

Extraction and Visual Test for Colour Fastness of Eco-Friendly Dye from Natural Plant (Calotropis Procera)

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Abstract

The study was on Extraction and Visual Test for Colour Fastness of Eco-Friendly Dye from Natural Plant (Calotropis Procera). The objectives of the study were the extraction of natural dye from Calotropis Procera plant; application of the extracted dyes on a fabric (white cotton); testing for color fastness of the dye on the fabric (cotton) using wash fastness; and testing for the color fastness of the dye on the fabric (cotton) using light fastness. The bark of Calotropis Procera was purchased from Itam market in Itu Local Government Area, Akwa Ibom State. The dye was extracted using maceration and Soxhlet techniques with distilled water and ethanol respectively. The dye was extracted in four states. Using Soxhlet technique with ethanol and distilled water as solvents, the colours obtained after filtration were Nespresso and marigold respectively. Using maceration technique with ethanol and distilled water as solvents yielded Hollister and honey colours respectively. The extracted dyes were applied on cotton fabric treated with mordant. Colours obtained after dyeing using Nespresso was yellow, marigold gave blonde, Hollister was honey and honey colour resulted to butter. The wash fastness visual results for cotton fabrics dyed with dye extracted using Soxhlet and maceration with distilled water as solvent were rated 1-2 poor while the ones extracted with ethanol as solvent using same techniques were rated 3 and 2-3 respectively.

Keywords: *Extraction, Visual Test and Colour Fastness*

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Background to the Study

Natural dyes, recognized for their eco-friendly attributes, are compounds that offer recyclability and biodegradability (Aiam et al., 2020). These dyes carry minimal ecological impact, being sustainable, cost-effective, and possessing mild yet vibrant shades. Additionally, they exhibit antimicrobial properties, are non-toxic, ensure safe production, generate minimal waste, and align with environmental concerns related to water consumption (Narayana et al., 2016; Aiam et al., 2020).

The escalating interest in natural dyes is attributed to stringent environmental standards imposed globally, responding to the toxic and allergic reactions associated with synthetic dyes (Kumaresan Palanisamy and Kumar, 2011). While synthetic dyes dominate commercial dyeing processes, the resurgence of interest in natural dyes is driven by a worldwide focus on eco-friendly and biodegradable materials (Agulei, 2016). Natural dyes have gained significance in the textile industry, aiming to replace synthetic dyes and mitigate environmental conflicts.

In Nigeria, diverse plant species harbor the potential to yield novel nature-inspired dyes (Nwonye, Ezema, and Thompson, 2017). These dyes can be extracted from various sources such as plants, animals, and minerals, with plant-based dyes being the most prevalent, derived from roots, bark, stems, leaves, flowers, fruits, and shells (Nwonye and Ezema, 2017). The advantages of natural dyeing encompass the gentle formation of colors, environmental friendliness, and functional benefits for textile users compared to synthetic dyes (Lami et al., 2019).

Synthetic dyes, synthesized from petrochemical sources and involving hazardous chemical processes, pose threats to the environment. They are known for causing carcinogenic effects on human skin, and various types of synthetic dyes, such as direct, acid, basic, reactive, mordant, metal complex, vat, sulphur, and disperse dyes, are associated with suspected harmful chemical releases (Mansour, 2013). The environmental impact of synthetic dyes extends to the generation of hazardous waste, containing toxic and heavy chemicals, contributing to ecological damage in surface water systems (Agulei, 2016).

The rebirth of interest in natural dyes is driven by environmental consciousness, with extraction being a key method in obtaining these dyes. Extraction involves using suitable solvents to attract the active substance from the plant material (Youstiana and Suhartono, 2016; Jadhav, 2023). Different extraction methods include aqueous extraction, maceration, Soxhlet, infusion, and percolation, each influencing the efficiency of the process based on factors like solvent properties, raw material particle size, and extraction duration (Mansour, 2017; Hidayat and Wulandari, 2021; Jadhav, 2023).

