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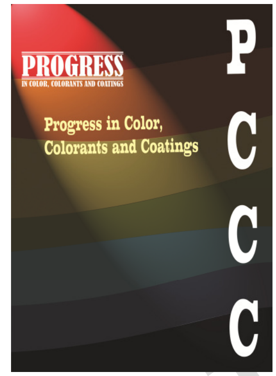

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Influence of Alkaline Pretreatment on the Reactive Dyeing Performance of Pineapple Leaf Fiber

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Abstract

This study evaluates the influence of alkaline pretreatment on the reactive dyeing performance and color fastness behavior of pineapple leaf fiber (PALF). Raw PALF extracted from Ananas comosus leaves was chemically treated with NaOH at concentrations of 3%, 5%, and 7% (w/v). This pretreatment removes non-cellulosic constituents and enhances cellulose accessibility. Microscopic analysis confirmed a transformation from coarse, fragmented structures to more individualized fibrillar networks following alkalization. Reactive dyeing using Fucozol Red UCX, Fuco Blue FSR, and Fucozol Navy/Blue NBF was performed under controlled salt, alkali, and temperature conditions. Fastness properties, including rubbing, perspiration, water, washing, and artificial saliva, were assessed according to ISO 105-X12, BS EN ISO 105-

E04, BS EN ISO 105-E01, BS EN ISO 105-C06, and GB/T 18886. Dyed PALF exhibited predominantly high fastness ratings (4-5), with only minor reductions in wet rubbing and staining on polyamide and cotton. pH analysis showed a shift from 7.4 to 7.0 after dyeing. It indicates effective neutralization and maintenance of skin-compatible conditions. Overall, alkaline pretreatment significantly improved PALF's morphological uniformity, dye uptake, and fastness stability, demonstrating its suitability as a sustainable textile fiber for coloration applications.

Keywords:

Pineapple Fiber, Alkaline Pretreatment, Color Fastness, Fiber Morphology, Reactive Dyeing.

1. Introduction

Pineapple fiber, also known as Pineapple Leaf Fiber (PALF), is gaining attention for its remarkable properties and potential applications in the textile industry. PALF exhibits high tensile strength and modulus comparable to synthetic fibers. This makesit suitable for various textile applications [1]. Alkaline treatment of PALF enhances its characteristics by increasing cellulose content and improving fiber quality [2]. PALF dyed with natural dye, ebony fruit exhibited good to excellent fastness to light, washing, and perspiration [3]. Besides natural dyeing, synthetic dyeing techniques are used for several dyestuffs. E.g., Direct Green 27 and Acid Orange 52 [4], synthetic reactive dye Levafixtm [5], and MX Basic Red 310 N 11b Procion MX reactive dye [6]. This research studied the dyeing behavior of Bangladeshi PALF species after alkalinization pretreatment. The reactive dyes usedwere Fucozol Red UCX, Fuco Blue FSR, and Fucozol Navy/Blue NBF. PALF was also evaluated for color fastness properties. The research process involved several steps, including fiber extraction, alkaline pretreatment, dyeing, and color fastness testing.

2. Experimental

2.1. Materials

PALF was extracted from the leaf of the *Ananas comosus* plant belonging to the *Bromeliaceae* family [7]. PALF was sourced from Madhupur Garh, Tangail, Bangladesh, a central pineapple-producing area [8].

2.2. Test

The testing laboratory has testedthe fastness properties of Pineapple leaf fibers to rubbing, perspiration, water, washing, artificial saliva, and pH.

2.2.1. Fiber Composition

Fiber composition was tested according to ISO 1833, and 100% of the sample was undyed pineapple leaf fiber [9].

2.2.2. Microscopic Image

Microscopic observation of the raw pineapple leaf fiber was performed to document its initial morphological characteristics before pretreatment and dyeing. The fiber samples were examined using a compound light microscope under transmitted light. A small portion of the raw fiber was placed on a clean glass slide, dispersed manually, and covered with a cover slip without additional staining.

