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RESEARCH ARTICLE

Dyeing and antibacterial finishing of cotton fabric using *Diospyros mespiliformis* leaves extracts

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Abstract

In this study, leaves of *Diospyros mespiliformis* was extracted using ethanol and macerated with chloroform and ethyl acetate and later screened for antibacterial properties. The antibacterial analysis was performed qualitatively via disc diffusion method (AATCC 147) against *Escherichia coli* and *Staphylococcus aureus* as gram negative and gram positive bacteria respectively. The extract shows a significant activity on the isolated micro-organisms as evidenced by a clear zone of inhibition, more so in the ethyl acetate petri dish indicating its potentials in preventing microbial growth. The extract was then used to dye cotton fabrics by pre-mordanting, simultaneous mordanting and post-mordanting processes. The materials were assessed for fastness properties, such as fastness to washing, light, pressing and rubbing based on AATCC standards. The results show a good to moderate fastness properties suggesting that extract of *Diospyros mespiliformis* can be successfully used for dyeing of cotton fabric in textile industries thereby reducing the pollution and environmental hazards associated with the use of conventional synthetic chemical dyes.

***Keywords*:** Antibacterial, *Diospyros mespiliformis,* dyeing, fastness textile

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INTRODUCTION

Textiles, particularly those obtained from natural fibers are an excellent shelter for the growing of microorganisms when the basic requirements such as oxygen, moisture, and nutrients, appropriate temperature are present. This growth of microorganisms is due to the large surface area of textiles and their capacity for moisture retaining. Microorganisms such as bacteria are part of our everyday life, and there are both beneficial and harmful types [1, 2]. The key antimicrobial agents for textile coating are chemicals such as quaternary ammonium compounds [3], which have deadly and environmental consequences. The concern for the environmental concerns resulted in an increased demand for natural dyes [4]. Natural dyes are found to be environmentally friendly and possess many advantages over their synthetic counterparts. The application of natural dyes dates back to ancient times where it found applications in many aspects including foods, cosmetics, medicine, and in leather industries [5-7]. Natural dyes are acquired from natural sources such as insects, animals, and plants. Once the synthetic dyes were discovered in 1856, the use of natural dyes in textile dyeing decreased to a large extent [8]. However, awareness for natural dyeing was later recovered due to their lower toxicity and biodegradability [9]. Numerous naturally obtained dyes from plants source display excellent antimicrobial properties. As a result, an antimicrobial coating of cotton using natural dyes from plant source plays a major role in the production of medical cloths [1]. There is an excessive demand for antimicrobial finishes of textiles to control the growth of microorganisms. These microorganisms include bacteria, fungi, or mildew, and prevent the textile from deterioration of strength and quality, staining, odors, and health concerns caused by microorganisms [10, 11]. The use of natural products such as natural dyes for antimicrobial finishing of textile materials has been widely reported [12, 13]. The use of natural antimicrobial agents on textiles dates back to ancient times when the earliest Egyptians utilized herbs and spices in the preservation of mummy wraps. Several natural dyes obtained from diverse vegetation are known to possess antimicrobial properties [14, 15]. It has become increasingly essential for antibacterial agents to meet low toxicity and environmental standards and at the same time retain their functionality. Hence, it is vital to research and develop environmental friendly antibacterial agents extracted from plants for use in textile materials. Effects of many plants on bacteria have been studied by some researchers [16, 17]. *Diospyros mespiliformis* belongs to the family *Ebenaceae* and found in woodlands, savannahs and along riverbanks mostly areas with permanent water that helps in natural regeneration. It is a tall plant usually 15-50 m high with dense rounded and buttressed stem. The mean annual temperature of 16 – 27 °C and an annual rainfall of 500 – 1270 mm and an altitude of 350 – 1250 are ideal for the growth of the plant [18]. In Nigeria, the plant is found in different parts of the country

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and called by various names such as Onye-Koji (Igbo), Kanya (Hausa) and Igidudu (Yoruba). The roots and the bark are also used in traditional medications [19-21].

