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# Sustainable Approach to Mordant-Free Natural Dyeing of Nylon Fabric with Neem Leaf Extract

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## Article

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## ABSTRACT

*The application of natural dyes in textile colouring procedures offers a viable path toward ecologically responsible and sustainable practices in the textile sector. This study was conducted to investigate the dyeing performance of nylon fabric using natural dye from neem leaf extract through a sustainable approach. Here, nylon fabric was dyed with a natural dye, neem leaf extract, without using any metallic mordant or pH buffer, which are harmful to the environment, as well as without using any natural mordant that can alter or change the actual hue of the dyed fabric. In this experiment, neem dye was extracted from neem leaves at varying concentrations using a chemical-free water extraction method. The absorption spectrum of the dye solution was measured using a UV-visible spectrophotometer. The collected grey nylon fabric was then dyed with different concentrations of the extracted dye at various temperatures, without the use of any mordant or chemicals. Following the application, the dyeing performance of the fabric was assessed by determining its colourimetric properties, including colour shade, colour strength, and colour coordinate value. Assessments of wash, water, rubbing, and light fastness performance were also carried out. Finally, FTIR analysis was performed to determine dye-fibre bonding. After analysing all the results, it was found that the dyeing performance of the fabric was excellent. The results showed that colour strength increased when the dyeing temperature and dye percentage increased. In terms of colour fastness properties, including wash, water, and rubbing, the results were excellent in each case. Light fastness was slightly less for light shades, but the results for deep shades were satisfactory. Additionally, FTIR results showed good absorption of natural dye by nylon fabric and the interaction between the dye and the fabric. The results suggest that dyeing nylon fabric with neem leaf extract, without the use of metallic mordants or a pH buffer solution, is a viable method for achieving the desired dyeing results and creating a pathway toward sustainable dyeing.*

## KEYWORDS

*nylon fabric, neem leaf, natural dye, mordant-free dyeing*

## INTRODUCTION

Before the invention of synthetic dyes, people were entirely dependent on natural dyes for dyeing clothes, and they had been using these natural dyes for many years [1-3]. Historical records indicate that ancient people utilised various natural colour shades, including red, yellow, and blue, to dye their clothing and materials [4,5]. With the invention of synthetic dyes and their commercialisation in the late 19th century, manufacturers began to utilise them due to their qualities of easy production and processing, high purity, variety, and bright shades, as well as an adequate supply [6,7]. Thus, the application of natural dyes has been significantly reduced due to the development of numerous colour variants of synthetic dyes and the

competitive production of advanced alternatives. Currently, with increasing public concern, the use of natural dyes to mitigate the environmental impact of synthetic dyes is gaining prominence. The great advantage of natural dyes is that it is biodegradable, non-toxic, and environmentally friendly in all aspects [8,9]. Although natural dyes have several limitations, including limited shade range, colour efficiency, and inadequate sources, they can be a great alternative to synthetic dyes for protecting the environment. Researchers have now focused on employing colour modification techniques to expand the natural dyes' colour palette, allowing for a wider range of colours, ranging from vivid hues to delicate hues [10,11]. This is achieved by adjusting the extraction parameters or combining different dye sources, providing textile designers with unsurpassed creative flexibility.

Natural dyes are usually extracted from plants, animals, fungi, or insects. One source of natural colouring is the neem leaf, which has numerous opportunities for potential development. The neem plant (*Azadirachta indica*), a member of the mahogany family *Meliaceae*, is also referred to as the Indian lilac, margosa, or holy tree. Like the Indian subcontinent, it grows in tropical and semitropical climates worldwide [12]. Neem leaf's primary components are minerals, calcium, phosphorus, vitamin C, carotene, protein (7.1%), and carbohydrates (22.9%). Additionally, the extract's chemical composition includes amino acids that resemble cystine, as well as fatty acids such as elcosanic, dodecanoic, and tetradecanoic, along with tyrosine, glutamic acid, alanine, aspartic acid, glutamine, and proline [13,14]. However, the primary source of green colour in the leaves of *Azadirachta indica* is a plant pigment called chlorophyll [15]. Neem leaves also contain quercetin compounds, belonging to the flavonoid group, which is the natural substance responsible for the reddish-brown colour [16,17]. Quercetin also exhibits anti-inflammatory and anticancer properties [18]. Neem leaf dye can produce different types of colours, from light yellows to deep greens, depending on the type of material or fabric being dyed, the amount of neem extract used, and other variables [15]. The most important thing is that neem leaf is available in all seasons, which overcomes the limitations of other seasonal natural dyes. It is a sustainable resource for dye production because they are also hardy and adaptable to a variety of environments [19]. There is extensive research on dyeing textile materials with natural neem leaf extract. A study on the eco-dyeing process of wool fabric using natural neem leaf dye was performed by Nabawia A. Abdel-Zaher, et al. (2019) [20]. Based on the results obtained, the author stated that the natural dye extracted from neem leaf has good potential for textile dyeing. Outstanding colour fastness results showed that the dyed wool textiles had good washing fastness characteristics. Another research work, examining the effects of various dyeing conditions on silk fabric using a natural dye extracted from neem leaves, was conducted by the same author Nabawia A. Abdel-Zaher, et al. (2018) [21]. Here, they demonstrated that neem leaf extraction can be utilised for dyeing silk fibre under various dyeing conditions, including pH values, temperature, and time, to achieve a wide range of soft hell shades. Additionally, a study on applying neem leaf to lycra filaments after water and methanolic extraction was conducted by Dr BH Patel, et al. (2014) [15]. The dyed samples showed good to moderate light and washing fastness. The sample treated with water

extract dye showed a light brown colour, and the sample treated with methanolic extract dye showed a pale to dark green colour without noticeably losing strength. Here, they dyed using a mordant, which improved colour strength. Other research studies have been conducted using neem leaf extract on cotton, silk, and hemp fabrics to dye, to enhance their antimicrobial properties [13,22,23].