2.2.3. Colorfastness & pH

The colorfastness of both raw and reactive-dyed pineapple leaf fiber (PALF) was evaluated against a range of physical and chemical stressors following internationally recognized test methods. All assessments were conducted under controlled atmospheric conditions (20 ± 2°C, 65 ± 4% relative humidity). The degree of color change in the specimen and staining on adjacent multifiber fabric (DW type, containing acetate, cotton, polyamide, polyester, acrylic, and wool) were rated using the greyscale for colour change (ISO 105-A02) and the greyscale for staining (ISO 105-A03) under standard illuminant D65[10, 11].

2.2.3.1 Color fastness to rubbing

Fastness to dry and wet rubbing was determined in accordance with ISO 105-X12:2016 [12]. A specimen was mounted on the base of a Crockmeter (SDL Atlas). A white cotton rubbing cloth was secured to the machine's finger. For the dry test, the finger completed 10 strokes over a 100 mm track under a force of 9 N. For the wet test, the rubbing cloth was wetted to achieve 95–100% moisture pick-up before performing the strokes. The transferred color on the rubbing cloth was evaluated against the staining greyscale.

2.2.3.2 Color fastness to perspiration

Resistance to acidic and alkaline perspiration was tested as per BS EN ISO 105-E04:2013 [13]. Specimens, composite with adjacent multifiber fabric, were immersed in artificial perspiration solutions (pH 5.5 and pH 8.0, prepared as per the standard) at a 50:1 liquor ratio for 30 min. They were then placed between acrylic plates under a load of 12.5 kPa in a perspirometer (James Heal) and maintained at 37 ± 2°C for 4 h. After drying, color change and staining were assessed.

2.2.3.3 Color fastness to water

The method described **in** BS EN ISO 105-E01:2013 was employed [14]. The composite specimen was wetted in distilled water (liquor ratio 50:1), placed between plates under 12.5 kPa pressure, and kept at 37 ± 2°C for 4 h in a perspirometer. After drying, ratings for color change and staining were assigned.

2.2.3.4 Color fastness to washing

Fastness to domestic washing was evaluated using BS EN ISO 105-C06:2010 [15], specifically test condition C2S (50 ± 2°C, 45 min). Specimens were agitated in a launderometer (Roaches) containing a standard reference detergent (ECE) and 25 stainless steel balls. After rinsing and drying, the specimen was assessed for color change, and the adjacent fabric was assessed for staining.

2.2.3.5 Color fastness to artificial saliva

This test was performed in accordance with the Chinese standard GB/T 18886-2019**.** It is critical for assessing safety in applications with oral contact. The specimen, sandwiched with adjacent cotton and wool fabrics, was immersed in artificial saliva solution (liquor ratio 50:1). It was then subjected to a pressure of 12.5 kPa at 37 ± 2°C for 4 h. After drying, the color change and degree of staining were rated [16].

2.2.3.6 Determination of pH of aqueous extract

The fiber surface has been measured according **to** ISO 3071:2020 [17]. A sample of 2.0 ± 0.1 g of cut fiber was extracted with 100 mL of 0.1 mol/L potassium chloride (KCl) solution by mechanical shaking for 2 h at laboratory temperature. The pH of the resultant extract was measured at room temperature using a calibrated pH meter (Mettler Toledo) with a combined glass electrode.

2.3. Pretreatment of Pineapple Fiber for Dyeing

The extracted PALF was prepared usingthe traditional method.The fiber was immersed in NaOH solutions at varying concentrations (3%, 5%, 7% w/v). Then itwasshaken for 10 minutes and then soaked at room temperature for 24 hours[18]. Then, the excess solvent was washed with water until the rinse water reacheda neutral pH (6.5). Lastly,the fibers were dried in an oven drier at 90°C for 4 hours (Table 1) [19].

Table 1. Pineapple Fiber Pretreatment.

