



## Determination Of Phytochemical Constituents Of Cuberoot Leaves And Evaluation Of Organoleptic Attributes And Acceptability Of Cotton Fabric Treated With Cuberoot (*Lonchocarpus Cyanescens*) Dye.

OZOUGWU, S. U

DEPARTMENT OF HOME SCIENCE, NUTRITION AND DIETETICS, FACULTY OF AGRICULTURE, UNIVERSITY OF NIGERIA NSUKKA

ANYAKOHA, E. U

DEPARTMENT OF VOCATIONAL TEACHER EDUCATION, FACULTY OF EDUCATION, UNIVERSITY OF NIGERIA, NSUKKA

**ABSTRACT**

*This study determined the phytochemical constituents of cuberoot (*Lonchocarpus cyanescens*) indigenous leaves and evaluated the organoleptic attributes and acceptability of 100 per cent cotton fabric treated with dye extracted from cuberoot leaves. The study design was quasi experimental and was conducted at the University of Nigeria, Nsukka. The population was 41 panelists comprising 17 Lecturers and 24 Postgraduate students from University of Nigeria, Nsukka. Phytochemical analysis of cuberoot dye was done using Spectrophotometric and gravimetric methods. Organoleptic attributes and acceptability evaluation [OAAE] instrument was used for data collection and data were analyzed with descriptive statistics. Null hypothesis was tested at 0.05 Level of significance using t-test. Findings include; presence of lutein (.007±.0014), tannin (.129±.0014), polyphenol (.0021±.0028), flavonoid (5.16±.014), carotenoid (2.23±.028), per 100g of cuberoot leaves. Seven cuberoot dye organoleptic attributes on cotton fabric were identified; five organoleptic attributes were unanimously accepted by both categories of evaluators; There were no significant differences (P>0.05) in the mean rating responses of lecturers and postgraduate students on the acceptability of the organoleptic attributes of the cubeeroot dye on cotton fabric in three instances but differences existed in three instances. The null hypothesis was therefore, accepted in three instances and rejected in three instances at 0.05 probability level.*

**KEYWORDS:** Phytochemical, Organoleptic attributes , Cuberoot plant dye, Cotton fabric

**Introduction:**

Cuberoot plant (*Lonchocarpus cyanescens*) is a leguminous plant belonging to the family, pipilionnocea. The common names are: 'Anunu', in Igbo, 'Farinloko' in Hausa, 'Apapo or Ipapo' in Yoruba, 'Agwa' in Ibibio and 'cuberoot' in English (Association for Identification, Conservation and Utilization of Medicinal Plants of Nigeria, ASCUMPON, 2006). Cube root is a woody liane reaching 15 to 20metres in length and when cultivated, forms a shrub. Cuberoot plant is highly valued for its medicinal-potentials for the treatment of cutaneous and subcutaneous parasitic infections, general healing and as laxatives (ASCUMPON, 2006). They are used as sauces, spices and flavouring in food and in the production of dyes for textile colouration, stains, inks, tattoos, mordant and other related uses (Burkhill, 1985) .

In Nigeria, specifically in the south east zone, cuberoot plant has not been adequately explored or utilized for textile and clothing coloration despite the clarion call by the Federal Government of Nigeria through the Raw Materials Research and Development Agency (RMDRC), to source the locally available raw materials to substitute or supplement the imported and expensive ones. Scarcity of raw materials including dye as processing chemical contributed to the reduction of 180 textiles and clothing industries that employed 800,000 workers around 80's to about 25 with 776.000 job losses presently in Nsigeria (Onwuualu,2006; Osagie, 2013).). Moreover, the unavailability of safe natural dyes in sustainable supply hinder effective teaching and learning and skill acquisition not only in textiles and clothing education programme but in other dye utilizing fields. Abundant species of dye yielding plants are locally available. Cube root plants are widely spread in Enugu State wasting in the wild. There is need, therefore to explore cube root plant for dye extraction. This will increase the volume of dye and contribute to ensuring sustainable supply of dye for industrial, educational and family utilization. In response to the call for exploitation of the locally available raw materials, Ozougwu and Anyakoha (unpublished Ph.D thesis), in a factorial experimental study preceding this present study, extracted cuberoot dye from cuberoot leaves using different extraction methods and tested the colour fastness of the dyes on samples of 100 percent cotton fabric pre- mordanted with different dye fixing agents. Findings revealed among others, that cuberoot dye was exceptionally fast in tannic acid and alum treated samples in alldye extraction media used. The study suggested among others, further study on the phytochemical composition of cuberoot leaves and organoleptic evaluation and acceptability of cuberoot dye which formed the focus of this study.

