.984 and 0.959, respectively, indicating a high level of correlation between the experimental and predicted data [Fig. 6b, c]. Also, MAE (mean absolute error) was calculated as 0.73%, 0.45%, and 0.47% for K/S , breaking force, and washing fastness

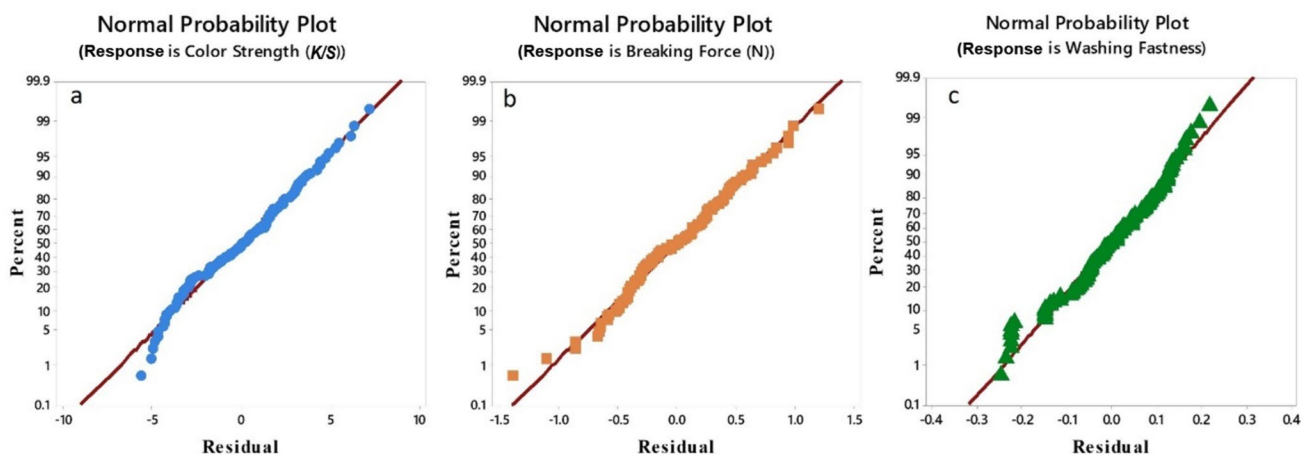


Fig. 5 Normal probability plots: **a** response is K/S , **b** response is breaking force, **c** response is washing fastness

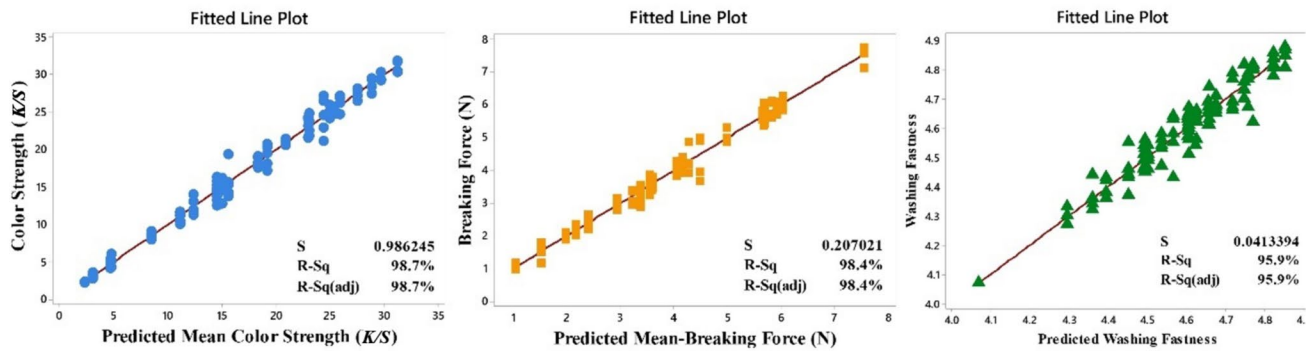


Fig. 6 Correlation between the actual and predicted values of *K/S*, breaking force, and washing fastness values

values, respectively, according to the actual experimental results, predicted results and absolute error percentages given in Table 10. This underscores the strong concordance between the predicted and actual fabric attributes according to the Taguchi mathematical model. In summary, based on R-squared values, MAE percentages, fitted line plots, and residual analyses, the developed Taguchi mathematical model exhibits a robust predictive capability and accuracy for the non-linear dyeing process.

3.2 Mathematical Model Development

Linear relationships have been established for color strength (*K/S*), breaking force, and washing fastness, correlating these results with the operational variables using Minitab 18 software (Table 11).

4 Conclusion

In summary, the study utilized the Taguchi method to streamline the dyeing process of PEI fibers with disperse dyes. Through systematic investigation of various parameters, including dye concentration and temperature, the

Taguchi method proved statistically robust and economically efficient, requiring only 25 experimental runs. Insights from parameter rank analysis emphasized the importance of factors like carrier concentration and dye concentration on key textile properties. The study developed mathematical models for quality attributes, validated their efficacy, and supported the Taguchi methodology as a robust tool for improving dyeing processes. The development and validation of mathematical models for quality attributes further underscored the efficacy of the Taguchi methodology in predicting real-world outcomes. Overall, the research highlights the utility and robustness of the Taguchi method in facilitating evidence-based advancements in textile dyeing research and industrial applications.

Table 11 Regression equations for color strength (*K/S*), breaking force (N), and washing fastness

Color strength (<i>K/S</i>)	=	$-3.35 + 0.1002 \text{ dyeing temperature } (^{\circ}\text{C}) + 0.01163 \text{ dyeing time (min)} + 5.822 \text{ dye concentration (\% owf)} + 1.137 \text{ carrier concentration (\% owf)} - 1.140 \text{ pH} - 0.0454 \text{ liquor ratio}$
Breaking force (N)	=	$11.122 - 0.03963 \text{ dyeing temperature } (^{\circ}\text{C}) - 0.00029 \text{ dyeing time (min)} - 0.2465 \text{ dye concentration (\% owf)} - 0.6530 \text{ carrier concentration (\% owf)} - 0.0556 \text{ pH} + 0.00365 \text{ liquor ratio}$
Washing fastness	=	$4.3940 + 0.00192 \text{ dyeing temperature } (^{\circ}\text{C}) + 0.000042 \text{ dyeing time (min)} - 0.08679 \text{ dye concentration (\% owf)} + 0.06112 \text{ carrier concentration (\% owf)} - 0.0018 \text{ pH} - 0.001244 \text{ liquor ratio}$

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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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