As textile and clothing industry processes contribute to atmospheric emissions and environmental imbalance, the long dyeing process, especially with synthetic dyes, generates harmful chemicals, posing threats to worker health and environmental pollution. The non-biodegradable nature of synthetic dyes leads to soil pollution, and the production of synthetic

dyes results in hazardous waste containing toxic and heavy chemicals, impacting aquatic life and human health (Nwonye Ezema, and Priscilla, 2017). Thus, the exploration of natural dyes becomes pivotal in fostering sustainability and minimizing the ecological footprint of the textile industry.

The prevalence of environmental issues stemming from the production and application of synthetic dyes, coupled with the evolving dynamics of modern development, has reignited consumer interest in natural dyes. The increasing global appreciation for the organic value of eco-friendly products has directed consumer attention towards textiles dyed with natural dyes. Despite the current dyestuff requirement for the textile industry being around 3 million tonnes, with natural dyes accounting for only 10-25%, integrating natural dyes into mainstream textile processing remains a significant challenge. Moreover, the existing scientific studies and systematic reports on the dyeing of textiles with natural dyes are insufficient, leaving numerous untapped natural products. Hence, the relevance of this research project becomes apparent.

Objectives of the Study: The overarching goal of this research was to conduct the Extraction and Visual Test for Colour Fastness of an Eco-Friendly Dye derived from the Natural Plant *Calotropis Procera*. The specific objectives included:

1. Extracting natural dye from the *Calotropis Procera* plant.
2. Applying the extracted dyes to white cotton fabric.
3. Testing the color fastness of the dye on cotton fabric using wash fastness.
4. Testing the color fastness of the dye on cotton fabric using light fastness.

Research Questions

1. What are the dye colors extracted from *Calotropis Procera* using maceration and Soxhlet methods?
2. What colors are obtained after applying the dye to white cotton fabric?
3. What is the wash fastness of the dye extracted from *Calotropis Procera*?
4. What is the color fastness achieved from the dye on cotton fabric using light fastness?

Materials and Methods

Materials: The bark of *Calotropis Procera* and tamarind plants was purchased from Itam central market in Itu Local Government Area, Akwa Ibom State. The white cotton fabric, ethanol, and alum were obtained from the same market. Equipment used for the work were Ph Meter 7010, thermometer, filter paper, distilled water, manual blender, sieve, ethanol, alum, beakers, cotton material, water bath, pot, soxhlet, extractor. The equipment was obtained from the chemistry laboratory of the Department of chemistry, University of Uyo, Uyo Akwa Ibom State, Nigeria. Water was also provided in the chemistry laboratory.

Treatment of Samples for *Calotropis Procera*

The outside of the bark (the dark part) of the plant was scraped, this is to remove dirt that may cause adverse effect on the extraction process of the dye stuff. The sample was reduced to

smaller size and grind to powder using electric blender. This is essential for the soxhlet extraction process because the smaller the particle size the greater the surface area of the powdered particle, large surfaces improve the contact of the powdered particles with solvent used for extraction (Das, Tiwari and Shrivasta, 2010).



Fig 1: Samples of the plant

Treatment of substrate (cotton fabric)

Scouring of the substrate: Scouring is the first procedure for any dyeing processes, the substrates (white cotton) was simmered in a solution of dish soap. This removed the oil, wax or dirt that might interfere with dye adhering to the fiber after the substrate was rinsed properly (Nwonye and Ezema, 2017).



Fig. 2: Scouring the Substrate

Mordanting the Substrate

In order to charge the substrate to be dyed. Potassium aluminum sulphate which is locally known as alum was used for the treatment. Two litres (2ls) of water were filled in a pot. 1 tablespoon (1tb) of alum was added to it plus the substrate. This was brought to a boiling point of 100c with constant stirring and simmering for two hours. The substrate was allowed to cool off while still inside the pot of alum solution overnight. After, the mordanted substrate was removed from the pot for dyeing.



Fig 3: Mordanting the Substrate

Method of Dye Extraction

Cold Maceration for Calotropis Procera

40gs of the samples (*Calotropis Procera*) was added to a bowl containing 1 litres of distilled water separately and agitation was done at intervals for 48 hours for complete extraction. The resultant mixture was filtered using cotton wool and filter funnel. Similar experiment was conducted with ethanol to compare the extraction power of the solvents. The temperature, and pH of each extract was taken and recorded.