Phytochemicals are chemical compounds that occur naturally in plants.

They include but not all; polyphenols, tannin, carotenoids, flavonoids, lutein, alkaloids, saponin. The presence oftannin, carotenoids, polyphenols, lutein and flavonoids contribute to the colour of plants (Win & Swe, 2008) and other organoleptic attributes of dyes produced from them. Organoleptic attributes of a dye refers to the qualities of the dye that can be seen, touched or felt, perceived or smelt and therefore involves the senses of sight, touch or feel and smell. The organoleptic attributes sought for in a dye include the different dimensions of colour, texture, odour and evenness of shade or level dyeing. Colour is an aspect of visual experience (Websters Collegiate Encyclopedia, 2000). The colour of a dye plays major role and most often the primary motivation for the purchase of textiles and clothing items and accessories. Different dimensions of color include- hue, value, chroma or intensity (Johnson and foster, 1990; Marshal, et al, 2000). Hue from physicist point of view is the wavelength reflected from a material. Different wave lengths indicate different hues and approximately 150 hues can be detected in the visible spectrum (Kolender, 2013). Hue from the artist or dyers' perspective is the name of a color family such as red, blue, green. Colour hue is being described as warm or cool. Warm hues are those found in the sun and fire such as red, yellow, orange and cool hues are those found in water including blue, green and violet. The warmth or coolness of a hue carries with it, an illusion of weight (Johnson and Foster, 1990).

Value describes the lightness or darkness of a color. Chroma or intensity describes the purity of a color expressed as the strength or weakness, dullness or brightness or the degree of saturation of a color. High chroma colors are pure, strong, brilliant, saturated colors and are acceptable, but low chroma colors are muted, weak, grayed and dull and unacceptable. Johnson and Foster (1990) emphasized that each hue in the color wheel is presented at its fullest, purest, chroma meaning that the colour is at its greatest saturation, its greatest brightness, it's most brilliant and fullest intensity.

Texture is a sensory impression involving touch and sight (Marshal et al 2000). Texture absorbs light differently and can change the colour of fabrics. The same dyes applied on different textures produce different colors. The visual aspect of texture is perceived by the eye because of the degree of light absorption and reflection on the surface of the material and can be hard or soft, rough or smooth, hot or cold. Such impressions are the result of sensory impression understood by sight and other sense organs (Bartley, 1996, Hobbs and Rush,1997). The tactile aspect includes the coarseness, softness or crispness and rigidity. Whatever dye applied to finish a fabric that renders it unusable for purposes for which it should serve is unacceptable. A good dye should be soluble

in water or dispersible in a solvent resulting in evenness of shade or level dyeing in fabric. Crocking is the rubbing off of dyes from fabric, an indication that dye was not well absorbed or firmly attached to the fibre. Grayness or crystals of dyes on fabric's surface produce un-even or un-level dyeing which is unacceptable. Finally, a good dye should have a pleasant odor on fabric. Any dye both synthetic or natural that gives offensive odor to fabric is unacceptable.

#### Purpose of the study:

The main purpose of the study was to determine the phytochemical composition of cuberoot leaves and to evaluate the organoleptic attributes and acceptability of cotton fabric treated with dye extracted from cuberoot (*Lonchocarpus cyanescens*) indigenous plant for Clothing and Textiles Education. Specifically, the study:

- extracted dye from cuberoot (*Lonchocarpus cyanescens*) leaves using boiling extraction method
- analyzed phytochemical constituents of cuberoot leaves for polyphenol, tannins, lutein, flavonoids, and carotenoids.
- applied the extracted dye to samples of cotton (100%) fabric mordanted with aluminum sulphate (alum)
- evaluated organoleptic attributes and acceptability of the dye on cotton fabric.

**Hypothesis:** One null hypothesis was tested by the study at 0.05 significant level.

**Ho1:** There is no significant difference in the mean responses of Lecturers and Post graduate students on the acceptability of the organoleptic attributes of cotton fabric treated with dye extracted from cuberoot leaves.

