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Extraction And Utilization Of Natural Dyes From Guinea Corn Leaves Using Solvent Extraction

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ABSTRACT

With increasing environmental concerns and demand for sustainable substitutions, natural dyes provide a biodegradable and non-toxic compound to synthetic dyes, which are often associated with health and environmental hazards. This research employed an experimental research design that focused on the extraction and utilization of natural dyes derived from Sorghum bicolor Leaves (SBL) solvent extraction methods. Natural dye extracted from SBL was used as sustainable and eco-friendly alternative to synthetic dye use in the coloring of cotton fabric for production of tie and dye (adire) using different mordant. The dye was extracted using absolute ethanol, acetone and distilled water as solvents. Alum ($KAl(SO_4)_2.12H_2O$), Sodium Chloride (NaCl) and Lime juice ($C_6H_8O_7$) were used separately as mordant. Cold maceration of each solvent was performed for 48hours, with agitation at intervals within the hours for complete extraction. Constant parameters (temperature, extraction time, solvent-to-solid ratio) were maintained. The extracts were filtered and concentrated using soxhlet extractor. Solvents used were recovered. Soxhlet extraction of each solvent was also performed at different temperature based on the boiling point of each solvent while pH, extraction time and solvent-to-solid ratio were kept constant. The extracted dyes were applied on cotton fabrics treated with different mordants. The dyes stability, colourfastness on the fabrics and compatibility of the mordants were assessed. The results showed that polar solvents (absolute ethanol and acetone) showed high extraction efficiency compared to distilled water and also confirmed the viability of solvent extraction for obtaining high-quality natural dyes, thus highlighting the potential for scaling up in eco-friendly industrial applications.

Keywords: Natural biodegradable dyes, Extraction, Utilization, Mordant

1.0 INTRODUCTION

Natural dyes are pigments from plants and animals that impart colour to products such as cosmetics, fur, hair, fabric, foods and drugs. Application of dyes to various materials improve their appearance, beautify and add values to such product. The plant based natural dyes can be extracted from different parts of plant, such as leaves, barks, roots, seeds, stems, fruits and flowers using solvent extraction. Among researchers who have extracted dyes from plants are Noor and Ijaz (2024) and Jabar*et al.*, (2021) who extracted dye from Peels of Citrus Fruits and Mucunaslonaei F leaves respectively. Hong (2018) extracted dye from *Coffee arabica* L. seeds; Bukhari*et al.*, (2017) extracted dye from *Juglansregia* L bark; Uddin (2015) extracted dye from leaf of *Mangiferaindica* L tree and Yusuf *et al.,* (2012) extracted from root of *Rubiacordifolia.*

The use of synthetic dyes has raised great concerns due to their environmental toxicity and non-biodegradability. The introduction and acceptance of synthetic dyes in industries years ago was a result of their cost-efficiency, ease of production, and colourfastness without putting into consideration the adverse effects they can have on man and the environment. Recent environmental concerns over the toxicity and non-biodegradability of synthetic dyes have rekindled interest in natural dyes as sustainable and eco-friendly alternatives.

Guinea corn (*Sorghum bicolor*), is a cereal crop widely cultivated in Africa and Asia. It produces substantial agricultural waste in the form of leaves and straw, which are often discarded and form an additional huge mass of environmental pollutants from crop harvest. Researchers have shown that various parts of the guinea corn plant contain phenolic compounds, tannins, and anthocyanins, which have potential applications as dyes. However, the extraction and application of dyes from Guinea corn leaves and straws remain under-explored, especially using optimized solvent extraction techniques followed by characterization.

Synthetic dyes are produced from petrochemical sources through hazardous chemical processes, which are detrimental to the environment. They release harmful chemicals that are allergic, carcinogenic and injurious to human health (Osabohien, 2014). Textiles industries produce large amounts of polluted wastes that are usually discharged to surface and ground water bodies. This chemical waste contribute several damages to the ecological system of the receiving water surfaces, creating a huge of problem to ground water resources. The world continual demand for fibres and safety dyes, people became mindful of environmental hazards posed by synthetic dyes, which includes skin cancer, disorders and allergic contact dermatitis. This consciousness had led to the renaissance of awareness in natural dye (Kultam and Gokhale, 2011). They are non-toxic, non-allergic to the skin, non-carcinogenetic, easily available and renewable (Thiyaparajan, Balakrishn and Tarnilaras, 2015). For proper technical extraction and application of natural dyes from guinea corn leaves blended with the straw, efficient method of extraction and adequate use of solvents that will be able to extract the dye from the plant has to be employed. This is the focus of this research work, which will invariably lead to production of colourfastness dye from agricultural wastes as alternatives to synthetic dyes.

