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Comparative Study of Dyeing Performance of Plasma-Treated and Untreated Cotton Fabrics Dyed with Almond Leaf Extract (*Terminalia catappa L.*)

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Abstract:
This study investigated the effects of plasma treatment on the dyeing performance of cotton fabrics dyed with a natural dye extracted from almond leaf (Terminalia catappa L.) with a material-to-liquor ratio (1:20) for 20 minutes using the microwave-assisted extraction method. The extracted dye liquors were applied to plasma-treated and untreated cotton fabrics. Dielectric barrier discharge (DBD) plasma treatment was performed at atmospheric pressure at three different voltages (10, 15, and 20 kV) and three different frequencies (15, 20, and 25 kHz). Colour characteristics were evaluated for colour strength (K/S), CIELAB colour coordinates values, and colour fastness to washing, rubbing, and light. Among the dyed samples, sample PA22 showed the highest K/S value (16.1). The dyed fabrics exhibited predominantly yellowish shades, with fair to moderate washing fastness, moderate to good resistance to staining on adjacent cotton fabric, and moderate light fastness (grade 3). Wet rubbing fastness ranged from fair to moderate, while dry rubbing fastness ranged from fair to good. These findings indicate that dielectric barrier discharge (DBD) plasma treatment affects the dyeing performance of cotton fabrics dyed with almond leaf extract.

Keywords: Natural dye, Almond leaf (Terminalia catappa), Microwave-assisted extraction, Dielectric Barrier Discharge (DBD), Cotton fabric, dyeing performance

1. Introduction

Nowadays, sustainable, eco-friendly natural dyes have received increasing attention in textile colouration. Numerous studies have focused on extracting natural dyes from various plant sources and applying them to cotton and silk fabrics. Among available plant-based dye sources, almond leaves (*Terminalia catappa* L.) are promising natural dyes [1] and contain high levels of tannins, flavonoids, and other phenolic compounds with colouring properties [2]. Almond leaves are economical, readily available, and renewable agricultural materials that can be used for ecological textile colouration, in addition to their dyeing properties [3]. However, natural dyes often have low dye affinity, weak colour strength and poor colour fastness [4]. Conventional mordanting is widely used to improve the affinity of natural colourants for textile fibres. Aluminium salts, especially alum, are commonly used to enhance colour development and fastness [4], but may create environmental and health risks [5].

Krisnawati et al. [6] examined almond leaves as a source of natural dye powder for cotton fabrics, using various extraction solvents and alum mordanting methods. The authors found that colour characteristics varied with the extraction solvent. These findings provide a basis for further research into almond leaf extract as a natural colouring material for cotton fabrics.

The extraction process plays an important role in determining the quality of natural dye liquor. Microwave-assisted extraction is an alternative to conventional methods, as microwave energy enables rapid heating and helps release colour compounds from plant materials [7]. Repon et al. reviewed natural dyes and mordants for textile applications and discussed different approaches to improving dyeing performance, including plasma, microwave and other surface modification techniques. The authors highlighted the importance of developing alternative dyeing methods that enhance colour performance while reducing dependence on conventional chemical auxiliaries [8].

Dielectric barrier discharge (DBD) plasma treatment has obtained considerable attention as a surface-modification technique for textile materials. Plasma comprises energetic electrons, ions, excited molecules, and reactive species that interact with fibre surfaces. Depending on the treatment conditions and gas composition, these interactions may result in surface cleaning, etching, and chemical functionalization [9].

Haji and Naebe reviewed plasma treatment in natural dyeing, given its potential to enhance dye uptake and reduce the need for additional chemicals in textile colouration. The authors examined the effects on various textile fibres and natural dye systems [10]. Zhang et al. also reviewed surface-modification strategies for enhancing the functional properties of cotton dyed with natural colourants, highlighting plasma and microwave technologies as ways to improve dyeing performance [11].