The objectives of the research are therefore to study the antibacterial activity of *Diospyros mespiliformis* plant extracts and to examine its effectiveness in dyeing cotton fabric.

EXPERIMENTAL

Materials

Fresh leaves of *Diospyros Mespiliformis* were collected at random from the agricultural farms located at Zogarawa, Dawakin Kudu Local Government Kano State, Nigeria. It was authenticated and identified at the Department of Plant Science, Bayero University Kano, Nigeria. The collected leaves were washed thoroughly and chopped into smaller pieces, air-dried under shade at room temperature and later made into powder by grinding. The coarse powder was percolated with ethanol at room temperature in a ratio of 1:5 of plant: solvent in a beaker. The beaker was left covered for 14 days the residue was separated from the extract by evaporation. Methanol (30 mL) was added to the left over ethanol extract acquired from the first maceration and was stirred to dissolve those parts of the ethanol extract which are soluble in methanol. An empty beaker was used to collect the resulting methanolic solution. The process was repeated two to three times with (20 mL) methanol until there was no longer dissolution of the ethanol component. The extract was reserved for further use. Similar procedure was adopted for the ethyl acetate and chloroform extracts.

The cultures of the gram positive organism, *Staphylococcus aureus*, and gram-negative *Escherichia coli* were used. The test microorganisms which were clinical isolates were sourced from Microbiology Department of Aminu Kano Teaching Hospital, Kano-Nigeria. The 5 mm disc was prepared from the Whatmann No. 1 filter paper and stored at 4ºC in dark place for further use. The absorption capacity of the disc was 0.008 µL per dip in distilled water. This absorption capacity was varied [7].

Preparation of the media

Nutrient Agar (23 g) was dissolved in one liter of distilled water, according to the instructions given by the manufacturers. The Nutrient Agar solution was put in an autoclave for 15 minutes at 121 °C. Sterilized Nutrient Agar (20 mL) was transferred into Petri-dishes and were allowed to cool and solidify [22].

Fabric preparation for coating

The mercerization and scouring of the textile fabric were carried out [23, 24]. The cotton fabric was pre-treated by caustic boil method. It was washed in hot water with 500 mL of Na_2CO_3 (g/L). Cotton fabric 5.05 g and 2.2-inch length was washed in dissolved 0.3155 g/L of Na_2CO_3. It was then put in a water bath, and the temperature was raised to boil with continued stirring for 15 minutes. The fabric was recovered and air-dried. The bleaching of the scoured fabric was achieved by treating with 2 g of ascorbic acid and 3 mL of sodium hypochlorite in 200 mL of water and kept at 90 °C for 10 min. The fabric was dried and washed with sterile water and was allowed to air-dried [7, 25].

Fabric coating

The fabric was coated using exhaust method and mordanted with acetic acid before dyeing. The mordant was 5% by weight of the fabric (0.25 mL of acetic acid for 5.05 g cotton fabric), and the liquor ratio was 40:1. The treated fabric was introduced into the plant extract i.e. 5% of *Diospyros mespiliformis* and was sustained at 50 – 55 °C for 45 minutes [7, 25].

Qualitative test (AATCC-147)

The Disc diffusion technique was adopted for the qualitative test. About 0.025 g of the extracts were dissolved in 0.2 mL of sterile H_2O. The sample of 0.01, 0.025 0.050 and 0.1 mL was slowly permeated drop-wise on 5 mm sterile Whatman paper disc and also to cotton fabric. The treated samples were placed in intimate contact with agar, which was previously inoculated with an overnight culture of *Staphylococcus aureus*, and *Escherichia coli*, and incubated at 37 °C for 18 -24 hours. The zone of inhibition was assessed.