The world is moving towards a sustainable research whereby the ecosystem can be protected through the use of eco-friendly and biodegradable materials. Plant-based dyes have a long history of use in various cultures for colouring textiles, crafts, and artworks. The dyes obtained from different parts of plants, have a wide range of coloursexhibit and offer additional bioactive properties like antimicrobial and antioxidant activity (Alegbe and Uthman2024). The extraction and characterization of these dyes using solvent extraction techniques is a crucial step toward their application in modern industries. There are many plant species that are well-known for their vibrant and distinctive colours, which have been harnessed for dyeing textiles, cosmetics, pharmaceutical and food industries. These colours are often the result of active chemical compounds found in the plants. The plants include Jatropha Curcas, indigo, turmeric, hibiscus leaf, onion, black walnut.

Guinea corn leaf is an important source of carbohydrate, protein and minerals such as calcium, selemum, manganese and iron in which the bioavailability depends on the level of interactions with various anti-nutrients. It is also rich in B-complex vitamins. Apart from its uses as food, it contains some chemical compounds that have protective mechanism against cardiovascular disease, menorrhagia, cancer, and tumor growth (Nwonye and Priscilla, 2017). Guinea corn leaves and straws contribute to the huge mass of environmental pollutants from agricultural practice after harvest. Researches have proven that guinea corn leaf is a potential source of nutrients and an essential antioxidant, which could supplement human and animal diets instead of constituting a waste and source of environmental pollution. Guinea corn is known to contain bioactive compounds such as anthocyanins, flavonoids, and phenolic acids, hence it is a good source for dye extraction.

Solvent extraction is the process used for the separation of organic substances from aqueous solutions. It is used for the separation of two or more components due to their solubilities in suitable solvents. Solvents such as ethanol, methanol, acetone, and water, are widely used for recovering natural dyes from plants, in particularly guinea corn leaves and straw. Different techniques can be used to extract dyes from plant; it includes maceration and soxhlet extraction. Maceration is a technique used in extracting dye from plants that involves leaving the pulverized plant to soak in a suitable solvent in a closed container. Cold maceration is done at room temperature by mixing the pulverized plant with the solvent and leaving the mixture for about 48hours while occasionally shaking or stirring. The extracts are thereafter, strained from the plant particles (Malidi and Altikriti, 2010, Neha and Vidya, 2011).

Mordants are chemical agents that allow a reaction to occur between the dye and the fabric thereby aiding in fixing the colour to the fabric. Mordants fix the dye to a substrate by combining with the dye pigment to form an insoluble compound (Wipperlinger, 2004) which set the colour permanently on the fabric and form a good and bright colour.Obenewaa (2010) commented that mordant is an essential part of the dyeing process.

1.2 Novelty of the Study

The novelty of this research lies in the integration of cold maceration with controlled solvent recovery and post-extraction soxhlet concentration to enhance pigment yield and purity from *Sorghum bicolor* leaves. Unlike conventional aqueous extraction methods commonly reported in natural dye studies, this work comparatively evaluates multiple polar solvents under standardized parameters to optimize extraction efficiency while minimizing environmental impact through solvent recycling. Additionally, the study uniquely bridges laboratory-scale phytochemical extraction with practical textile application (adire production), thereby linking green chemistry principles to indigenous textile innovation. The systematic assessment of mordant compatibility further advances the understanding of natural dye–fiber interactions, offering a replicable framework for sustainable dye production in small- and medium-scale textile enterprises.

MATERIALS AND METHODS

Materials

Dried guinea corn leaves were purchased at Oke Oja market, Ilora town, Oyo state, Nigeria. Cotton fabric was purchased at Gaza Shopping Complex, Osogbo, Osun State, Nigeria and the mordants were purchased at Oba Market in Akure, Ondo State, Nigeria. All chemical reagents used were analytical grade.

Methods

Preparation of the leaves

The leaves were thoroughly washed several times under running tap water to remove dirt and impurities then sliced into small sizes in order to aid drying process. They were dried at room temperature and thereafter pulverized using electric grinding machine and labelled as Guinea Corn Leaves Powder (GCLP).

Dye Extraction

Cold Maceration

50g of GCLP was soaked in a 1000 ml corked vessel with 500ml distilled water for 48hours with intermittent agitation to enable complete extraction. The resultant mixture was filtered using cotton wool and filter funnel. The same quantity GCLP was prepared for both ethanol and acetone to compare the extraction performance of the three solvents. Extraction parameters such as temperature and the pH of each extract were taken and recorded.

Soxhlet Extraction

50g of the sample was measured into the thimble and inserted into the soxhlet extractor. 500ml of each solvent; ethanol, acetone and distilled water was added using funnel passing through the sample into the round bottom flask fitted into the soxhlet. The heating mantle was switched on and extraction was done for 6 hours. In each case the temperature were set base on the boiling point of each solvent, ethanol at

78^{O}C, acetone at 56^{O}C and distilled water at 100^{O}C. Several cycles of solvent were run to ensure maximum extractions.