Among the various synthetic polymer fibres in use today, nylon is one of the most important, which is used alone or blended with other fibres. It is mainly composed of repeated chains of high-molecular-weight amides [24]. Nylon fabric has good elastic recovery and elasticity. However, when exposed to a slight external force, it will readily wrinkle and deform. Nowadays, novelties in nylon blends, like nylon spandex, have gained popularity in sportswear and activewear. The development of high-performance nylons with enhanced properties, like moisture-wicking and antibacterial properties, expands their applications. Moreover, the textile industry has placed an increasing emphasis on sustainable practices in recent years. An attempt has been made to create environmentally friendly nylons, such as biorenewable nylon and nylon made from recycled post-consumer waste [25,26]. Nylon fabric is typically dyed with synthetic acid dyes, and most research focuses on this aspect [27]. Very little research has been conducted on dyeing nylon fibres with natural dyes [28]. The method of dyeing nylon fabric with natural dyes from onion, Lac, and turmeric using a mordant was performed by H. T. Lokhande, et al. (1999) [29]. The authors employed open-bath and high-temperature high-pressure dyeing methods, where the results of the high-temperature high-pressure dyeing method were better. There were also very good wash fastness and light fastness results. There are some studies on dyeing nylon fabric with natural dyes, including Madder, *Berberis vulgaris* root, Curcumin, and Saffron, after mordanting, plasma treatment, and UV/ozone pretreatment [30-32]. Whereas in all cases, the colour effect results were excellent.

However, to the best of our knowledge, no research has been conducted on the mordant-free and pH buffer-free colouration of nylon fabric using natural dyes extracted from neem leaves. The above-mentioned and previous studies have shown that dyeing polyamide fabric with natural dyes is typically achieved using a mordant or through surface treatment. Very few were dyed without any treatment. Metallic mordants can cause adverse environmental effects when mixed with wastewater [33,34]. Additionally, metallic and natural mordants can both alter the actual colour of dyed fabric [33,35]. When dyeing, additives are frequently used to regulate pH. After dyeing, it stays in the wash water as well. On the other hand, in the case of natural dyes, colour fastness and colour strength results are not very good during dyeing without a mordant [36,37]. That's why modern free dyeing is a challenging task, and it is more challenging when natural dyes are applied to synthetic fibres. Thus, the main objective of this research work was to investigate the dyeing performance of nylon fabric with a neem leaf-extracted natural dye, without the use of any mordant or pH buffer. Currently, in the textile and apparel sector, issues of waste management, life cycle assessment of textile products, and the chemicals used in them have become the core of sustainable practices for the circular economy [38,39,40]. Wastewater management in the textile dyeing sector, particularly after dyeing, is a major

challenge on the path to a circular economy [41]. In such cases, environmentally friendly dyes and a chemical-free dyeing process can effectively meet this challenge [42,43]. Another objective of conducting our research work was to adopt a sustainable approach towards the circular economy through a chemical-free dyeing process using natural dyes, by keeping wastewater free from contamination after dyeing. Additionally, in the case of green merchandising, it will serve as an indicator that would influence consumer purchasing decisions [44,45].

## EXPERIMENTAL PROCEDURE

In the experimental work, the neem leaf dye was first subjected to aqueous extraction, and the extracted dye was then used to dye grey nylon fabric. Various characterization techniques were employed to evaluate the dyeing performance of the nylon fabric. A schematic representation of the overall experimental procedure is shown in Figure 1.