Soxhlet Extraction

This method of extraction is carried out when a compound of low solubility needs to be extracted from a solid mixture (Nwonye and Ezema, 2017). This technique involves building a rig using retort stand and clamps to support the extraction apparatus. Following this, the solvent (250ml of ethanol) was added to round bottom which was attached to Soxhlet extractor and condenser on the heating mantle. 40gs of the powdered sample material was loaded into the thimble, which was placed inside the soxhlet extractor. The solvent was heated using the heating mantle, the refluxing solvent repeatedly washes the solid extracting the desired compound into the flask. This happens when the solvent begins to evaporate, moving through the apparatus to condenser. The condensate then drips into the reservoir containing the thimble. Once the level of solvent reaches the siphon it pours back into the flask and the cycle begins again. Soxhlet extractor comprises of the condenser, thimble, siphon tube, solvent tube and round bottom flask. The extraction process was carried out for 8 hours on the sample and the temperature for extraction was 78.5°C which is the boiling point for ethanol. The extraction process was carried out four (4) times. The same procedure was used for the soxhlet extraction using water as solvent with a temperature of 100°C which is the boiling point for water. Several cycles of solvent were run so as to extract all the compound from the sample.

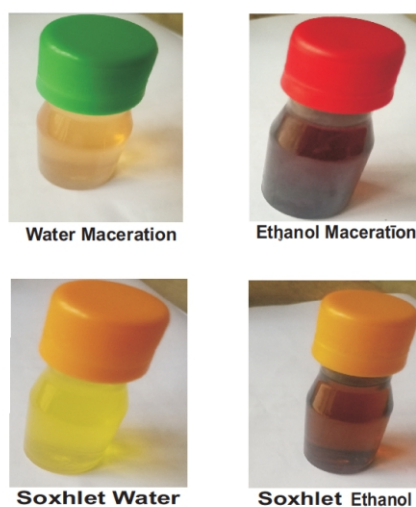


Fig 4: Extracted Dyes from Calotropis Procera

Dyeing Procedure

The magnitude of the fabric to be dyed with the dye mixture was constant in all the operations. The measurements of the substrates were 15cm by 11cm. 100ml dye solution was measured into a bowl, the substrates were submerged into the bowl differently. The substrates were allowed to remain in the bowl and stirred approximately for 35 minutes respectively and was later removed and aired for oxidation to take place for 5minutes. They were both rinsed separately and dried under the shades without squeezing. This operation was achieved by the dyes obtained from Maceration and soxhlet techniques.



Calotropis water maceration



Calotropis ethanol maceration



Calotropis water Soxhlet



Calotropis ethanol soxhlet.

Fig 5: Different Shades of Colours obtained using the extracted dye on mordanted fabrics.

Test for Colour Fastness

Light fastness for Calotropis Procera

Light fastness was carried out by exposing the dyed cotton substrates to sunlight for five sunny days between hours of 10:30am to 4:30pm, the second set were kept under a shade. The exposed fabric was compared to the unexposed dyed fabric (Osabohein and Ukponmwan, 2002). The exposed fabric turned out to be lighter in colour than the unexposed fabric.

Washing Fastness for Calotropis Procera

Each of the dye fabrics of (calotropis procera) was stapled with an undyed piece of white fabric separately, this is to check for staining after the washing. The fabrics was washed dipped into a separate soap solution made by adding 5g of Wash detergent into 200ml of deionized water and stirred vigorously for 30 minutes, the washed fabric was rinsed and dried under a shade while another set of fabric that was dyed with same techniques was set aside for control purpose. The two sets were rated according to their different washing fastness. The treated fabrics were compared with the untreated fabric.

Assessment Procedure: Four final year clothing and textile students were used as assessors. Their total number was four (04). This group was purposively selected because they have been exposed to the courses involving extraction of dye, textile printing and colour fastness. They were educated on how to respond to the scoring sheet by the researcher and one research assistants. After their scoring, the data was transferred to the appropriate column.

Result

Research Question 1

What is the dye colours extracted from the plants (calotropis procera and tamarind) plants using soxhlet and maceration techniques of extraction?