The effectiveness of plasma treatment may depend on operating conditions, as excessive treatment may cause undesirable changes in fibre surfaces. Therefore, selecting appropriate plasma conditions is essential to improving the dyeing performance of plasma-treated cotton fabric [12]. Accordingly, this study investigates DBD plasma treatment as an alternative surface-modification technique for mordant-free dyeing of cotton fabric using microwave-assisted almond leaf (*Terminalia catappa* L.) extract. The plasma treatment was conducted at three different voltages (10, 15, and 20 kV) and three different frequencies (15, 20, and 25 kHz) for 1 min, using a 3 mm electrode gap and a polycarbonate dielectric barrier. After plasma treatment, the cotton fabrics were dyed directly without a mordant. This study evaluates the dyeing performance of plasma-treated and untreated fabrics in terms of their colour characteristics, including color strength (K/S) and CIELAB color coordinate values, as well as their colour fastness properties to washing, rubbing, and light.

2. Material and Method

2.1 Materials

In this study, Fresh almond leaf (Terminalia catappa L.) was used as the natural dye source and collected from the campus of Yangon Technological University. Plain-woven 100% cotton fabric was used as the textile substrate. Potassium aluminum sulfate (alum) was selected as the mordant to improve interaction between the natural colourant and cotton fabric.

2.2 Preparation and Microwave-Assisted Extraction of Natural Dye from Almond Leaf (Terminalia catappa L.)

The collected leaves were thoroughly washed to remove dust and other foreign materials, then sun-dried until completely dry. The dried leaves were ground and sieved using a laboratory sieve shaker with a 75 µm sieve. A predetermined amount of almond leaf powder was mixed with distilled water at a material-to-liquor ratio of 1:20 for 20 minutes. After extraction, the solutions were cooled, filtered, and then used for subsequent dyeing operations.

2.3 Plasma Treatment of Cotton Fabrics

The surface of the cotton fabric was modified using a dielectric barrier discharge (DBD) plasma treatment comprising an AC high-voltage supply, electrodes and a polycarbonate dielectric barrier. The electrode gap was set to 3 mm. Plasma treatment was conducted at three different voltages (10, 15, or 20 kV) and three different frequencies (15, 20, and 25 kHz) for 1 min at room temperature and atmospheric pressure. After that, the plasma-treated fabrics were removed from the discharge region and dyed to investigate the influence of surface modification on dye uptake and colour characteristics.

2.4 Dyeing Process of Pre-mordanted and Plasma-treated Cotton Fabrics

Before dyeing, cotton fabric was scoured with 2% (o.w.f.) soda ash and 2% (o.w.f.) washing agent at 95–100°C for 60 minutes using a material-to-liquor ratio of 1:20. After scouring, the fabric was rinsed and dried at room temperature. The scoured cotton fabric was then pre-mordanted with 5% (o.w.f.) alum at 60°C for 30 minutes, maintaining a material-to-liquor ratio of 1:20. The mordanted fabric was rinsed and dried before dyeing. In this study, the pre-mordanted fabric was used as the untreated reference sample. Untreated fabrics were then dyed using a jigger dyeing machine at 60°C for 60 minutes with a material-to-liquor ratio of 1:20. Plasma-treated fabrics were dyed under the same conditions. After that, dyed fabrics were rinsed and dried at room temperature. Designations of the dyed cotton fabrics are provided in Table I.

Table I. Designation of The Dyed Cotton Fabrics

<table>
<tr><th>Sr No.</th><th>Sample Code</th><th>Extraction Time (min)</th><th>Extraction Ratio (M:L)</th><th>Particle Size (µm)</th><th>Voltage</th><th>Frequency</th><th>Gas Type</th><th>Dyeing (M:L)</th></tr>
<tr><td>1.</td><td>Untreated</td><td rowspan="10">20</td><td rowspan="10">1:10</td><td rowspan="10">75</td><td>-</td><td>-</td><td>-</td><td rowspan="10">1:20</td></tr>
<tr><td>2.</td><td>PA_{11}</td><td>10</td><td rowspan="3">15</td><td rowspan="9">air</td></tr>
<tr><td>3.</td><td>PA_{12}</td><td>15</td></tr>
<tr><td>4.</td><td>PA_{13}</td><td>20</td></tr>
<tr><td>5.</td><td>PA_{21}</td><td>10</td><td rowspan="3">20</td></tr>
<tr><td>6.</td><td>PA_{22}</td><td>15</td></tr>
<tr><td>7.</td><td>PA_{23}</td><td>20</td></tr>
<tr><td>8.</td><td>PA_{31}</td><td>15</td><td rowspan="3">25</td></tr>
<tr><td>9.</td><td>PA_{32}</td><td>20</td></tr>
<tr><td>10.</td><td>PA_{33}</td><td>25</td></tr>
</table>