Sample	Weight (g)	NaOH Ratio (%)	Time (h)	M:L
F1	5	3	24	1:80
F2	5	5	24	1:80
F3	5	7	24	1:80

This cottonization of PALF involves alkaline degumming and mechanical treatment to remove non-cellulosic matter (lignin, pectin, waxes). The treatment individualizes the fibers and reduces their linear density, making them behave like cotton-type staple fibers [5].

2.4. Reactive Dyeing Procedure

The reactive dyeing graph is shown in Figure 1. The auto dispenser was used to prepare the dye bath. The procedure started by dissolving Fucozol Red UCX 0.1595%, Fuco Blue FSR 1.99%, and Fucozol Navy/Blue NBF 0.2395% dye in warm 3.21 mL distilled water,with an M: L ratio of 1:10. Then, the solution was added to the dye bath,and wetting agents were subsequently added to ensure uniform dyeing.

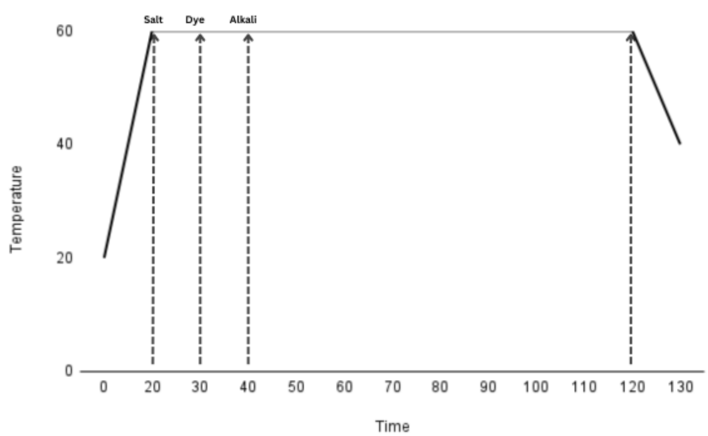

Figure 1. Reactive dyeing graph

Exhaustion dyeing was conducted in Fariha Knit Tex Ltd. The UTSTESTER D021B Oscillating Sample dyeing machine was used to add pineapple fiber to the dye bath at 30°C. Gradually increase the temperature to 60°C for 20 minutes.Slowly add Glauber salt $Na_2SO_4.10H_2O$ 60 g to enhance dye uptake anddye for 30-45 minutes [20]. A wetting agent was added to ensure uniform dyeing. Then salt was added, and the mixture was run for 10 minutes. Salt was added to enhance dye uptake and achieve uniform dyeing. The dye was added to the bath and run for 10 minutes. Fixationwas performed byadding 20 g of Na_2CO_3 to the bath. Maintain the pH 10-11 and temperature at 60°C for an additional 70-80 minutes. After dyeing, rinse with cold water, then soap at 60-70°C. Then, cold-water rinsing was repeated to remove the unfixed dyes.

3. Result and Discussion

3.1. Microscopic image before dyeing

The microscopic image reveals the raw state of pineapple fibers. The raw fiber exhibits irregular structures characteristic of its natural, unprocessed form. At this stage, the fibers are still embedded with cellulose, residual lignin, hemicellulose, wax, and some other residues [21]. The microscopic structure of raw PALF is presented in Figure 2.

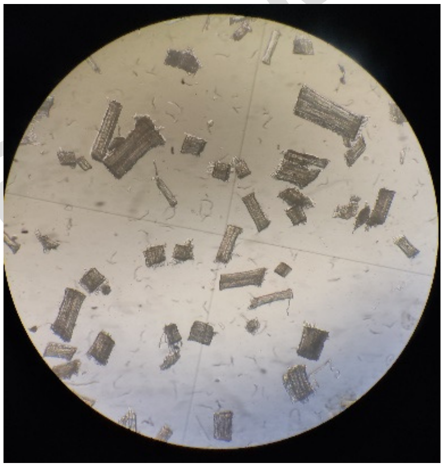

Figure 2. Microscopical view of raw pineapple fiber under a microscope.