#### Methodology

**Design of the study:** Quasi experimental research design was adopted in this study.

**Area of the Study:** The study was carried out at Analytical and Clothing and Textiles Laboratories, Department of Home Science, Nutrition and Dietetics, University of Nigeria, Nsukka, (UNN) Enugu State, Nigeria.

#### Population for the Study:

The study population comprised 41 evaluators made up of 17 lecturers and 24 Postgraduate students purposively sampled from different departments of Vocational Teacher Education (Home Economics Unit), Home Science, Nutrition and Dietetics, Pure and Industrial Chemistry, Fine and Applied Arts, all from UNN.

**Instrument for Data Collection:** Data were collected using the Organoleptic attributes and Acceptability of the Treated Fabrics Evaluation (OAAE) instrument. The instrument comprised three sections. Section A elicited information on the personal data of the evaluators. Section B collected data on the organoleptic attributes such as colour and its dimensions including the degree of warmth or coolness of hue, lightness or darkness of value, brightness or dullness of chroma or intensity; smoothness or roughness of texture (Sight), softness or coarseness of texture (Feel or touch), pleasantness, odorless or offensiveness of odour and dyeing related quality such as level dyeing or even shade of dye on fabric. Sections B and C were rated on 5 point scale. Section C evaluated the acceptability of the organoleptic attributes of the beetroot dyed cotton fabric samples by the judges where 5 indicates very highly accepted (VHA), 4 indicates highly accepted (HA), 3 indicates averagely accepted (AA), 2 indicates unaccepted (UA) and 1 highly unaccepted (HU) for each of the attributes identified. Cronbach alpha coefficient determined the reliability coefficient of specific clusters in the testing protocols and established at 0.713 and 0.875 for sections B and C respectively.

**Method of data Collection:** Data were collected in three phases. Phase 1 of the study dealt with collection and authentication of plant material, processing of the leaves and phytochemical analysis.

**Procedure:** Fresh cuberoot leaves were collected from the wild at Ibeagwa, Nsukka and authenticated at the Botany Department, UNN: About 2kg leaves were dried under room temperature in the Green House 72 hours at 40°C. After drying, it was milled into fine powder us-

ing Thomas Willey milling machine.

#### Quantitative determination of the chemical constituents.

**Lutein Analysis** was carried out using the diethyl ether spectrophotometric method as described by Nurhidayati & Irianty (2012). About 1g of the powdered sample was treated with 30ml of acetone containing 0.1mg Magnesium Carbonate in a glass beaker with constant stirring for 16 hours. This was filtered and the residue re-extracted with 20 ml acetone until the filtrate became clear. Then 10ml of 50% KOH was added and the filtrate KOH mixture seated on a hot plate for 10 minutes. The solution was put in a separating funnel and 30ml diethyl ether and 20 ml water added. The aqueous layer was discarded while the ether layer was recovered and measured in a spectrophotometer at 445nm against a blank diethyl ether.

**Tannin analysis** was done using spectrophotometric method according to Van-Burden and Robinson (1981). A 500 mg of sample was weighed into a 50 ml plastic bottle. 50 ml of distilled water was added and shaken for 1 h in a mechanical shaker. This was filtered into a 50 ml volumetric flask and made up to the mark. Then 5 ml of the filtrate was pipette out into a test tube and mixed with 2 ml of 0.1 M FeCl<sub>3</sub> in 0.1 N HCl and 0.008 M potassium ferrocyanide. The absorbance was measured at 720nm within 10 min.

#### Polyphenols determination was by spectrophotometric method by AOAC (2005):

The fat free sample was boiled with 50 ml of ether for the extraction of the phenolic component for 15 min. 5 ml of the extract was pipette into a 50 ml flask, then 10 ml of distilled water was added. 2 ml of ammonium hydroxide solution and 5 ml of concentrated amylalcohol were also added. The samples were made up to mark and left to react for 30 min for colour development. This was measured at 505 nm.

#### Flavonoid determination by gravimetric method as described by Bohm and Kocipai- Abyazan (1994) method:

Ten grams of cuberoot sample was extracted repeatedly with 100 ml of 80% aqueous methanol at room temperature. The whole solution was filtered through whatman filter paper No 42 (125 mm). The filtrate was later transferred into a crucible and evaporated into dryness over a water bath and weighed to a constant weight.