2.5 Evaluation of the Dyeing Performance of Dyed Cotton Fabrics

Colour characteristics (colour strength (K/S) and CIELAB colour coordinates (L*, a*, b*, C*, and h˚) of dyed cotton fabrics were measured using a CIELAB spectrophotometer. Colour fastness to washing, rubbing, and light was determined in accordance with ISO 105-C06, ISO 105-X12, and AATCC standards, respectively [13].

3. Results and Discussions

Define abbreviations and acronyms the first time they are used in the text, even after they have been defined in the abstract.

3.1 Comparative Effects of Untreated and Plasma Treatment on the Colour Characteristics of Dyed Cotton Fabrics

The results showed that plasma-treated fabric improved colour strength compared with untreated fabric. The K/S values of all plasma-treated dyed fabrics ranged from 14.10 to 16.10, whereas the untreated fabric exhibited a K/S value of 13.62. Among the plasma-treated dyed fabrics, sample PA22 (15 kV and 20 kHz) showed the highest colour strength (K/S = 16.10), followed by PA32 (20 kV and 20 kHz) with a K/S value of 15.95. The increased colour strength may be attributed to surface modifications induced by reactive species generated during air plasma treatment. These species can promote surface oxidation and etching, changing surface morphology and introducing oxygen-containing polar functional groups. Air-plasma treatment increases surface oxygen content and forms polar functional groups, including hydroxyl and carbonyl groups, on cotton fabric [14]. Similarly, atmospheric-pressure plasma treatment produces surface etching and increased oxygen-containing functional groups. Such modifications may improve the wettability and accessibility of cotton fibres to the aqueous almond leaf dye solution, thereby facilitating dye penetration and adsorption. Furthermore, interactions between phenolic hydroxyl groups in the natural colouring compounds and cellulose hydroxyl groups may contribute to dye retention [15]. These combined effects may result in higher K/S values for air DBD plasma-pre-treated dyed fabrics compared with untreated fabric. Figures 1 and 2 show the colour strength and colour coordinate values of dyed cotton fabrics, respectively.

The increase in colour strength may be associated with changes in the surface characteristics of cotton fibres. During plasma treatment, generated energetic species can modify the fibre surface by removing impurities and introducing oxygen-containing functional groups. Such modifications may improve surface wettability and facilitate the interaction between the fibre surface and dye liquor. Consequently, dye compounds may access the cotton fabric surface more readily [16].

The CIELAB colour coordinates values provide additional information on colour development in the dyed cotton fabrics. The measured CIELAB values showed variations in colour appearance among the dyed cotton fabric samples. The L* values ranged from 69.6 to 79.6 for all dyed samples. Negative a* values indicated greenish hues, while positive b* values across all samples confirmed the dominant yellow colouration from almond leaf extract. Furthermore, the C* values ranged from 36.4 to 38.12, with higher values observed under conditions that promoted greater extraction efficiency, indicating increased colour saturation. The hue angle values ranged from 91.92° to 92.27°, indicating that all dyed fabrics remained within the yellow hue region.

