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Optimization of Dyeing Conditions of Natural Dye Obtained from *Vaccinium corrymbosum* (Blueberry) On Cotton Fabric

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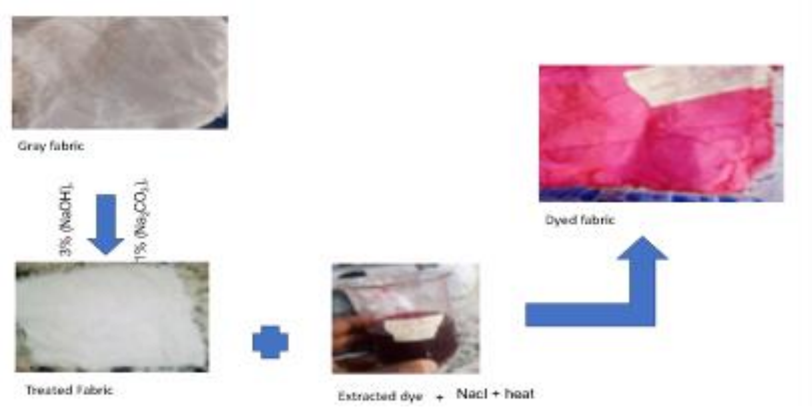
Using plants as natural sources of dyes promotes sustainable practices in textile production by reducing reliance on synthetic dyes, which often pose environmental and health concerns. *Vaccinium corymbosum* (blueberry) is a commercially and culturally significant plant, providing nutritious fruit and supporting biodiversity in its native habitats. In this study, natural dye extraction from *V. corymbosum* was performed using a mixture of 15 ml methanol, 20 ml distilled water, and 1% HCl. The extracted dye was optimized for dyeing cotton fabric by studying the effects of dye concentration, temperature, and dyeing time. Dye concentrations were varied at 0.1, 0.15, 0.25, and 0.3 g/cm³, with the optimum concentration identified as 0.3 g/cm³. Dyeing temperatures were adjusted to 60, 70, 80, 90, and 100 °C, with optimal results at 90 °C. Dyeing times were varied at 30, 40, 50, 60, and 70 minutes, with 70 minutes being the optimal duration. Color fastness of the dyed fabrics was assessed using a gray scale. Results showed that wash fastness was good (rating of 3), perspiration fastness in acidic and basic media ranged from fairly good (rating of 2) to better (rating of 3.5), and light fastness was moderate (rating of 5). The study found that increasing dye concentration led to greater dye absorption by the fabric. As temperature increased from 60 to 90 °C, dye molecule movement accelerated, enhancing diffusion into the fiber structure and resulting in deeper, more intense coloration. However, at 100 °C, excessive heat caused bond breakage, leading to faded colors. Extended dyeing time also improved the dye fastness on the fabric. This research demonstrates the potential of *V. corymbosum* as a natural dye source, contributing to more sustainable textile dyeing practices.

Graphical abstract



Keywords

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1. Introduction

Vaccinium corrymbosum has both commercial and ecological importance, supporting not only the agricultural industry but also various wildlife species that rely on its fruit for food. The dyeing process is a complex and multi-step procedure that requires careful consideration of the type of dye, substrate, and desired outcome. Advances in dye chemistry and technology continue to enhance the efficiency, sustainability, and creativity of dyeing applications. Color has always played a dominant role in human life since time immemorial. In every civilization from Stone Age to the „Silicon slip age‟, it has played an important role in adding beauty to the world [1]. Dyes are the colorant matters which penetrate into the fiber and appear to become a part of it. Dyes may be either natural or synthetic and both these dyes are used for dyeing fibers, yarns and fabrics [2].

Because synthetic compounds are no biodegradable and carcinogenic, they are utilized to dye textile fabrics, which lead to concerns with waste management and water pollution. Consider alternatives to synthetic dyes that can make the environment and human health safe in order to eliminate these health and environmental risks. From this vantage point, natural dyes might be a useful option for the textile industry. Numerous natural dyes found in various plants and vegetables across the universe can be employed in addition to synthetic dyes [3, 4].