#### Carotenoid was determined by gravimetric method as described by Harborne (1973):

A measured weight of each sample was homogenized in methanol using a laboratory blender. A 1:10 (1%) mixture was used. The homogenate was filtered to obtain the initial crude extract. 20ml of ether was added to the filtrate to take up the carotenoid mixed well and then treated with 20ml of distilled water in a separating funnel. The other layer was recovered and evaporated to dryness at low temperature (35-50°C) in a vacuum dessicator. The dry extract was then saponified with 20ml of ethanoic potassium hydroxide and left over night in a dark cupboard. The next day, the carotenoid were taken up in 20ml of ether and then washed with two portions of 20ml -distilled water. The carotenoid extract (ether layer) was dried in a dessicator and then treated with a light petroleum (petroleum spurt) and allowed to stand in a freezer (-100°C). The next day, the precipitated steroid was removed by centrifugation and the carotenoid extract was evaporated to dryness in a weighed evaporation dish, cooled in a dessicator and weighed. The weight of carotenoid was determined and expressed as a percentage of the sample weight. Phase 11 of the study focused on extraction of dye from cuberoot leaves, mordanting and dyeing of the cotton fabric.

**Procedure:** Extraction of dye from cuberoot leaves was done using boiling method as described by Kolender (2003). A portion of 80g cuberoot powder was dissolved in 160ml distilled water in the ratio 1:2 (W/V) and heated at 80°C for 20mins and allowed to cool. The heated portion was filtered with 0.5mm mesh (Particle size) to collect the dye liquor. To pre mordant the fabric, 25g (40"x40") was scoured thoroughly in warm water with detergent three times to remove all sizing. 1litre distilled water was heated and 6.25g aluminum sulphate (alum) and 0.5g sodium carbonate (washing soda) was dissolved in. The wet scoured cotton fabric was immersed and gently but thoroughly stirred so that it opened out in the solution. It was heated at 80°C for 1 hour and allowed to cool overnight in the solution then squeezed off excess water for dyeing. The mordanted cotton fabric was immersed in the dye bath for 1 hour at a temperature of 80°C using the contemporary plain

dyeing method. The colour was modified with additional 0.25g ferrous sulphate added to the dye bath. The dyed fabric was taken out, washed and dried under a shade.

In Phase iii, rating of the organoleptic attributes and acceptability of the dyed fabric was done by a panel of 41 evaluators using a set of 41 copies of the OAAE instrument. The evaluators' mean rating responses were collated for data analysis and the rating was done in a single session.

**Data Analysis:** Data collected from the judges were analyzed using descriptive statistics (Mean and standard deviation. Mean 3.00 and above indicate positive and accepted organoleptic attribute whereas mean below 3.00 indicate negative and unaccepted organoleptic attribute. t-test statistic was used to test one null hypothesis at 0.05 probability level

**Summary of Findings**

- Phytochemical analysis of cuberroot leaves indicated the following components; lutein(.007±.0014), tannin(.129±.0014), polyphenol (.0021±.0028), flavonoid (5.16±.014) and carotenoid (2.23±.028), percentages per 100g of cuberroot leaves (Table 1)
- Seven cuberroot dye organoleptic attributes on cotton fabric were identified(Table2).
- Five organoleptic attributes were accepted by both categories of evaluator. There were no significant differences (P>0.05) in the mean rating responses of lecturers and postgraduate students on the acceptance of the organoleptic attributes of the cuberroot dye on cotton fabric in six instances but difference existed in one instance. The null hypothesis was therefore accepted in six instances but rejected in one instance at 0.05 probability level (Table 3).

**Table 1: Percentage of crude lutein, tannin, polyphenol, flavonoids, and carotenoid on cuberroot leaves per 100g**

Constituents	N	Mean	Standard Error	Standard Deviation
Lutein	2	.007	.00100	.004161
Tannin	2	.1290	.00200	.00283
Polyphenol	2	.1595	.00150	.00212
Flavonoid	2	5.1600	.01000	.01414
Carotenoid	2	2.2300	.02000	.02828

Table 1 above indicated that among the phytochemical colour related components determined, flavonoid content was the highest in quantity contained (5.16±.014) followed by carotenoid (2.23±.03). Polyphenol and tannin were present in reasonable amounts (.16±.002 and .13±.002 respectively). Lutein was the least phytochemical component possessed by cuberroot leaves (0.007±.001).