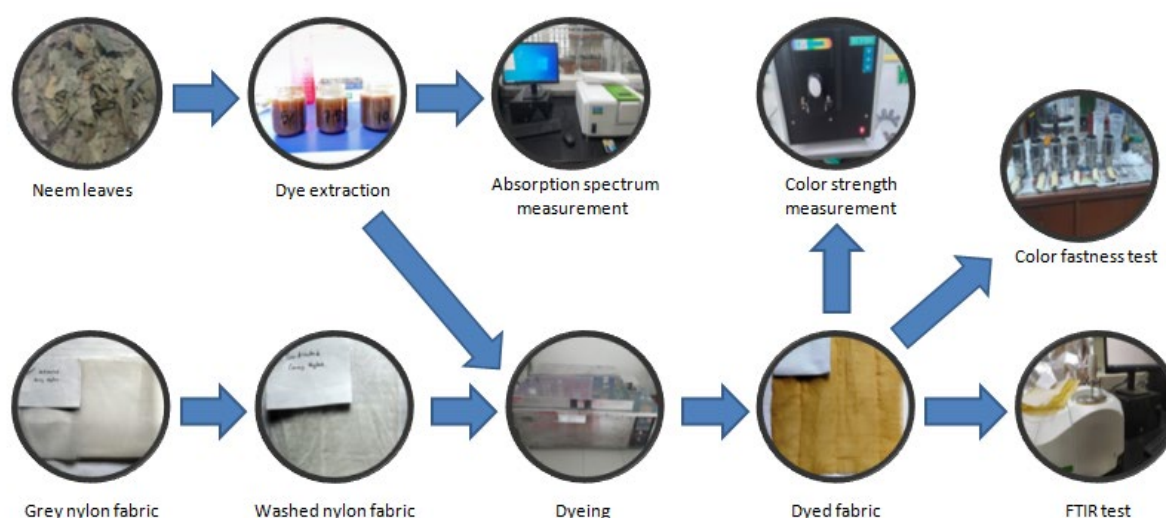


Figure 1. Schematic diagram of experimental procedure

## Materials and Chemicals

A plain weave nylon 6 fabric with 50 GSM and 170 EPI × 170 PPI, supplied by M/S. Keranigonj Enterprise, Bangladesh, was used. For the dyeing of nylon fabric, neem leaves from the neem plant (*Azadirachta indica*) were collected as a natural dye from the Meherpur district of Bangladesh. For pre-washing and cleaning of the grey fabric, soaping agent Felosan Fox LF (nonionic surfactant; Germany) was taken from the R.H. Corporation (local distributor), Bangladesh.

## Dye Extraction

The water extraction method was employed to extract the dye from neem leaves. After collection, the neem leaves were washed, cleaned, and dried in the sun for 2 days. After drying and removing all moisture, it was crushed into a powder form using an electric grinder. Extracted dye solutions were prepared at concentrations of 5%, 7.5%, and 10%. For this, 50 g, 75 g, and 100 g of neem leaf powder were taken in three beakers to prepare the dye solution. Afterwards, 1 litre of distilled water was added to each beaker and boiled for 1 hour on an electric burner at a boiling temperature. The volume of the solution was then adjusted by adding distilled water to match the initial measurements [31]. Finally, filtering was done. The concentrations of the respective resulting solutions were 5%, 7.5%, and 10% (W/V), respectively. After preparing the extracted solution, the pH of the dye solution was measured with a previously calibrated electronic pH meter. The values were 5.45 for 5% dye concentration and 5.30 and 5.25 for 7.5% and 10% dye concentration, respectively. The colour of the dye solutions varied from light brown to dark brown, depending on pH and concentration.

## Colouration Process

The collected nylon fabric was first cleaned of impurities using 2 g/L Felosan Fox LF (a nonionic surfactant, Germany) as a soaping agent at 60 °C for 30 minutes. The fabric was subsequently dyed for 60 minutes at different temperatures (70, 80, 90, and 100 °C) using 5%, 7.5%, and 10% concentrations of the extracted dye with a material-to-liquor ratio of 1:40 to achieve optimum dye exhaustion. The standard dyeing time of 60 minutes was set to achieve equilibrium and maximise dye absorption [46,47,48]. Here, the extract was directly used without any dilution [49]. At the beginning of the process, the fabric was immersed in the dye bath at room temperature (25 °C). After that, the temperature was progressively increased, and dyeing was continued for the specified duration. After dyeing, each sample was carefully washed in cold water to remove any unfixed colour and then allowed to air dry.

## Sample Marking

Samples were marked based on the dyeing temperature and the extracted percentage of dye used.

An, Bn, Cn, Dn

Here, A denotes dyeing at 100°C, B at 90°C, C at 80°C, and D at 70°C. n = 1, 2, 3; If the value of n is 1, the dye extraction percentage will be 5%; if the value of n is 2, the extraction percentage will be 7.5%; and if the value of n is 3, the extraction percentage will be 10%. Examples of sample codes for understanding: A1 indicates dyeing was performed with 5% dye at 100 °C, and B2 indicates dyeing was performed with 7.5% dye at 90 °C. The following Table 1 lists the code along with the dyeing parameters for each sample-

Table 1. Sample identification code along with dyeing parameter

| Serial no | Sample code | Extraction percentage | Dyeing parameter  |
|-----------|-------------|-----------------------|-------------------|
| 01        | A1          | 5%                    | Temperature-100°C |
| 02        | A2          | 7.5%                  | Time- 60 minutes  |
| 03        | A3          | 10%                   | M:L- 1:40         |
| 04        | B1          | 5%                    | Temperature-90°C  |
| 05        | B2          | 7.5%                  | Time- 60 minutes  |
| 06        | B3          | 10%                   | M:L- 1:40         |
| 07        | C1          | 5%                    | Temperature-80°C  |
| 08        | C2          | 7.5%                  | Time- 60 minutes  |
| 09        | C3          | 10%                   | M:L- 1:40         |
| 10        | D1          | 5%                    | Temperature-70°C  |
| 11        | D2          | 7.5%                  | Time- 60 minutes  |
| 12        | D3          | 10%                   | M:L- 1:40         |

## CARACTERISATION

### Absorption Spectrum Measurement of the Extracted Solutions

Utilising a Lambda 365 Double Beam Spectrophotometer (Parkin Elmer, USA; software version: UV Express version 4.1.3) with a bandwidth of 1.0 nm, the absorption spectrum of the aqueous extracted neem leaves in the UV-visible range (200–700 nm) was assessed to determine the optical density of the extracted solution. According to the Beer-Lambert law (qualitative analysis), this function applies [50].