Natural dyes/colorants derived from flora and fauna are believed to be safe because of its nontoxic, oncarcinogenic and biodegradable in nature [5]. They are not producing any undesired by-products and at the same timethey help in regenerating the environment, therefore natural dyes are the safe dyes [6, 7].

Natural dyes can be obtained from natural resources like plants, minerals, insects and fungi but most of the dyes are taken from plant parts i.e. leaves, barks, flowers, fruits and roots. Natural dyes have some special properties like soothing color, biodegradable, non-hazardous, non-carcinogenic and antimicrobial resistance etc. [8]. Natural dyes offer significant ecological benefits due to their renewable sources, non-toxicity, and biodegradability. However, challenges such as color fastness, production scalability, and economic feasibility need to be addressed to enhance their adoption. As technology advances and consumer preferences shift towards sustainability, the use of natural dyes is likely to increase, contributing to a more environmentally friendly textile industry. Natural dyes have better biodegradability and are obtained from renewable sources. Efforts are now being made to identify the raw materials from plant sources and to standardise the recipes for their use. Thus, utilizing natural dyes to impart colour on the fabric has a number of advantages over synthetic dyes. These include (i) eco-friendliness, (ii) possibility of premium pricing (iii) variety of colours produced from single dye source (iv) no health hazard to humans [9]. Natural dyes offer significant ecological benefits due to their renewable sources, non-toxicity, and biodegradability. However, challenges such as color fastness, production scalability, and economic feasibility need to be addressed to enhance their adoption. The usage of natural dyes appears to increase as consumer attitudes shift toward sustainability and technological advancements make textile production more ecologically friendly.

Application of dyeing to the substrate helps in the formation of color, which attracts the appearance of the substrate. This process is achieved under the influence of conditions such as temperature, concentration, time, pH, and material-toliquir ratio [10] applied selectively extracted few natural dyes to a silk fabric by an exhaustion dyeing process where aluminum potassium sulfate, ferrous sulfate, copper sulfate, and stannous chloride were used as mordants. Natural colors are thought to be more environmentally friendly than synthetic dyes since they are biodegradable, less poisonous, and less allergenic. Natural dyes are known for their use in coloring of food, leather, wood, as well as natural fibers like wool, silk, cotton and flax since ancient times [11]. Woolen fabrics have been reported to be dyed with acacia pennata plant extract by [12]. The plant's bark was used to extract the pigment. In another experiment, [13] used the Pressurized Hot Water Extraction Method (PHWE) to study the effect of temperature on the color of natural dyes from the Poseur plant. The effect of PHWE was examined at temperatures of 50, 75, 100, and 125 °C. Using a mass-to-liquid ratio of 1:20, 2 g of grounded powder were boiled on a hot plate for 30 minutes at atmospheric pressure. There is ongoing research concerning the analysis of the effect of temperature on anthocyanins extracted from onion plants, viz., optimizing temperature conditions as reviewed [14].

High-level polluting businesses are subject to strict regulations due to global concern over environmental contamination. Among these industries is the textiles industry, which produces a significant amount of toxic effluent. The development of fundamentally new technologies and/or modifications of existing ones for cleaner and environmentally friendly production processes has been the subject of numerous studies in recent years [15]. As a result, the use of natural dyes that are nontoxic, antibacterial, and environmentally friendly on textiles—ideally made of natural fiber products—has gained significance as people become more conscious of the environmental risks associated with synthetic dyes. However, due to growing interest in their potential medical benefits, natural dyes are becoming more and more popular [16]. Cotton, rayon, hemp, and other cellulose-based fibers and dye molecules form a permanent bond.