Preparing the fabric and Mordanting:

The fabrics were desized using detergent, in order to remove oil, wax or dirt that might interfere with the dye adhering to it and then rinsed properly. In a 1000ml beaker, the prepared mordants were added to the fabric and allowed to submerge in it properly in order to charge the substrate to be dyed.

Dyeing Procedure

All the extracts were obtained in aqueous form. 50ml of each of the liquid dye was measured into the fabric with the mordant. The fabric was immerse into the beaker at room temperature and disturbed approximately for 20 minutes. The dyed fabric was removed and air-dried for oxidation to take place. Each of the dyed fabric was rinsed separately using clean water and dried.

3.1 RESULTS

Extracted Colourant**(Ethanol)**

Extracted Colourant**(Acetone)**

Extracted Colourant**(Water)**

Fig 1: Extracted colouration of each solvent using soxhlet apparatus

Table 1: Cold maceration of the sample with different solvents

Solvents	Temperature (^{O}C)	pH	Time (hour)	Yield (%)	Extracted Colour
Ethanol	25	6	48	11.3	Deep Wine
Acetone	25	6	48	13.6	Wine
Distilled Water	25	6	48	4.1	Light Brown

Table 1: Soxhlet extraction of the sample with different solvents

Solvents	Temperature (^{O}C)	pH	Time (hour)	Yield (%)	Extracted Colour
Ethanol	87	6	6	12.4	Deep Wine
Acetone	56	6	6	17.2	Dark Red Wine
Distilled Water	100	6	6	9.4	RedBrown

Resultant effect of each mordant on dyed cotton fabric

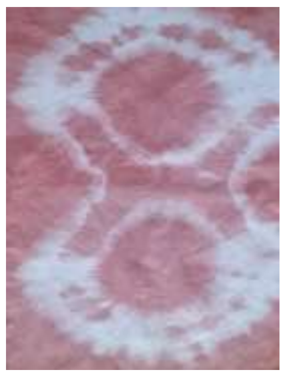
Ethanol with Alum mordant

Ethanol with Sodium Chloride mordant

Ethanol with Lime Mordant

Fig 2: Ethanol extract on each mordant

Acetone with Alum mordant



Acetone with Sodium Chloride mordant

Acetone with Lime Mordant

Fig 3: Acetone extract on each mordant

Water with Alum mordant

Water with Sodium Chloride mordant

Water with Lime Mordant

Fig 4: Water extract on each mordant

DISCUSSION OF RESULTS

4.1 Extraction Efficiency and Solvent Polarity Effects

The extraction yields obtained under controlled parameters for cold maceration (25^{O}C, pH 6, 48 hours) and soxhlet extraction (various boiling pointof each solvent, pH 6, 6hours) revealed marked differences in solvent performance. Acetone produced the highest yields (13.6% and 17.2% for cold maceration and soxhlet extraction respectively), followed by ethanol (11.3% and 12.4% for cold maceration and soxhlet extraction respectively), while distilled water showed significantly lower extraction efficiency (4.1%and 9.4% for cold maceration and soxhlet extraction respectively). The observed trend strongly correlates with solvent polarity, solubility parameters, and the chemical nature of bioactive pigments present in *Sorghum bicolor* leaves.

From an analytical chemistry perspective, solvent extraction efficiency depends on intermolecular interactions between solvent molecules and target phytochemicals. Sorghum leaves are known to contain phenolic compounds, flavonoids, tannins, and anthocyanin-like pigments. These compounds exhibit moderate polarity and are more soluble in organic polar solvents such as acetone and ethanol than in highly polarprotic solvents like water. Acetone, possessing both polar and non-polar characteristics (dipolar aprotic nature), likely enhanced the solvation of both phenolic and slightly hydrophobic pigment fractions, thereby explaining its superior yield.

The low yield observed with distilled water (4.1% and 9.4% for cold maceration and soxhlet extraction respectively) highlights a critical analytical insight: water, though environmentally benign, may not effectively disrupt plant cellular matrices to liberate pigment-bound phenolic complexes. This underscores the importance of solvent selection based on polarity index, dielectric constant, and hydrogen-bonding capacity in phytochemical extraction processes.

4.2 Influence of Controlled Parameters on Extraction Reliability

The maintenance of constant temperature (25^{O}C), pH 6, extraction time (48 hours), and solvent-to-solid ratio enhanced reproducibility and allowed solvent effects to be isolated as the primary variable. Maintaining pH at 6 likely stabilized anthocyanin-type pigments, which are known to degrade under alkaline conditions. From an analytical standpoint, pH control minimizes pigment structural transformation, ensuring consistent chromophore stability and spectral integrity.