The fibers appear as small, irregularly shaped fragments. This is typical in raw forms since they have not yet undergone extensive processing. These pieces reflect the natural breakdown of the fiber bundles during extraction [22]. Some of the pieces exhibit visible linear grooves, indicating the material's fibrillar structure [23]. The grooves reflect cellulose layers, which give these fibers their stiffness [24]. The fiber fragments may appear rough and uneven, a characteristic of unprocessed fibers, indicating the presence of lignin and other natural substances [25]. Smaller particles are remnants of plant tissues or non-fibrous materials left during extraction [18].Before dyeing, the raw fiber exhibits fragmented, irregular structures with rough surfaces.This indicated the presence of lignin, hemicellulose, and other plant residues. Fibers appear unprocessed and coarse, with visible fragments suggesting further cleaning or retting would enhance uniformity [26].

3.1.2 Microscopic Image after Dyeing

The microscopic view of the dyed pineapple leaf fiber (PALF) demonstrates the structural and visual changes induced by reactive dyeing, as shown in Figure 3. After dyeing, the fibers exhibit a blue tint. This confirmed successful reactive-dye absorption via chemical bonding to cellulose. Some areas exhibit frayed, separated filaments, indicating the impact of alkaline dyeing on the fiber structure. The uneven color distribution suggests variability in porosity or incomplete pretreatment, with smooth and coarse regions visible [27]. The comparison highlights the transformation from a raw, fragmented structure to a more open one. The dyed fiber was enhanced with color. Slight surface irregularities and minor structural changes occurred due to dyeing.

Figure 3. Microscopical view of dyed pineapple fiber under a microscope

The blue tint suggests successful uptake of the reactive dye superficially, which forms a covalent bond with the cellulose component of the pineapple fiber [28]. Uneven patches of coloration indicate variability in absorption due to differences in fiber porosity or surface accessibility.The fibers appear more frayed and less tightly aligned due to swelling induced by dyeing in an alkaline medium. Individual filaments are more visible, indicating the alkalization process. The alkalization process has broken up some fiber bundles, separating the fibrils [29]. The linear fibers entangled at the top left suggest that some fibers have maintained their tensile strength and flexibility after dyeing. Reactive dyeing enhances the fiber's structural integrity by chemically bonding the dye to cellulose [30].

3.2. Color fastness and pH results

The post-dyeing evaluation of pineapple leaf fiber (PALF) confirms that the reactive dyeing process preserved excellent color fastness and neutral chemical stability across all standardized tests (Table 2). The summarized comparison shows that PALF retained high fastness properties across all ISO and GB/T standards after reactive dyeing. Minor reductions were observed in wet rubbing and polyamide staining, but all results remained within acceptable industry ranges (≥ Grade 3). The neutral post-dyeing pH (7.0) further validates PALF's skin compatibility and chemical stability, supporting its application in sustainable and wearable textiles.