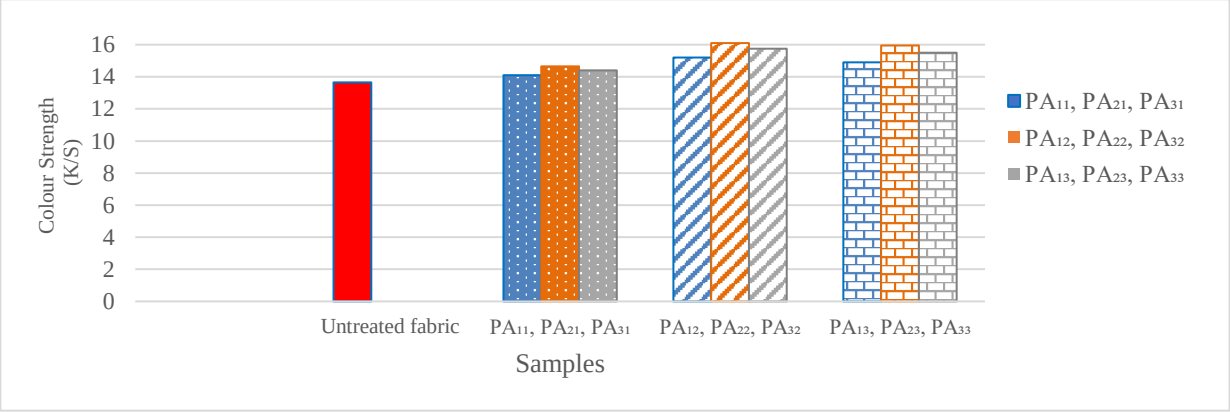


Figure.1. Colour Strength of Dyed Cotton Fabrics

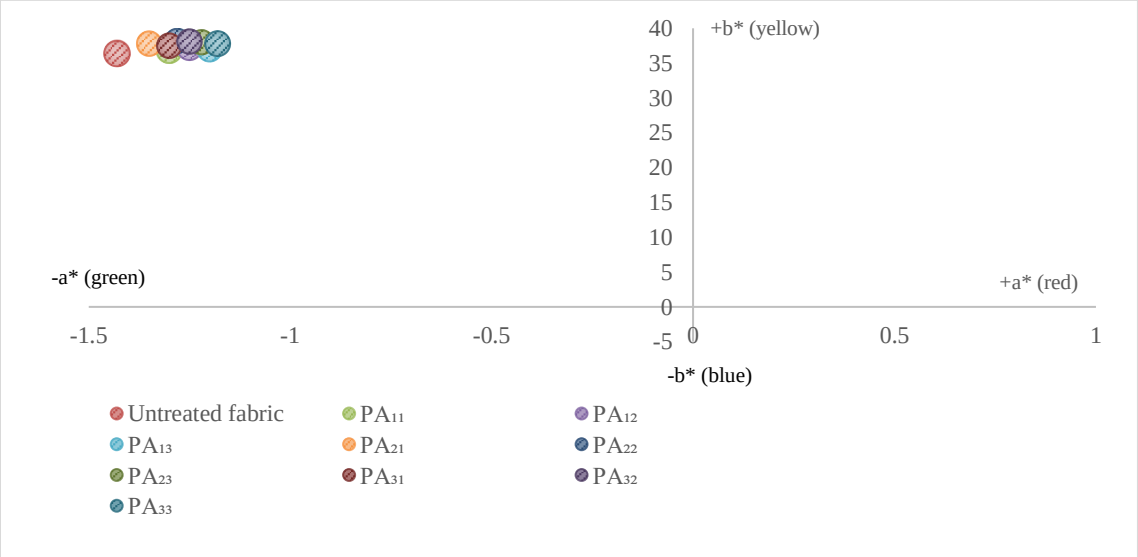


Figure.2. Colour Coordinate Value of Dyed Cotton Fabrics

3.2 Comparative Effects of Untreated and Plasma Treatment on the Colour Fastness Properties of Dyed Cotton Fabrics

3.2.1 Washing Fastness

Table II shows the fastness properties of dyed cotton fabrics. The washing fastness results showed that the untreated and the plasma-treated dyed fabrics exhibited relatively similar resistance to colour change during washing. The untreated dyed fabric and plasma-treated dyed samples PA21–PA23 achieved a rating of 3, whereas the remaining plasma-treated samples showed ratings of 2–3, indicating fair to moderate resistance. The comparable results may be related to the retention of almond leaf colourants through interactions with the cotton substrate. In the untreated fabric, aluminum ions may contribute to dye–metal complex formation, which helps retain the natural colourants on the fabric. In plasma-treated fabrics, surface oxidation and the introduction of polar functional groups may improve interaction between the cotton surface and the dye components. However, these surface modifications do not necessarily produce stronger dye fixation under washing conditions. Natural dye molecules may still be removed during laundering because some of their interactions with cellulose are relatively weak and can be disrupted by water and detergent [17,18]. The staining results showed that both untreated and plasma-treated fabrics achieved the same rating of 4, indicating good resistance to colour transfer onto the