Cold maceration over 48 hours, combined with intermittent agitation, facilitated diffusion-driven mass transfer. This method prevented thermal degradation of heat-sensitive phytochemicals, a limitation commonly encountered in high-temperature extraction techniques. Furthermore, concentration using a Soxhlet apparatus and solvent recovery introduced an analytical refinement rarely integrated in natural dye studies. Solvent recovery not only improves sustainability but also enhances concentration precision and reproducibility. These are key principles in green analytical chemistry.

4.3 Colour Variation and Chemical Implications

The extracted colour differences: Deep Wine (ethanol), Wine (acetone) and Light Brown (water), suggest qualitative differences in pigment profiles. Ethanol's deep wine coloration may indicate enhanced extraction of anthocyanins or condensed tannins with extended conjugated systems responsible for strong chromophoric absorption in the visible region.

Acetone's wine coloration, though slightly lighter, may reflect a broader extraction spectrum including both flavonoids and phenolic acids. The light brown colour from aqueous extraction suggests predominance of oxidized polyphenols or degraded pigment fractions.

From a spectro-analytical viewpoint, these differences imply variation in absorbance maxima (λmax) and molar absorptivity. Future UV-Vis spectrophotometric profiling could quantify chromophore concentration and confirm pigment identity. The results therefore bridge extraction chemistry with instrumental analysis, reinforcing the analytical chemistry relevance of this study.

5. Novelty and Research Gaps Addressed

This study fills several notable research gaps:

1. **Comparative Solvent Evaluation Under Standardized Conditions**
   Many natural dye studies rely predominantly on aqueous extraction without systematically comparing organic polar solvents under constant experimental parameters. This research provides controlled comparative data demonstrating the superiority of acetone and ethanol.
2. **Integration of Solvent Recovery and Sustainability Metrics**
   The recovery of solvents post-extraction introduces an environmentally responsible approach aligned with green chemistry principles—an aspect often neglected in small-scale dye research.
3. **Linkage Between Laboratory Extraction and Indigenous Textile Application**
   By applying the extracted dye to cotton fabric using traditional adire methods, the study bridges analytical chemistry with cultural textile innovation. This translational approach strengthens the practical viability of the research.
4. **Quantitative Yield Documentation**
   Precise yield percentages allow reproducibility and benchmarking for future optimization studies.
5. **Controlled pH Extraction Strategy**
   Stabilizing the extraction environment at pH 6 demonstrates awareness of pigment structural chemistry, particularly for phenolic compounds sensitive to pH variations.

Thus, this work advances natural dye research from descriptive experimentation toward controlled analytical validation.

6. CONCLUSION

The results clearly demonstrate that solvent polarity significantly influences the extraction efficiency of natural dyes from *Sorghum bicolor* leaves. In both cold maceration and soxhlet extraction methods, acetone yielded the highest extraction efficiency (13.6% and 17.2%), followed by ethanol (11.3% and 12.4%), while distilled water showed minimal extraction capacity (4.1% and 9.4%). The superior performance of organic polar solvents highlights their ability to solubilize phenolic and anthocyanin-based pigments effectively.

The study confirms that controlled solvent extraction, combined with solvent recovery and careful parameter stabilization, provides a viable and scalable method for producing high-quality natural dyes. Beyond environmental sustainability, the findings establish a scientifically grounded approach to optimizing pigment yield and chromatic intensity for textile applications.

By integrating analytical rigor, green chemistry principles, and indigenous textile relevance, this research contributes meaningfully to sustainable materials chemistry and eco-friendly dye production.

7. RECOMMENDATIONS FOR FURTHER STUDY

To deepen scientific understanding and industrial applicability, the following areas are recommended:

1. **Spectroscopic Characterization**
   Conduct UV-Vis, FTIR, and HPLC analyses to identify and quantify specific pigment compounds.
2. **Optimization via Response Surface Methodology (RSM)**
   Investigate solvent concentration, particle size, solvent-to-solid ratio, and extraction time to maximize yield statistically.
3. **Kinetic Modeling of Extraction Process**
   Apply diffusion and mass transfer models to predict extraction efficiency.
4. **Thermal Stability and Photostability Studies**
   Evaluate degradation kinetics under heat and UV exposure.
5. **Toxicological and Environmental Impact Assessment**
   Assess biodegradability and eco-toxicity compared with synthetic dyes.

6. **Scale-Up Feasibility Analysis**
   Explore pilot-scale solvent recovery systems and cost analysis for industrial production.
7. **Advanced Green Extraction Techniques**
   Compare cold maceration with ultrasound-assisted extraction, microwave-assisted extraction, or supercritical fluid extraction for improved efficiency.

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