Dyeing and mordanting

5.05 g and 2.2-inch length of cotton cloth were treated with different metal salts 1% of mordant. (Mordants used 0.5% Cupric-Sulphate and 0.5% ferrous sulphate). Three processes of mordanting were used; pre-mordanting simultaneous mordanting and post-mordanting 4% dye and 2% mordant and 1:40 mordant: Liquor ratio was used, and the dyeing was carried out at 80 °C for 60 mins. After dyeing, the material was washed with cold water and dried at room temperature [7, 26].

Assessment of colour fastness properties

The assessment of the wash fastness of the dyed samples was performed using standard procedures [27]. The specimens were cut into 5 cm by 4 cm and place in between two pieces of un-dyed cotton fabrics of equal size. This sample was sewn together and made into a composite specimen; the composite specimens were separately immersed into washing liquor containing 100 $cm^3$of 4 g dm^{-3}detergent solutions and agitated for 30 minutes at 50 ºC. The specimens were thoroughly rinsed, opened and air dried. The change in color of the dyed material and the degree of staining of the un-dyed fabric were assessed using grayscale [28].

The light fastness was evaluated using the standard methods (BS 1006: BOI 1978). The test was carried out on artificial light fastness tester MK 1 fitted with mercury-tungsten (MBTF) 500-watt lamp for 96 hours. The samples were exposed to light along with the eight blue wool standards. The fading of each sample was observed against the fading of blue wool standards [29]. In order to assess the fastness to rubbing of the dyed fabric, a piece of dyed sample was rubbed on a plane dry white cloth and then a wet white cloth. The staining of the white cloth was observed and assessed using grayscale [28].

Dry pressing was carried out by positioning a colored specimen on a piece of dry white cloth. Iron was placed on top for 15 s. Damp pressing was assessed by placing the dry white cloth on an ironing board; the wetted specimen of cotton fabric has been put on top while a piece of wet cloth was below. Iron was then positioned on top and moved for 15 s [30].

RESULTS AND DISCUSSION

Qualitative test results

The experiment was carried out twice and under sterile conditions to avoid any possible contamination that might occur, and zone of inhibition of each concentration was measured and recorded (Table 1).

It can be seen from Table 1 that, *Diospyros mespiliformis* extract in both chloroform, ethanol and ethyl acetate shows a higher zone of inhibition at 100 mg/mL concentrations followed by those with lower concentrations. This higher inhibition is an indication that the activity depends on the concentration of the active specie, ~~as~~ since higher concentration reveal a higher zone of inhibition. It can, therefore, be established from the above results that the plant extract exhibits a broad spectrum of activity.

The stock solution of ethyl acetate shows higher antibacterial activity against both the *Staphylococcus aureus* (gram positive bacteria) and *Escherichia coli* (gram-negative bacteria) followed by Chloroform stock solution, and high resistance in the case of the Ethanolic stock solution against the *Staphylococcus aureus* (gram positive bacteria) was observed. This study reveals the antibacterial efficiency of *Diospyros mespiliformis.* Previous studies on the antibacterial activity of Diospyros mespiliformis showed inhibitory activity against a broad spectrum of microorganisms [7].

Colour fastness properties

The performance of dyed textile fabrics when exposed to various conditions is generally assessed by appropriate color fastness testing [30, 31]. These conditions include perspiration, heat, washing, light and atmospheric pollution. The color fastness test environments are selected to closely resemble the real life treatments encountered during manufacture or end-use [32]. The fastness test results are achieved by

comparing the test samples with a standard reference known as gray scale [7, 32]. Results of the color fastness tests are presented in the Tables 2 – 4.

Table 1 Zone of inhibition and concentrations of the extract

Extract	Concentration (mg/ml)	Zone of inhibition *(mm)*	
		E. coli	*S. aureus*
Chloroform	100	+++	++
	50	+++	++
	25	+++	++
	12.5	++	++
Ethanol	100	+++	+++
	50	+++	++
	25	+++	++
	12.5	+++	++
Ethyl Acetate	100	+++	+++
	50	+++	++
	25	+++	++
	12.5	+++	++

Zone of inhibition (mm); 0 -3 +, 4-6 ++, 7-10 +++.