A good dye is characterized by being soluble in water or dispersible in solvent and transferable to the fabric or other materials to be dyed by the process of absorption and exhaustion. Dye must have affinity for the fabric, be colorfast, and be organoleptically acceptable to consumers. Colourfastness is the ability of a dye to resist fading or staining caused by sunlight, washing, perspiration (dilute acids and alkalis), crocking or rubbing and other organic solvents used in laundering and dry-cleaning [17]. A single dye may not be fast in all circumstances. However, a dye should remain viable and gracefully age with the product [18]. The fastness or stability of a dye in fabric is determined by the type of fabric fiber (natural or synthetic), method of dye application (for instance, conventional exhaust procedure), and class of dye (acidic, basic, direct, vat, fiber reactive, disperse, azoic, or mordant dye).

2. Material and Methods

Distilled water, Dye, NaCl, Na_2CO_3, grey fabric and other glass wares. Dye prepared and characterized by [19] at Modibo Adama University Yola.

2.1 Sample Collection

Grey fabric material (sample) was purchased from Jimeta Main Market, opposite JAIZ Bank, Yola Adamawa St. Grey fabric material (sample) was purchased from Jimeta main market, opposite JAIZ Bank, Yola Adamawa State. The physical properties of the sample include being gray in color, having a size of 8 × 8 cm^2, absorbent, nontoxic, hypoallergenic, high wet modulus (stronger when wet), being biodegradable, and being wipe-dry performance.

2.2 Fabric pretreatment

The fabric was pretreated in two stages; scouring and bleaching [20].

2.3 Scouring process of grey cotton fabrics

Scouring process is the removal of dirt particles, oil, and other unwanted substances to make it hydrophilic. The process occurs as follows: 0.8 g of the cotton fabric was steeped in the solution containing 3% sodium hydroxide (NaOH), 1% sodium carbonate (Na_2CO_3), saponification reaction and emulsification reaction took place during this time which converted all the insoluble material to soluble form. Then it was subjected for hot and cold wash with water.

2.4 Bleaching process of grey fabric

Bleaching process is the art of removing coloring matter from the fabric. The process occur as follows: given cotton material was weight accurately. The material was steeped in a solution containing hydrogen peroxide, sodium silicate and sodium hydroxide at 40 ^{0}C. The temperature increased to 100 ^{0}C and it was bleached at 120 minutes. Then subjected to hot and cold wash with water and dried.

2.5 Dyeing process

Extraction of dye from *vaccinium corrymbosum* was done using method proposed by [19]. The dye was applied to cotton fabric at different dyeing conditions (temperature, time, and concentration) using the direct dyeing method. The study showed that the dye obtained displayed fairly good saturation on cotton with medium to good fastness properties. The strength of the dye extracts obtained at room temperature was minimum, slightly better at 60 °C, and maximum when extraction was carried out at 90 °C. As pertaining to time and salt concentration, the K/S value increased to its maximum when stirring was done between 80 and 100 minutes.

2.6 Determination of Optimum Dyeing Condition

Three dyeing conditions were varied (concentration. temperature, and dyeing time) in other to ascertain for the optimum dyeing conditions most suitable for application

2.7 Determination of optimum dyeing concentration

For determining the optimum dye concentration, five different concentrations of reactive dye, i.e., 0.1, 0.15, 0.2, 0.25, and 0.3 g/cm^3, were taken, and samples were dyed at 95–100 °C for 30 minutes. The absorbance of dye solutions before and after dyeing was recorded at the optimum wavelength. The dye solution giving the maximum dye absorption was taken as the optimum dye concentration [21].

2.8 Determination of optimum dyeing temperature

Temperature has a great impact in dye uptake as the heat is involved in a process. To optimize dyeing temperature, dyeing was carried out using the optimum concentration of dye at five different temperatures i.e., 60, 70, 80, 90, and 100 °C. The temperature giving maximum dye absorption was taken as the optimum dyeing temperature.

2.9 Determination of optimum dyeing time

The fabrics samples were dyed using optimum dye concentration for five different time durations i.e., 30, 40, 50, 60 and 70 minutes. The optimum dyeing time was selected on the basis of maximum dye absorption.

2.10 Determination of color fastness properties

The color fastness to washing and light of dyed cotton fabrics were determined according to American Association of Textile Chemists and Colorists (AATCC) Test Method 61 (2009) and AATCC Test Method 16 (2004), respectively. The color fastness to perspiration of dyed cotton in alkaline and acidic conditions was determined according to AATCC Test Method 8 (2001).