### Colour Strength and Related Parameters Measurement of Dyed Fabrics

In illuminant D65, at 10° standard observer, a Premier Colourscan spectrophotometer (model: SS5100H, India) was used to measure the K/S values and the CIE LAB coordinates ( $L^*a^*b^*$ ) of the dyed nylon fabrics. Equation 1 illustrates the Kubelka-Munk formula used to measure this colour strength [51].

$$K/S = (1-R)^2/2R \quad (1)$$

Where K is the dye absorption coefficient, S is the dye scattering, and R is the reflectance percentage.

### Determination of Colour Fastness Properties

According to ISO 105-C03, a wash fastness tester was used to assess the colourfastness of dyed fabric samples against washing. The samples were washed for 30 minutes at 60 °C in a 5 g/l ECE soap and 2 g/l soda ash solution, and a grayscale was used to assess the level of staining and colour change on the dyed fabric.

An oven and acrylic resin plates were used in accordance with ISO 105-E01 guidelines to test the colourfastness of dyed fabric samples against water. First, 4 × 10 cm dyed samples were stitched with multifibre fabric and then immersed in distilled water. After removing excess water, the wet samples were mounted on acrylic plates and placed in an oven at 37 ± 2 °C for 4 hours. Finally, the samples' colour staining and colour change were rated by evolving the grey scale.

Rubbing fastness testing was performed using a Crockmeter (Model: M238AA, Brand: AATC Standard Instrument) in accordance with the ISO 105-X12 method. The process was carried out by rubbing the dyed samples on white crocking cloth, in both dry and wet conditions. Through a cork meter, dyed samples were rubbed on a white crocking cloth, and the collected samples were rated by evolving the grey scale.

In compliance with the ISO 105 B02 standard procedure, light fastness tests were conducted in this study using blue wool reference samples and a mercury-blinded tungsten lamp. Samples were evaluated twice: once during the test and once after the test. A light fastness rating was assigned to each sample according to the Blue Wool Standard, which indicates the corresponding degree of colour change.

### Chemical Characterisation by FTIR

Using an FTIR spectrophotometer (Brand: Perkin-Elmer, USA) of UATR-IR Technologies, the specific functional groups or chemical bonds that might form between nylon fibre and neem dye after dyeing were identified. With the scanning area range between 4000-400 cm<sup>-1</sup>, the data were plotted into the spectrum. The neem leaf powder and the dyed and undyed nylon fabrics were subjected to an FTIR spectra analysis in the reflection mode.

## RESULTS AND DISCUSSION

### Absorption Spectrum Analysis of Extracted Solutions

To determine the maximum absorbance at a specific wavelength, the absorbance of the extracted solutions was measured over a wavelength range of 200–700 nm. Any colour's absorption curve displays peaks at its predominant wavelength, which identifies its primary component. The highest absorbance, 5.07 at the peak of  $\lambda_{\text{max}}$  451 nm, was observed in the absorbance spectrum of a 10% dye solution. At the peak of  $\lambda_{\text{max}}$  433 nm, the 7.5% dye solution showed the highest absorbance of 4.94, while 5% dye solution showed the highest absorbance peak of 4.86 at  $\lambda_{\text{max}}$  420 nm in the visible range. That is represented in Figure 2. Three high peaks of absorbance (3.765, 3.402, and 3.569) were observed at  $\lambda_{\text{max}}$  320 nm, 370 nm, and 400 nm in recent

research by Abd El Aty et al. (2018), while the extraction made from neem leaves was taken into consideration [13]. Furthermore, research by Dr BH Patel et al. (2014) demonstrated that a green hue with a noticeable yellow portion is indicated at a higher peak of 430 nm [15]. According to the test results, the extracted solutions exhibited the highest absorbance value between 420 and 460 nm; therefore, the dye solutions may appear green-yellow in colour. Compared to the other extracted solutions, the 10% extracted solution's maximum absorbance value indicates that it would have the highest dye concentration [52]. Additionally, its aqueous content was lowered during the extraction procedure. On the other hand, the visible spectra of the extracted 10%, 7.5%, and 5% dyes showed additional small absorption peaks at 650–700 nm. This peak could be caused by plant pigments, such as chlorophyll, which are typically assessed using a UV/visible spectrophotometer at 654 nm, as reported in research [15,53].