Table 2: Color fastness and pH results of raw and reactive-dyed PALF

Test Name	**Testing Standard**	**Raw PALF Rating (Before Dyeing)**	**Dyed PALF Rating (After Dyeing)**	**Performance Observation**
Color Fastness to Rubbing	ISO 105 X12	Dry: 4–5 Wet: 4–5	Dry: 4–5 Wet: 3	Excellent dry rubbing resistance in both states; minor reduction in wet fastness after dyeing due to fiber swelling.
Color Fastness to Perspiration	BS EN ISO 105-E04	Acidic: 4–5 Alkaline: 4–5 (All fibers)	Acidic: 4–5 Alkaline: 4–5 Staining: Polyamide 3–4, Cotton 4	Stable under both perspiration types; slight staining on polyamide and cotton post-dyeing.
Color Fastness to Water	BS EN ISO 105-E01	Color Change: 4–5 Staining: 4–5 (All fibers)	Color Change: 4–5 Staining: Cotton 4, Polyamide 3–4	Excellent water resistance; minor staining on polyamide and cotton after dyeing.
Color Fastness to Washing	BS EN ISO 105-C06	Color Change: 4–5 Staining: 4–5 (All fibers)	Color Change: 4–5 Staining: 4–5 (All fibers)	Outstanding wash fastness maintained; no notable change post-dyeing.
Color Fastness to Artificial Saliva	GB/T 18886	Color Change: 4–5 Staining: 4–5	Color Change: 4–5 Staining: 4–5	Excellent biological stability; unchanged fastness to artificial saliva.
Overall Color Fastness Rating Guide	ISO Scale (1–5)	Grade 4–5: Slight to negligible change	Grade 3–5: Moderate to negligible change	Overall high fastness with minimal variations; Grade 3 was observed only in wet rubbing and polyamide staining.
pH Value (Extracted with KCl)	ISO 3071:2020	7.4 (Neutral–Slightly Alkaline)	7.0 (Neutral)	Maintains neutral pH after dyeing; confirms skin-friendly and stable fabric surface.

3.2.2. Color Fastness to Rubbing

Both raw and dyed fibers maintain high resistance to color loss, with ratings of 4-5 in dry-rub fastness. The dyeing process did not compromise the fiber's stability under dry conditions.There is a noticeable drop in fastness from 4-5 for the raw fiber to 3 after reactive dyeing, as measured by wet-rubbing fastness. A rating of 3 indicates moderate staining.This is a common issue with dyed fibers, especially when wet [31]. Reactive dyes tend to penetrate the fiber well.But due to the dyes' nature and the fixation process, wet abrasion can still cause some dye transfer.This decline occurs because fiber swelling during wet conditionsakes the dye more prone to transfer[32].

3.2.3. Color fastness to perspiration

No color change.The 4-5 (minimal to no change) remained consistent. For color staining, acetate andacrylic showed 4-5 (slight to no staining, comparable to raw fiber).Cotton andwool declined slightly to 4 in some conditions, meaning minor noticeable staining.Polyamide/Nylon dropped to 3 under acidic perspiration, suggesting noticeable staining. The polyester wasmostly stable, with values around 4-5.

After dyeing with reactive dyes, the overall colorfastness to perspiration is primarily maintained. However, a slight decline was observed in specific fibers, such as polyamide (value of 3) and cotton (value of 4), particularly under acidic conditions, signaling increased staining. The reduced staining resistance of these fibers may be attributed to chemical interaction between reactive dyes and perspiration components[33].

3.2.4. Color fastness to water

Both before and after dyeing, the color change score remained consistent at 4-5. This suggests excellent resistance to fading when exposed to water across both conditions.Acetate, Polyester, Acrylic, and Woolshowedno significant difference. They maintained a high score of 4-5 for both pre-dyeing and post-dyeing tests.However, cotton showed a slight reduction in performance after dyeing.The score changed from 4-5 to 4, indicating a minor increase in potential staining.Polyamide/Nylon alsoshowed a more pronounced drop after dyeing, from 4-5 to 3-4, suggesting reduced resistance to staining on this substrate.The drop in staining resistance on polyamide and cotton indicates that dye uptake and fixation processes could alter the fabric's interaction with water and other fibers [34]. Thisrelates to the dye's affinity for specific fiber types or to changes in fiber surface properties after dyeing. Polyamides are known to be more prone to staining due to their chemical structure. Polyamides have limited terminal amine groups, which makes them susceptible to staining and reduces dye uptake. The presence of both hydrophobic and hydrophilic sections in polyamide fibers influences dyeing properties, leading to variations in post-dyeing results and fastness. [35].