2.11 Fastness to washing test

The dyed sample of size 8 by 8 cm2 was cut and placed between two undyed, unfinished samples of size 4 by 4 cm2 (the composite test sample) and sewn to both sides of the fabric. The composite sample was treated in a bath containing water (a water shaking bath) for 30 minutes at 50 ^{0}C. After 30 minutes, the sample was taken out of the washing solution. The sample was rinsed twice in cold distilled water and washed in running cold water for 10 minutes. Then the sample was squeezed carefully to remove the stitching solution, and the sample was dried. The contrast between the treated and untreated samples was compared with the color change gray scale (ISO 9001 2000 group).

2.12 Fastness to perspiration

Specimens of the textile in contact with adjacent fabrics were treated in both acidicand basic solutions containing histidine, drained and placed between two plates under a specified pressure in a test device. The specimens and the adjacent fabrics were dried separately. The change in colour of each specimen and the staining of the adjacent fabrics are assessed by comparison with the grey scales or instrument (ISO 105-E04:2013(E)

2.13 Fastness to light

Light fastness Tester GT-D02A-1 was used at room temperature,Four pieces of test specimens were cut (8 cm by 8 cm) length and width and attached with the specimen holder. The holder was set into the light fastness tester. Experiment continued at 72 hours. After 72 hours expiration the specimen was taken from the light fastness tester, then the specimen was tested and compared with the blue scale ranging from 8 (no fading) to 1 (very poor).

Procedure (alkaline solution)

Alkaline solution,freshly prepared, using grade 3 water complying with ISO 3696, containing, the following solutions per litre:

— 0,5 g of l-histidine monohydrochloride monohydrate ($C_6H_9O_2N_3·HCl·H_2O$);

— 5 g of sodium chloride (NaCl); and

— 5 g of disodium hydrogen orthophosphate dodecahydrate ($Na_2HPO_4 \cdot 12H_2O$)

The solution was brought to pH 8 (±0.2) with 0.1 mol/l sodium hydroxide solution.

Acid solution

freshly prepared, using grade 3 water complying with ISO 3696, containing the following solutions per litre

— 0,5 g of l-histidine monohydrochloride monohydrate ($C_6H_9O_2N_3 \cdot HCl \cdot H_2O$);

— 5 g of sodium chloride (NaCl);

— 2.2 g of sodium dihydrogen orthophosphate dihydrate ($NaH_2PO_4 \cdot 2H_2O$).

The solution was brought to pH 5.5 (±0,2) with 0,1 mol/l sodium hydroxide solution.

Adjacent fabrics (ISO 105-A01).A multifibre adjacent fabric complying with ISO 105-F10.

3. Results and Discussion

Table 1. Effect of Dying Concentration.

Concentration(g/cm³)	Absorbance
0.10	0.20
0.15	0.30
0.20	0.40
0.25	0.40
0.30	0.60

Table 2. Effect of Dyeing Temperature.

Temperature °c	Absorbance
60	0.20
70	0.30
80	0.40
90	0.5
100	0.4

Table 3. Effect of Dyeing Time.

Time(min.)	Absorbance
30	0.22
40	0.30
50	0.40
60	0.54
70	0.63

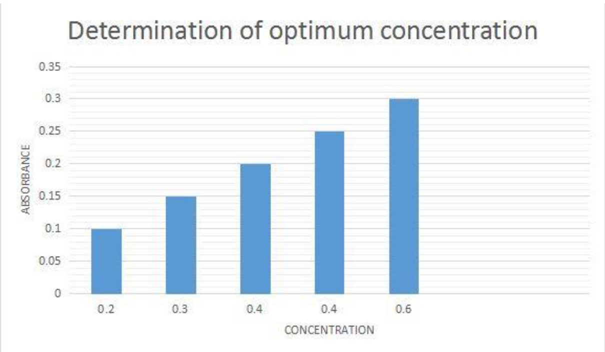

Fig. 1. Determination of optimum dyeing concentration.