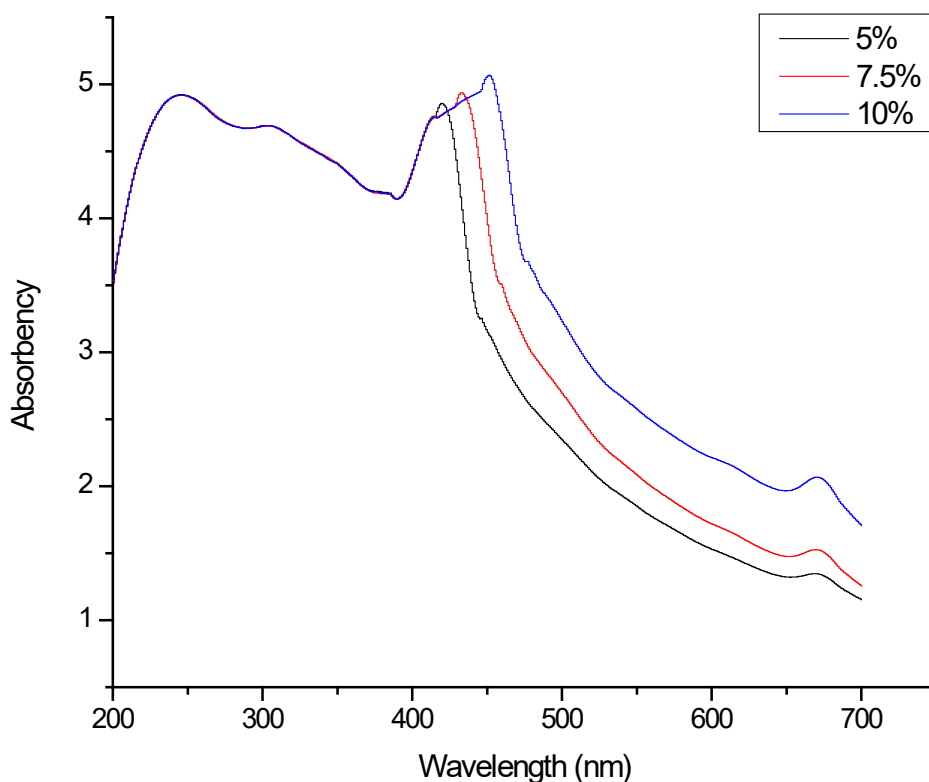


Figure 2. Uv-vis absorption spectrum of neem leaf extract

### Colour Strength Analysis of Dyed Fabrics

Fabric dyed with neem leaf extract at different temperatures with different percentages of extracted dye produced different colour shades. It is generally observed that increasing the dye percentage results in fabrics

with brown to brownish-yellow shades, whereas decreasing the dyeing temperature leads to a shift from darker to lighter shades. The K/S value provides information about the amount of dye present in the fabric and how effectively it scatters and absorbs light [54]. Stronger colours are typically indicated by higher K/S values. Lower K/S values, on the other hand, might indicate a softer or less vibrant colour. According to the test results, the nylon samples dyed with 10%, 7.5%, and 5% dye concentrations at 100°C exhibited the highest colour strength values (6.203, 5.745, and 5.399, respectively) compared to the other samples. On the other hand, the lowest colour strength values of 5.109 and 5.063 were shown by the D1 and D2 samples, which were dyed with 5% and 7.5% dye at a low temperature of 70°C (Figure 3). According to the test results, the sample's colour strength value decreased as the dyeing temperature and percentage of dye decreased, but it increased as the temperature and percentage of dye increased. Thus, by raising the dyeing temperature and using a higher concentration of dye extract, it is possible to increase the colour strength or shade when dyeing nylon fabric with neem leaf extract. The colour coordinate values of nylon fabric dyed with neem leaf extract are displayed in Table 2. Colour coordinate values specify the phenomenon of colour according to its primary colour or main wavelength. It indicates the position of the colour on the colour wheel, as well as colour hue and chroma [55,56]. This chart indicates that different shades of the samples are produced for a combination of different values of lightness, red/green coordinate, yellow/blue coordinate, chroma, and hue. Whereas samples A3, B3, C3, and D3 dyed with a high percentage of dye had higher chroma and hue values, and lower lightness values, compared to samples dyed with a low percentage of dye. Therefore, the samples in each group dyed with a high percentage dye were deeper shades than the others.