adjacent cotton fabric during washing. The similar performance suggests that the different pre-treatment methods did not produce a noticeable difference in dye transfer under the washing conditions used in this study. This behaviour may be associated with retaining most of the colouring matter within or on the cotton fabric after dyeing and subsequent washing. Although plasma treatment can modify the surface chemistry and wettability of cotton, such modifications do not necessarily result in a substantial difference in resistance to dye migration. Similar washing fastness behaviour has also been reported for natural-dyed cotton fabrics subjected to different pre-treatment and mordanting methods [17,19].

3.2.2 Rubbing Fastness

For wet rubbing fastness, untreated fabric and plasma-treated samples (PA21–PA23 and PA31–PA33) achieved the same rating of 3, while samples (PA11–PA13) showed slightly lower ratings of 2–3. The lower ratings may be attributed to moisture, which facilitates the migration and transfer of loosely attached colouring compounds during rubbing. This may be associated with differences in dye retention and surface interaction resulting from plasma-induced modifications [17,18]. In dry rubbing fastness, untreated fabric and plasma treated samples (PA11-PA13 and PA31-PA33) showed moderate resistance, whereas samples (PA21–PA23) exhibited slightly higher ratings of 3–4. This may be associated with differences in dye retention and surface interaction resulting from plasma-induced modifications. The absence of moisture during dry rubbing also reduces dye migration compared with wet rubbing [17,18].

3.2.3 Lightness Fastness

The light fastness results showed that untreated and plasma-treated fabrics achieved the same rating of 3, indicating similar resistance to colour change under light exposure. This is because light fastness is mainly influenced by the chemical structure and photochemical stability of the colouring compounds rather than changes in the physical surface properties of cotton alone. Although plasma treatment can modify the fabric surface and improve its interaction with dye molecules, it does not necessarily protect the chromophoric structures of natural colourants from light-induced degradation [17,20]. Table II describes the light fastness test results.

Table II. Fastness Properties Of Dyed Cotton Fabrics

Sr. No.	Sample Code	Washing Fastness		Rubbing Fastness		Light Fastness
		Change in Shade	Staining on Cotton Fabric	Wet	Dry	
1.	Untreat-ed	3	4	3	3	3
2.	PA_{11}	2-3	4	2-3	3	3
3.	PA_{12}	2-3	4	2-3	3	3
4.	PA_{13}	2-3	4	2-3	3	3
5.	PA_{21}	3	4	3	3-4	3
6.	PA_{22}	3	4	3	3-4	3

7.	PA_{23}	3	4	3	3-4	3
8.	PA_{31}	2-3	4	3	3	3
9.	PA_{32}	2–3	4	3	3	3
10.	PA_{33}	2–3	4	3	3	3

4. Conclusion

This study investigated dielectric barrier discharge (DBD) plasma treatment as a surface-modification technique to improve the dyeing performance of cotton fabric using a microwave-assisted almond leaf (*Terminalia catappa* L.) extract without mordant. The results demonstrated that plasma treatment influenced the colour characteristics and colour fastness properties of dyed fabrics under different voltages and frequencies. Compared with untreated and plasma-treated dyed fabrics, plasma-treated samples exhibited the highest colour strength (K/S = 16.10), suggesting that plasma surface modification may enhance the dyeing performance of cotton fabric. Regarding colour fastness, the plasma-treated fabrics showed fair to moderate resistance to colour change during washing and good resistance to staining on adjacent cotton fabric (grade 4). Rubbing fastness showed moderate to good results, while light fastness remained at grade 3, indicating a need to improve colour durability. These findings suggest that DBD plasma treatment may improve the dyeing performance. However, further investigation into the plasma operating system (different electrode types, gases, and dielectric materials) is necessary to establish the effectiveness of this approach and its potential for broader application in natural dyeing processes.

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