3.2.5. Color fastness to washing

The color Change rating indicated minimal color change in both conditions. A rating of 4-5 suggested high color stability under washing conditions, with almost no noticeable fading.In Staining ratings, consistent ratings across substrates such as acetate, polyester, cotton, and others indicate that both pre-dyed and post-dyed fabrics exhibit excellent resistance to staining other fibers during washing. The dye's fixation quality appeared stable across the process.

3.2.6. Color fastness to saliva

Before dyeing, when exposed to artificial saliva, the raw pineapple fiber shows excellent resistance to staining and color change. A rating of 4-5 indicates negligible to no visible change or staining. This demonstrated the fiber's inherent stability when interacting with mildly acidic or enzymatic fluids such as saliva.

After dyeing (Reactive Dye Application), the pineapple fiber maintains the same high level of color fastness, with ratings of 4-5 across both color change and staining tests. The reactive dye was well-fixed to the fiber and did not degrade the fiber's resistance to artificial saliva. Reactive dyes form strong covalent bonds with cellulose-based fibers, contributing to their excellent durability under various conditions, including exposure to saliva[36].

Both before and after dyeing, the pineapple fiber shows no significant difference in colorfastness ratings. The stability across both conditions is a testament to the high quality of the fiber and the effectiveness of the reactive dyeing process. Reactive dyes are particularly suitable for such fibers due to their permanent bonding, which ensures the color remains intact even in mild fluids such asrtificial saliva[37].

3.2.7. pH of aqueous extract

Before dyeing, the pH was 7.4, indicating that the raw pineapple fiber was slightly above neutral. However, it was safe and in the expected range for untreated fibers. This suggests that the fiber is in good condition for further processing. After dyeing, the pH was 7.0, indicating a perfectly neutral state. This slight decrease was likely due to the dyeing process, in which pH regulators were used to optimize dye fixation [38]. Reactive dyes require alkaline conditions during fixation, but are often neutralized during rinsing to stabilize the fiber [39]. The slight pH change (from 7.4 to 7.0) after dyeing reflects the controlled chemical changes during the process. Reactive dyeing involves an alkaline environment (pH ~9–11) during dye application [40]. This is followed by neutralization to prevent fiber damage and ensure durability. The resulting neutral pH of 7.0 ensures the fabric will be skin-friendly and less prone to degradation[41].Maintaining a neutral pH after dyeing is important for comfort, prolonged material handling, and fiber pretreatment stability. A neutral pH ensures that the fiber will not irritate the skin. That aligns with textile industry standards for sustainable, wearable fabrics [42].

4. Conclusion

The current study has successfully demonstrated that PALF fiber achieves notable color fastness, structural integrity, and durability when treated with reactive dyes. The microscopy analysis revealed that dyeing in an alkaline medium altered the fiber's morphology. It enhanced color uptake but introduced minor structural changes, such as filament fraying, possibly due to alkaline swelling. The results highlight how reactive dyeing chemically bonds to the cellulose component of PALF. It promotes color stability even under challenging conditions. Colorfastness tests indicate that PALF dyed with reactive dyes maintains a high resistance to rubbing, washing, perspiration, and artificial saliva, with minimal color change. Additionally, it shows moderate staining across fiber types such as acetate, cotton, polyester, and acrylic. However, some reduction in staining resistance was observed on polyamide and cotton.That aligned with findings on reactive dyes' interaction with these fibers. This slight decline, particularly under wet conditions, suggests that fiber swelling may expose bound dye particles. Itmade them susceptible to transfer in specific environments. The pH evaluation before and after dyeing (from 7.4 to a neutral 7.0) supports the notion that the reactive dyeing process can be controlled to avoid adverse effects on fiber comfort and stability, ensuring skin-friendly properties suitable for prolonged use. The neutral final pH also demonstrates that reactive dyeing can produce textile materials thatmeet industry standards for safe, wearable products. Overall, this study underscores PALF's suitability for sustainable textile applications. The successful application of reactive dyes on PALF further emphasizes its potential to meet both performance and sustainability criteria in the modern textile industry,making it a promising alternative to synthetic fibers.

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