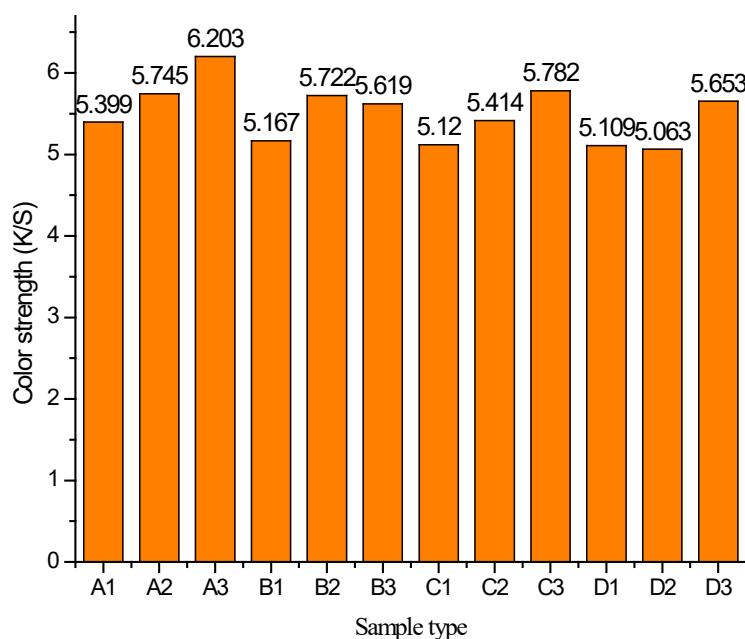


Figure 3. Color strength chart of dyed sample

Table 2. Colour coordinate value of dyed samples

| Sample | L*     | a*     | b*     | c*     | H*     |
|--------|--------|--------|--------|--------|--------|
| A1     | 60.412 | 3.822  | 18.842 | 19.226 | 78.502 |
| A2     | 60.551 | 2.606  | 21.448 | 21.606 | 83.039 |
| A3     | 58.389 | 2.072  | 27.258 | 27.337 | 85.619 |
| B1     | 63.669 | 3.172  | 18.039 | 18.316 | 79.995 |
| B2     | 64.328 | 1.212  | 21.872 | 21.906 | 86.793 |
| B3     | 61.772 | 1.076  | 28.107 | 28.128 | 87.772 |
| C1     | 65.956 | 1.494  | 19.586 | 19.643 | 85.604 |
| C2     | 66.272 | 1.208  | 20.195 | 20.231 | 86.542 |
| C3     | 64.574 | 0.665  | 27.734 | 27.742 | 88.591 |
| D1     | 69.060 | -2.041 | 22.848 | 22.939 | 95.139 |
| D2     | 69.869 | -1.507 | 21.625 | 21.677 | 94.021 |
| D3     | 68.658 | -2.815 | 29.652 | 29.785 | 95.457 |

Note: L\*=lightness value, a\*=red/green coordinate value, b\*=yellow/blue coordinate value, c\*=chroma value, H\*=hue value

Analysis of Colour Fastness Properties

The ability of a dyed or printed textile material to resist colour fading or bleeding when exposed to different external factors, such as washing, rubbing, light, perspiration, water, and chemicals, is known as colour fastness [57,58]. The wash, water, rubbing, and light fastness characteristics of nylon fabric treated with neem leaf extract are displayed in Table 3 below. All dyed fabrics showed very good to excellent wash fastness. The results were very good both in terms of colour change and colour staining. It has been established that the dye's molecular weight, shape, and number of functional groups, along with the interactions between the dye and fibre (Including Chemical bonding, hydrophobic interactions, and electrostatic attributes), all influence the wash fastness property of the dye [59]. Similar to wash fastness, the water fastness results for each sample, in terms of colour change and colour staining, were good to excellent. Which were between 4/5 and 5 on the grayscale rating. It is also noted that the number of functional domains on the dye molecule causes an increase in the amount of complex formation that occurs inside the fibre structure, which may influence the water fastness property [28].

Every sample showed excellent results in terms of rubbing fastness. The results were very similar in both dry and wet conditions. On the other hand, the light fastness property was satisfactory. The light fastness of fabric dyed with 5% and 7% extracted dye was slightly lower than that of fabric dyed with 10% extracted dye, which may be mainly due to the light shade. The light fastness of dyed fabrics with light shades was relatively lower than that of dyed fabrics with dark shades. The majority of natural dyes are less stable in light than the

best synthetic dyes [33]. Light breaks chemical bonds in dyes, which is why degradation takes place; as a result, old historical textiles often display colours that differ from their original shades. However, the obtained results showed better than light fastness of nylon fabric dyed with natural dyes using artificial mordants and UV treatment of Khaled Elnagar, et al. (2014) [32] and showed almost equal results as the light fastness rate of nylon fabric dyed with madder by applying artificial mordant, done by M. Sadeghi, et al. (2015) [30]. The light fastness can be increased by increasing the percentage of dye. In general, all fastness properties indicate that the overall fastness performance of the dyed fabric was good enough.

Table 3. Colour fastness results to wash, water, rubbing, and light

| Colour fastness |                 | Sample code |     |     |     |     |     |     |     |     |     |     |     |
|-----------------|-----------------|-------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
|                 |                 | A1          | A2  | A3  | B1  | B2  | B3  | C1  | C2  | C3  | D1  | D2  | D3  |
| Wash            | Colour change   | 5           | 4/5 | 4/5 | 5   | 4/5 | 4/5 | 5   | 5   | 4/5 | 5   | 5   | 4/5 |
|                 | Ae              | 4/5         | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
|                 | Cn              | 4/5         | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
|                 | Colour staining | Nn          | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
|                 | Pr              | 4/5         | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
|                 | Ac              | 4/5         | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
|                 | Wl              | 4/5         | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 5   | 5   | 4/5 |
| Water           | Colour change   | 5           | 4/5 | 4/5 | 5   | 4/5 | 4/5 | 5   | 4/5 | 4/5 | 5   | 5   | 4/5 |
|                 | Ae              | 4/5         | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
|                 | Cn              | 4/5         | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
|                 | Colour staining | Nn          | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
|                 | Pr              | 4/5         | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
|                 | Ac              | 4/5         | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
|                 | Wl              | 4/5         | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
| Rubbing         | Warp direction  | Dry         | 4/5 | 4   | 4/5 | 4   | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
|                 | Wet             | 4           | 4   | 4   | 4/5 | 4   | 4   | 4   | 4   | 4   | 4   | 4   | 4   |
|                 | Weft direction  | Dry         | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
|                 | Wet             | 4/5         | 4   | 4/5 | 4/5 | 4/5 | 4   | 4   | 4   | 4   | 4/5 | 4   | 4   |
| Light           |                 | 2/3         | 3   | 4   | 2/3 | 2/3 | 3/4 | 2   | 2/3 | 3   | 2   | 2   | 3/4 |

Note: Ae=Acetate, Cn=Cotton, Nn=Nylon, Pr=Polyester, Ac=Acrylic, Wl=Wool

## FTIR Analysis

FTIR analysis of neem leaf powder in the spectral range 400–4000  $\text{cm}^{-1}$ , the number of major peaks lies between 3292  $\text{cm}^{-1}$ , 2948  $\text{cm}^{-1}$ , 2177  $\text{cm}^{-1}$ , 1719  $\text{cm}^{-1}$ , 1605  $\text{cm}^{-1}$ , 1366  $\text{cm}^{-1}$ , 1243  $\text{cm}^{-1}$ , 1036  $\text{cm}^{-1}$ , and 477  $\text{cm}^{-1}$  (Figure 4).