**Table2: Mean Rating Responses of Lecturers and Postgraduate Students on the Organoleptic Attributes of Cotton Fabric Treated with Dyes Extracted from Cuberroot Plant.**

S/N	Cuberroot Organoleptic Attributes	Xi	Ni	SDi	Xii	Nii	SDii
1	Colour hue (Army green)	3.18	17	1.381	2.96	24	1.122
2	Colour value	3.35	17	.931	3.83	24	.381
3	Colour chroma	3.65	17	1.057	3.21	24	.833
4	Texture (sight)	3.94	17	.556	4.00	24	.417
5	Texture (feel)	4.12	17	.485	4.00	24	.511
6	Odour (smell)	2.71	17	1.160	2.50	24	.933
7	Evenness of shade	3.41	17	.600	3.67	24	.482

Key Xi - Mean of Lecturers Nii - No of Postgraduate Students  
 Ni -Number of Lecturers SDii -Standard Deviation for Postgraduate Students  
 SDi- Standard Deviation for Lecturers t/cal-T calculated d/f- Degree of Freedom  
 Xii- Mean for Postgraduate Students NS -Non Significant S-Significant

Table 2 shown that all the cuberroot organoleptic attributes except odour (2.71) were rated positively by lecturers ranging from 3.18±1.38 to 4.12±.485. Five were rated high by the Pg students and five of the

attributes were agreed on simultaneously. While Lecturers agreed that cuberroot hue was fairly warm, it was dull for Pg students (3.18±1.38). Both agreed that colour value and chroma were fairly light and bright respectively. Texture as it relates to sight was fairly smooth for Lecturers but smooth for Pg students. For feel texture, both agreed that it was soft. Cuberroot odour was offensive for both categories of evaluators (2.7±1.16 and 2.50±.933 respectively). The highest scored organoleptic attribute was feel related texture (4.12±.485) by the lecturers and for PG students (4.00±.511), showing soft textured feel of the cotton fabric treated with cuberroot dye.

**Table 3: Mean Rating Responses and t-test Results of Lecturers and Postgraduate Students on the Acceptability of the Organoleptic Attributes of Cuberroot Dyes on Cotton Fabric.**

Acceptability of the organoleptic attributes.	Xi	Ni	SDi	Xii	SDii	Nii	t/Cal	Df	P-value	Re-mark
Colour hue (Army green)	4.29	17	.588	3.33	.537	24	2.368	39	.023	S
Colour value	4.24	17	.437	3.92	.504	24	2.105	39	.042	S
Colour chroma	4.12	17	.332	3.92	.408	24	1.673	39	.102	NS
Texture (sight)	3.76	17	.562	3.88	.537	24	.636	39	.529	NS
Texture (feel)	4.06	17	.429	3.96	.359	24	.815	39	.420	NS
Odour/smell	3.00	17	.791	2.42	.830	24	2.261	39	.029	S
Evenness of shade	4.24	17	.562	4.04	.464	24	3.143	39	.003	S

Key Xi - Mean of Lecturers Nii - No of Postgraduate Students  
 Ni -Number of Lecturers SDii -Standard Deviation for Postgraduate Students  
 SDi- Standard Deviation for Lecturers t/cal-T calculated d/f- Degree of Freedom  
 Xii- Mean for Postgraduate Students NS -Non Significant S-Significant

The mean values on cuberroot attribute acceptability in Table 3showed that all the attributes including hue,value, chroma, texture and evenness of shade were highly accepted by the lecturers with mean scores above 4.00. Odour was the least accepted attribute for lecturers (3'00±.791) but unaccepted by Pg students (2.42±.830). For Pg students,evennessof shade was highly accepted (4.04±.464), while other attributes were averagely accepted except odour which was unaccepted (2.42 ±.830).