Table 1: Colour obtained from the extraction of calotropis procera using soxhlet and maceration techniques.

Plant	Technique	Solvent	Temperature	Ph	Time of extraction	Colour observed after filtration
Calotropis	Soxhlet	Ethanol	24 ^o C	5	6 hours	Nepressco
Calotropis	Soxhlet	Water	24 ^o C	5	48hours	Merigold
Calotropis	Maceration	Ethanol	78.5 ^o C	5	6 hours	Hollister
Calotropis	Maceration	Water	100 ^o C	5	48hours	Honey

Source: Field work (Nwonye, et al, 2023)

Table 1 showed the colours obtained from calotropis procera. Using soxhlets and cold maceration technique of extraction. For ethanol soxhlet of calotropis procera colour observed after filtration was Nepressco. The water soxhlet gave Merigold after filtration. While for ethanol maceration Hollister was observed after filtration. In water maceration honey was observed after filtration.

Research Question 2

What are the colours obtained from the dye after application on the cotton fabric?

Table 2: Colours obtained from the dyes after Application on the cotton Fabric

Plant	Technique	Solvent	Type of fabric	Time of dyeing	Colour observed after dyeing
Calotropis	Soxhlet	Ethanol	White cotton	1 hour	Yellow
Calotropis	Soxhlet	Water	White cotton	1hour	Blonde
Calotropis	Maceration	Ethanol	White cotton	1hour	Honey
Calotropis	Maceration	Water	White cotton	1hour	Butter

Source: Field work (Nwonye, et al, 2023)

Table 2 shows the colours obtained after applying the extracted dyes on a white cotton fabric. Ethanol soxhlet gave yellow colour after dyeing. Colour obtained from water soxhlet after dyeing was blonde. While colour observed after dyeing with dye extracted through ethanol maceration was honey. Water maceration gave butter after applying the dye gotten through the technique on white fabric.

Research Question 3

What are the colour fastness properties obtained from the dyes on cotton fabric using wash fastness?

Table 3: Colour fastness properties of Calotropis Procera Plant obtained from the dye on cotton fabric using wash fastness.

Plant	Technique	Solvent	Type of textile white	Colour obtained	Time (min)	Colour change
CalotropisCi	Soxhlet	Ethanol	Cotton	Yellow	30	3
CalotropisCii	Soxhlet	Water	Cotton	Blonde	30	1-2
CalotropisCiii	Maceration	Ethanol	White	Honey	30	2-3
CalotropisCiv	Maceration	Water	White	Butter	30	1-2

Source: Field work (Nwonye, et al, 2023)

5 outstanding; 4-5 excellent; 4 very good; 3-4 good; 3 moderate; 2-3 slightly fair; 2 fair; 1-2 poor; 1 very poor

Table 3 revealed that fabrics dyed with dye obtained from calotropis plant within the washing time of 30minutes, using ethanol soxhlet gave a 3 moderate colour. While fabrics dyed with dye obtained from water soxhlet and water maceration rated 1-2 which is a poor colour, ethanol maceration exhibited a slightly fair colour.

Research Question 3

What are the colour fastness obtained from the dye on cotton fabric using light fastness?

Table 4: Colour fastness obtained from the dye on cotton fabric using light fastness.

Plant	Technique	Solvent	Type of cotton	Colour obtained	Time (min)	Color change
Calotropis Ca	Soxhlet	Ethanol	Cotton	Yellow	5days	3
Calotropis Cb	Soxhlet	Water	Cotton	Blonde	5days	2-3
Calotropis Cc	Maceration	Ethanol	Cotton	Honey	5days	1-2
Calotropis Cd	Maceration	Water	Cotton	Butter	5days	2-3

5 outstanding; 4-5 excellent; 4 very good; 3-4 good; 3 moderate; 2-3 slightly fair; 2 fair; 1-2 poor 1 very poor

Table 4 showed that the colour change of calotropis procera using ethanol soxhlet as exposed of the fabric to sunlight for 5 days gave a moderate colour 3, it showed that water soxhlet gave slightly fair of 2-3, ethanol maceration observed 1-2 poor colour, and water maceration gave 2-3 slightly fair. When compared with the unexposed fabric, the table revealed that only ethanol soxhlet gave a moderate colour.