The hydroxyl group (bonded) is represented by the broad band ( $3600\text{--}3000\text{ cm}^{-1}$ ). Aliphatic  $\text{CH}_3$  and  $\text{CH}_2$  could be responsible for the band at about  $2900\text{ cm}^{-1}$  [52]. The presence of an aromatic aldehyde's C–H stretching vibration in neem powder is indicated by the band at  $2948\text{ cm}^{-1}$ , which is supported by the findings of Noorjahan et al (2015) report [60]. It is usually possible to clearly see the presence of alkyne or nitrile groups in the bands between  $2800$  and  $2000\text{ cm}^{-1}$  because these absorptions are normally free of other absorption [52]. The stretching vibration of carbonyl functional groups, such as ketones, aldehydes, and carboxylic acids, may be responsible for the peaks at  $1719\text{ cm}^{-1}$  [61]. Similarly, the C=C group of an aromatic ring may be responsible for the bands ranging from  $(1638\text{--}1484)\text{ cm}^{-1}$ . The  $\text{CH}_3$  and  $\text{CH}_2$  bands at  $(1433\text{--}1366)\text{ cm}^{-1}$  and the present bands at  $(1300\text{--}1000)\text{ cm}^{-1}$  indicated the C–O group, while the presence of C–H deformation of an aromatic proton was indicated by the bands below  $900\text{ cm}^{-1}$ . This verified that the neem extract contained flavonoid compounds, indicating that the bands in the  $1650\text{--}1050\text{ cm}^{-1}$  range corresponded to the flavonoid skeleton [52].

Conversely, the FTIR spectra of the undyed nylon fabric with A1, A2, and A3 samples dyed with neem leaves alone were analysed; the results are shown in Figure 5. In the grey nylon FTIR Spectrum, the amine stretching absorption peak at  $3298\text{ cm}^{-1}$ , the amide carbonyl absorption peak at  $1634\text{ cm}^{-1}$ , and the carboxylic acid absorption peak at  $1719\text{ cm}^{-1}$  were normalised with respect to the C–H stretching peak at  $2933\text{ cm}^{-1}$  [31]. Moreover, Strong C–H stretching and bending vibrations of  $\text{CH}_2$  groups, vibrations of  $\text{CH}_3$  groups in relation to polyamide terminal chains, and C–N stretching were detected at  $2862$ ,  $2933$ ,  $1462$ , and  $1261\text{ cm}^{-1}$  [62,63]. After the nylon was dyed with neem leaf extract, peaks at  $1719\text{ cm}^{-1}$  were shifted and lifted, indicating the bending and stretching vibrations of the CHO groups, which may suggest the successful complex formation between the dye and the nylon. Furthermore, the peaks of the polyamide's amine stretching and strong C–H stretching shifted from  $3298\text{ cm}^{-1}$  to  $3296\text{ cm}^{-1}$  and from  $2862\text{ cm}^{-1}$  to  $1867\text{ cm}^{-1}$  after the neem dye was applied. This might be due to a resonance between the polyamide chains and the C=C groups of the powdered neem leaf. The lifted and shifted peaks suggested good dye adsorption, dye particle deposition, and dye-fibre interaction, indicating good dyeing performance.