As the concentration increases, the intensity of the dye also increases. From figure 1 above, the dye absorption increases with an increase in concentration throughout. The concentration of 0.3 g/cm³ (at the amount of dye of 0.6 g) was considered the optimum concentration of the dyed fabric. This shows that the color strength pattern increases with an increase in the concentration of the dye the.

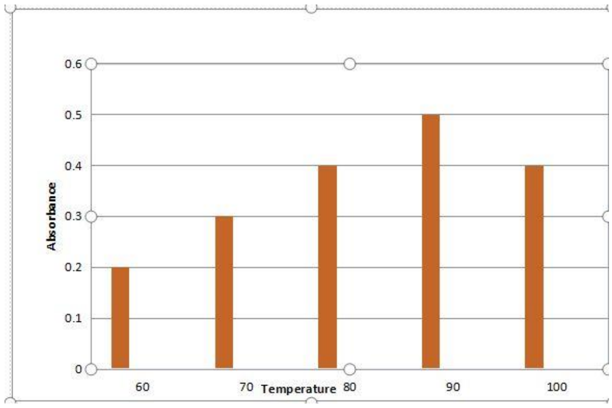

Fig. 2. Effect of temperature.

From figure 2, dyeing was carried out at five different temperatures, i.e., 60, 70, 80, 90, and 100 °C, respectively. The mean absorption value for temperatures 90 and 100 °C is significantly higher than that of temperatures 70 and 80 °C, while there was no significant difference between the absorptions of temperatures 90 and 100 °C. Therefore, 90 °C was selected as the optimum dyeing temperature. It has been reported that this increase in dye uptake can be attributed to better dye exhaustion at higher temperatures.

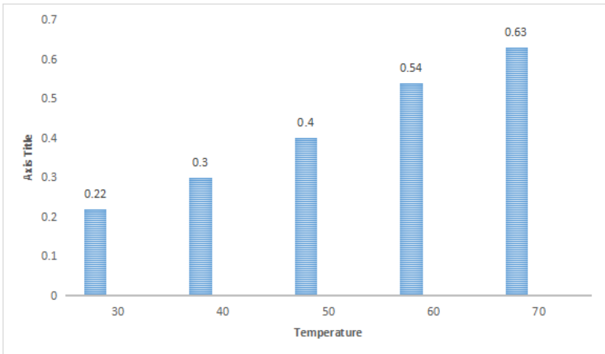

Fig. 3. Effect of time.

As the time for dyeing increased, the color strength value also increased. From figure 3 above, it is clear that dyeing for 70 minutes shows higher color strength, so dyeing for 70 minutes was considered the optimum time. Summarily, the optimum concentration, temperature, and time for the dyeing conditions are shown in tabular form below. Summarily, the optimized dyeing concentration, temperature, and time were 0.3 g/cm³, 90 °C, and 70 min, respectively.

The result of fastness properties was found to be; wash fastness property good (3), perspiration in acidic medium fairly good (2.5) and that of basic medium better (3.5) and light fastness property was very good (4).

a) b) c)

Fig. 4. a) Gray fabric (untreated). b) Treated fabric. c) Dyed fabric.

4. Conclusions

The study demonstrates that increasing the concentration of a dye increases its intensity and color strength. The optimum concentration for dyeing fabric was 0.25 g/cm^3, with a color strength pattern that increased with concentration. The dyeing process was conducted at different temperatures, with 90 °C being the optimum temperature due to better dye exhaustion. The optimal dyeing time was 70 minutes, with good wash fastness, fair perspiration in acidic medium, and excellent light fastness in basic medium.

Author Contributions

Musa Yahaya Abubakar: Conceptualization, Methodology, Investigation. Usman lawal: Supervision, Project administration, Writing - Original draft preparation. Ma'aruf Abdulmumin Muhamad: Investigation. Fatima Garba: Supervision. Ansar Bilyaminu Adam: Writing - Reviewing and Editing. Yilni Edward Bioltif: Supervision

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