Discussion of Findings

The findings revealed the colours of natural dye obtained from the plants Calotropis procera through maceration and soxhlet techniques as shown on Table 1, the effect of these techniques on the colour fastness on 100% cotton mordanted with alum to sunlight (light fastness) and washing (wash fastness).

The findings unveiled the colours obtained from the plants Calotropis procera after filtration, using soxhlet technique. Ethanol soxhlet of calotropis procera gave Nepressco colour, water soxhlet gave Merigold colour. For maceration, ethanol maceration gave Hollister, water maceration gave honey colour. After observing the colours obtained from the extraction of the plant using soxhlet and maceration technique. The result agrees with the findings of Yusuf et al (2017) that some plants may possess more than one shade of colour depending on the part of plant they are extracted, and solvent used for extraction. From another point of view, Amruta et al (2023) stated that colour content and colour yield of natural dyes are comparatively less than synthetic. The finding also unveiled the colours obtained from the dye after application on the cotton fabric. Ethanol soxhlet of calotropis procera gave yellow colour, water soxhlet gave blonde colour, for maceration technique, ethanol maceration gave honey colour, water maceration gave butter colour. The different in shades of this colours may be as a result of techniques employed in extracting the dyes. This finding agrees with the statement of Korankye (2010) that Natural colours are beautiful to behold.

The findings unveiled that the colour fastness properties of the plant carried out using washing fastness for 30mins. It revealed their colour changes respectively, ethanol soxhlet of calotropis gave moderate colour 3, water soxhlet gave 1-2, ethanol maceration gave slightly fair 2-3, while water maceration observed 1-2 very poor colour. This finding agrees with the finding of campbell, (2013) and Bukhari, et al (2017) which confirmed that the degree of colour fastness of fabric, is evaluated according to the discoloration of a sample and the staining of the

undyed lining fabric. And it also agrees with the finding of Nwonye and Ezema (2017) which states that the use of mordant in textile application is found to be fruitful to improving colour adherence and shades of the dyes.

The findings also rated the colour fastness obtained from the dye on cotton fabric using light fastness. For Calotropis the plants had a moderate rating of 3 using ethanol soxhlet, water soxhlet 2-3 which is slightly fair, ethanol maceration 1-2 very poor and water maceration 2-3 same slightly fair. While water soxhlet, ethanol maceration, and water maceration rated slightly fair 2-3. Ethanol soxhlet revealed that the colour change was 2-3 slightly fair, water soxhlet was 3-4 good colour while ethanol maceration gave 3 moderate colour and water maceration rated a very poor colour of 1-2. This finding is in line with that of Vilaren (2013) which states that light fastness of dyed fabric is influenced by chemical, physical state and concentration of dye, nature of the fibres and mordant type.

Conclusion

In summary this work showed that different hues of dye can be extracted from the plant of calotropis procera. The process of the extraction was eco-friendly. The dye hue obtained had the dyeing potential which can be used as a source of textile dyeing. The results of the experiment with the part of the identified plants were a remarkable range of colourful direct dyes of various shades and hints. The use of mordants in textile application is found to be fruitful to improving colour shades of the dyes. The study reveals that their properties are preferable to synthetic dyes are toxic. Fabrics dyed with dye obtained from solvent soxhlet have a good colourfastness properties than the ones extracted by maceration. From this study it was possible to conclude that the dyes obtained from natural plants of (calotropis procera) can be successfully used for dyeing of cotton fabrics to obtain a wide shade range.

Recommendations

1. The government should support further research on natural dye exploration to mitigate environmental damage caused by synthetic dyes.
2. Home economics departments in educational institutions should incorporate natural dye extraction into their syllabi to educate students on the health benefits of natural dyes.
3. Students should engage in practical extraction of dyes from natural plants, fostering cost-effective and sustainable practices.
4. Educational institutions can use the study as a resource material to teach creative skills in dyeing fabrics, promoting sustainable practices.
5. Vocational institutions and NGOs involved in skills training can encourage trainees to cultivate herbal plants for dye extraction, fostering sustainable practices and health benefits.

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