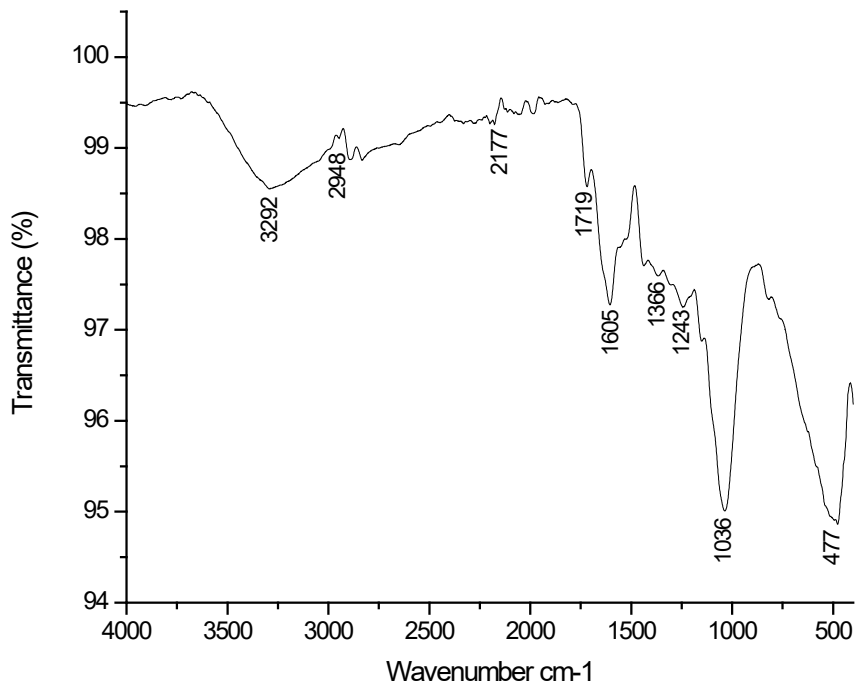


Figure 4. FTIR Spectrum of neem leaf powder

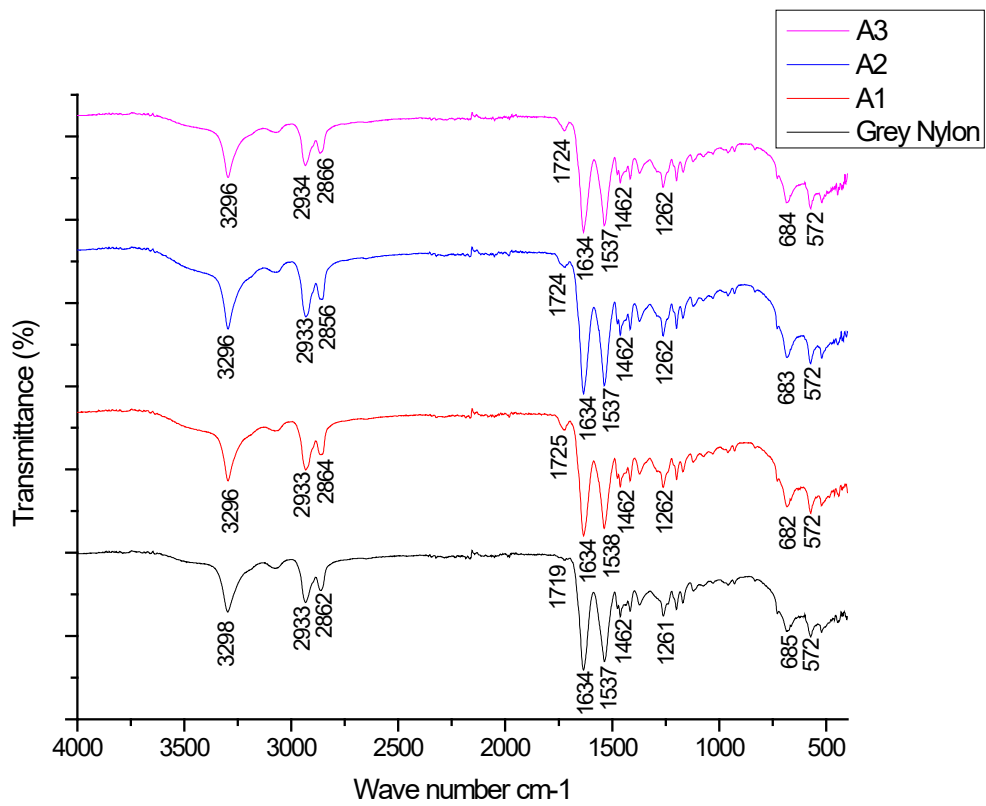


Figure 5. FTIR spectrum of grey nylon and dyed nylon with 5% (A1), 7.5% (A2), and 10% (A3) extracts

## CONCLUSION

The main intent of the research was to contribute to the expanding field of sustainable research practices by investigating the dyeing performance of a natural plant extract from neem leaves on nylon fabric, without the use of any mordant or pH buffer. In this experiment, excellent colour shades were produced. Whereas the colour strength value of the sample increased as the temperature and dye percentage increased, it decreased as the dyeing temperature and dye percentage decreased. Therefore, more colour strength can be achieved by increasing the dyeing temperature and using a higher concentration of dye extract. Colour coordinate values also showed that samples dyed with a high percentage of dye were deeper shades than the others. Colour fastness properties to wash and water were excellent in each case, which was between 4/5 and 5 on the grayscale rating. Every sample displayed outstanding rubbing fastness results. In both dry and wet conditions, the outcomes were remarkably similar. In terms of light fastness, it was slightly lower for light shades but was good for deep shades. Therefore, light fastness can be increased by increasing the percentage of dye. FTIR results indicated good absorption of natural dyes by polyamide, dye particle deposition into the fabric, and the interaction between the dye and the fibre. The findings indicate that outstanding fastness properties and colourimetric values can be achieved by applying natural dye from neem leaves to synthetic materials like nylon without the use of a mordant. Neem leaves have the potential to be utilised as a natural dye source, which is readily available throughout all seasons. In a nutshell, natural dyes offer a sustainable and environmentally beneficial alternative to traditional synthetic dyes in the dyeing of textile materials. This study will support a more responsible future for textile production by utilising mordant-free natural dyes in fabric dyeing processes.

### *Author Contributions*

Conceptualisation– Azom MG and Uddin MZ; methodology–Azom MG and Uddin MZ; formal analysis– Azom MG and Uddin MZ; investigation– Azom MG and Uddin MZ; resources–Azom MG; writing-original draft preparation–Azom MG; writing-review and editing– Azom MG and Uddin MZ; visualisation– Azom MG; supervision–Uddin MZ. All authors have read and agreed to the published version of the manuscript.

### *Conflicts of Interest*

The authors declare no conflict of interest.

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