Table 2 Fastness to washing.

Method of Mordanting	Mordant Ratio ($CuSO_4$: $FeSO_4$)	Fastness Rating	Colour Staining
Pre-mordanting	1:3	3	½
	3:1	3	½
Simultaneous Mordanting	1:3	3	2
	3:1	3	1
Post Mordanting	1:3	3	2
	3:1	3	2

It is evident from Table 2 that, the dyed samples show poor fastness to washing, as they completely changed from greenish color to the brownish color and similarly all the samples shows poor staining. Thus, *Diospyros mespiliformis* leaves extract to have moderate fastness to washing when dyed on cotton. The rating indicates that the cotton fabric samples dyed a similar fastness rating of 3 irrespective of either it was free mordanted, simultaneous mordanted or post-mordanted.

Table 3 Fastness to rubbing.

Method of Mordanting	Mordant Ratio ($CuSO_4$: $FeSO_4$)	Dry Rubbing		Wet Rubbing	
		Fastness	Staining	Fastness	Staining
Pre-mordanting	1:3	5	4/5	4	3
	3:1	5	4	4	3
Simultaneous Mordanting	1:3	5	4/5	4	3/4
	3:1	5	4	4	2/3
Post Mordanting	1:3	5	4/5	4	2/3
	3:1	5	4	4	2

For the wet rubbing, all the samples show partial color change. A moderate staining was also observed for the samples dyed using pre and simultaneous mordanting, while poor staining was seen in the samples dyed using post-mordanting process (Table 3).

Table 3 Fastness to pressing.

Method of Mordanting	Mordant Ratio	Dry Pressing		Wet Pressing	
		Fastness	Staining	Fastness	Staining
Pre-mordanting	1:3	4/5	4/5	4/5	3/5
	3:1	5	4/5	4	3
Simultaneous Mordanting	1:3	4/5	4/5	2/3	3/4
	3:1	2/3	4	4/5	3
Post Mordanting	1:3	5	5	5	3
	3:1	2	4/5	4	¾

Table 4 Fastness to light.

Method of Mordanting	Mordant Ratio	Fastness
Pre-mordanting	1:3	6
	3:1	6
Simultaneous Mordanting	1:3	4
	3:1	3
Post Mordanting	1:3	5
	3:1	5

All the samples show excellent color staining, but the poor color change was observed in the post and simultaneous mordanting samples dyed with 75% $FeSO_4$, while the rest shows a very good color change. The dyed samples show good fastness property to wet pressing as well as fastness to light (Tables 4 and 5). It is evident that the mordant ratio has some effect on the hue as well as the fastness properties. The mordants form an additional linkage with the dye molecules when compared to the fabric dyed in the absence of mordant. The practice of mordanting makes the dye to be more aggregated and more bonded to the fiber thereby rendering reducing the light accessibility to the fiber surface, and hence, improving the fastness to light [7, 33-35].

CONCLUSION

The objective of the present study was to assess the performance of *Diospyros mespiliformis* plant extracts *as an* antibacterial agent and to examine its effectiveness in dyeing cotton fabric. The study highlighted that fabric coated with *Diospyros mespiliformis* leaves exhibit antibacterial property. It was much active when extracted with ethyl acetate rather than in ethanol and chloroform, which was more suitable for antibacterial finishing of a textile fiber. The *Diospyros mespiliformis* leaves extract when dyed to a textile fiber shows excellent fastness to light and rubbing. However, the fastness to washing was moderate as such; further treatment is required to improve the washing fastness. The availability and environmental friendliness qualify *Diospyros mespiliformis* to be used as an effective antibacterial agent and a substitute to conventional synthetic chemical dyes commonly used. Further research on dyeing and fastness tests should be carried out, and study the possible ways to enhance the fixation of the *Diospyros mespiliformis* plant extract to the cotton fabric. Its application on other microorganisms should be explored to justify its wider implementation and acceptability.

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