**Hypothesis 1;** There is no significant difference in the mean responses of Lecturers and Postgraduate students on the acceptability of the organoleptic attributes of cuberroot dye on 100 percent cotton fabric. The P-values in Table 3 showed that there were no significant differences (P>.05) in the mean ratings of the evaluators on colour chroma (p=.10), sight texture (p=.52), feel texture (p=.42). Significant differences existed (P<.05) on hue (p=.02), value (p=.04), odour (p=.02) and evenness of shade (p=.03) at .05 probability level. The null hypothesis stating that there is no significant difference in the mean ratings of lecturers and postgraduate students on the acceptability of the organoleptic attributes of the dyes on cotton fabric are therefore accepted inthree instances for chroma, sight andfeel textures but rejected for hue ,value, odour ,and evenness of shade at 0.05 level of significance.

**Discussion**

The phytochemical qualitative and quantitative analysis of cuberroot leaves revealed the presence oflutein (.007±.0014), tannin (.13±.0014), polyphenol (0021±.0028), flavonoid (5.16±.014) and carotenoid (2.23±.02),percentages per 100g . This finding Shows that cuberroot leaves are rich in these components and supports the finding by Bassey,Bakare, Osinubu & Oremosu (2012), who confirmed in the presence of tannins and flavonoids in their phytochemical screening of cuberroot bark in a study on 'Staining properties of dye from Lonchocarpus cyanescens extract on histomorphology of the testes of sprague dawley rats . While Bassey et al study could not carry out quantitative analysis of the phytochemical constituents of cuberroot leaves or bark, the present study covered this gapFinding revealed that flavonoid was the highest phytochemical of all that were determined followed by carote-

noid. Tannin level was higher than polyphenol and lutein had the least phytochemical content in cuberoot leaf. These components are very essential for colour formation and organoleptic attributes of plant including cuberoot plant. Tannins are the most important ingredients which are necessary for dyeing with natural dyes. (Win & Swe, 2011).

The study also identified seven cuberoot organoleptic attributes. Colour hue (army green) was warm, of light value, smooth and soft textured on 100 percent cotton fabric. Odour was offensive but the shade was even. The phytochemical make up of cuberoot leaves including lutein, tannin, polyphenols, flavonoids and carotenoid contents of the plant contributed to the organoleptic properties of the dye. However, contrary to the blue hue developed by Basseyet al (2012), the present study identified army green hue. The difference in hue may be attributed to environmental factors, plant variety, mordant or dye fixatives or other chemical compounds used (Jansen & Cardon, 2005; Naomi, 2010). This study also revealed that all the organoleptic attributes minus odour of cuberoot dye were accepted by both categories of evaluators. Spenser (2010), Chenghaiah, Rao, Kumar, Alagusundaram & Chetty (2010), Ashis and Agarwal (2009), among others, have written extensively on the aesthetic and other benefits of natural dyes. The highest accepted attribute was hue (army green), was described as being warm followed by evenness of shade. This finding agrees with Jansen and Cardon (2005), that natural dyes give interesting colors that appeal to people aesthetically.

The null hypothesis stating that there are no significant differences in the mean ratings of lecturers and postgraduate students on the ac-

ceptability of the organoleptic attributes of the dyes on cotton fabric are therefore accepted in three instances for chroma, sight and feel textures but rejected for hue, value, odour, and evenness of shade at 0.05 level of significance.

### Conclusion

This study determined the phytochemical constituents of cuberoot leaves and the organoleptic attributes and acceptability of cuberoot dye on 100 percent cotton fabric. Finding revealed the following constituents; lutein ( $.007 \pm .0014$ ), tannin ( $.129 \pm .0014$ ), polyphenol ( $.0021 \pm .0028$ ), flavonoid ( $5.16 \pm .014$ ), carotenoid ( $2.23 \pm .028$ ), per 100g of cuberoot leaves. Seven cuberoot dye organoleptic attributes were identified including warm army green hue, fairly light and bright value and chroma, fairly smooth and soft texture, offensive odour and even shade of dye. Five organoleptic attributes were accepted by both categories of evaluator. There were no significant differences ( $P > 0.05$ ) in the mean rating responses of lecturers and postgraduate students on the acceptance of the organoleptic attributes of the cuberoot dye on cotton fabric in three instances but differences existed in three instances. The null hypothesis was therefore accepted in six instances but rejected in one instance at 0.05 probability level. Cuberoot dye should be explored for textile and fabric colouration for its organoleptic attributes.

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