Introduction

Natural dyes are widely used in the coloring of food, leather and wood [1]. Traditionally they were the only colorants for textile fibers like wool, silk, cotton and flax. Their use in textiles declined drastically with the introduction of synthetic dyes in 1856 [2-6]. Presently the uses of synthetic dyes is estimated at around 10,000,000 tons per annum [7-11]. The manufacture and use of this large quantity of dyes give rise to sizeable amounts of waste and unixed colorants creating serious health hazards and distressing the eco-balance of nature [12]. Currently, the heightened consciousness about environmental has had the effect in the imposition of strict eco-standards in several parts of the world in reaction to harmful reactions connected with synthetic dyes [13]. For this reason, there has been a growing demand for natural dyes in the past few decades [14]. The sources of natural dyes are diverse which include timber and food industry wastes [15]. A number of studies have been done comparing the use and performance of natural against synthetic dyes on wool and silk [12,16] and cotton [17,18]. Nevertheless, natural dyes are not readily obtainable due to few organized manufacturers and non-standardization of raw material and production process. The process of application is intricate thus further hindering the use of natural dyes.

Ethiopia has a rich tradition of using natural sources for textile coloration [19,21]. This is verified by the vibrant colors present in the ancient hand-written bibles and traditional garments worn by the monks in the monasteries. There are also publications regarding the use of certain indigenous plants as dye sources [22]. However Ethiopia has a broad range of flora that offer uncharted dye sources. Qerte (Osyris quadripartita) is found in Tigray, Oromia and Addis Ababa areas of Ethiopia. The plant is locally used for dyeing of baby holding garment (locally called Ankelba). The objective of this research is to investigate the extraction and application of dye from leaves of Qerte on cotton fabric. The parameters of dye extraction and application have been optimized. Characterisation of the extracted dye has also been carried out using aqueous extraction method. Lastly, the desirable properties of the dyed fabric were evaluated in accordance with international standards.

Materials and Methods

Materials

The leaves of the Osyris quadripartita plant, as shown in Figure 1, were collected from Bahir Dar country side. Conventionally desized, scoured and bleached plain weave cotton fabric (220 ends /dm, 180 picks/dm, 120 g/m²), obtained from Bahir Dar Textile Share Company, Ethiopia, was used for the study. Analytical reagent (AR) grade ferrous sulphate, aluminium sulphate, potassium dichromate, stannous chloride, sodium chloride and standard soap were used. Depending upon the metallic salt (mordant) used, the colour obtained on dyed fabric from the leaves of Osyris quadripartita extract differed.

Methods

Dye extraction and optimization: Aqueous extraction method was employed for extracting coloring components from Qerte leaves. The raw material (leaves) was collected and dried then ground into fine powder with the use of mortar and pestle. The
powder was soaked in distilled water at MLR of (1:100, 1:125, 1:150, 1:175, 1:200, 1:225 and 1:250) heated to a temperature of (60°C, 80°C, 100°C and 120°C) and the extraction time of 30 min, 45 min and 60 min was used. The solution was cooled, filtered and evaluated for its absorbance using a UV visible spectrophotometer (Model lambda25). The temperature, concentration and time combination that yielding maximum absorption were taken as the optimum condition of extraction of dye.

Determination of percentage of dye yield: The dye percent of yield was calculated using equation (1)

\[
\text{Percent of dye yield} = \frac{\text{mass of plant leaves} - \text{mass of dried precipitate}}{\text{mass of plant leaves}} \times 100
\]  

Dyeing of cotton fabrics: The cotton fabric samples were entered into ECO IR dyeing machine with optimum extracted dye. The dyeing was carried out for 45 minutes at 80°C with MLR of 1:10, 1:20, 1:30 and 1:40. The dyed samples were washed and dried, after the color strength was observed using color eye 3100. Higher K/S value means good absorption of the dye and this value was selected as an optimized condition.

Evaluation of dyed cotton sample: Wash fastness of the samples dyed under the optimized conditions was tested according to ISO 105-CO3 method, rubbing fastness was tested by using ISO 105-X 12 methods and Color fastness of textile material to day light was tested according to ISO 105B02 method.

Result and Discussion

Extraction of dye from Osyris quadripartite plant

The absorption result of natural dye extracted from leaves of Osyris Quadripartita using aqueous extraction method are shown in Tables 1 (Figure 2). The optimized condition was determined using UV-Visible spectroscopy (Lambda 25). From the results, the optimum conditions were 1:125 MLR extracted at a temperature of 80°C for 45 minutes (Table 2, Figure 2).

From Figure 3, the MLR of 1:125 was selected because it had a high absorption as compared the others within the wavelength range of 480-520 nm. This MLR was used in optimization of time and temperature. Figure 4 show the UV spectrophotometer of the extracted dye with the dilution of 1:21 at different temperatures.

From Figure 4, the temperature of 80°C was optimum since its absorption was greater than 60°C and 100°C but lower than at 120°C, which is high and may cause degradation of the dye. Table 3 shows the optimization of extraction time while keeping other parameters constant.

Table 3 shows different extractions carried out using 1:125 MLR, temperature of 80°C and different extraction time. Sample 3 had the highest absorption value of 0.35125, compared to other...
samples 1 and 2. For this reason, the dye extracted from 1:125 MLR dye material concentration, at temperature of 80°C for 60 minutes was considered optimum condition, since high absorbance value was obtained with these conditions. The dye extract from these conditions was used for dyeing cotton fabric samples.

Table 1: Varying the mass of plant leaves to get optimized MLR.

| Samples | Material Liquor Ratio of Leaves to Water | Mass of Plant Leaves in gm. | Mass of Dried Precipitate | % of Dye Yield | Absorption (A) (Dilute by 1:21) |
|---------|----------------------------------------|-----------------------------|---------------------------|---------------|-------------------------------|
| 1       | 1:100 (10g/l)                          | 1.5                         | 0.6                       | 60            | 2.4538                        |
| 2       | 1:125(8g/l)                            | 1.2                         | 0.54                      | 55            | 2.28472                       |
| 3       | 1:150 (6.67g/l)                        | 1                           | 0.42                      | 58            | 2.28472                       |
| 4       | 1:175 (5.714g/l)                       | 0.8757                      | 0.39                      | 55.46         | 2.27648                       |
| 5       | 1:200 (5g/l)                           | 0.75                        | 0.36                      | 52            | 1.64298                       |
| 6       | 1:225 (4.4g/l)                         | 0.67                        | 0.32                      | 52.23         | 2.04534                       |
| 7       | 1:250 (4g/l)                           | 0.6                         | 0.29                      | 51.67         | 1.64252                       |
| 8       | 1:275 (3.63g/l)                        | 0.55                        | 0.285                     | 48.15         | 1.44266                       |
| 9       | 1:300 (3.33g/l)                        | 0.5                         | 0.28                      | 44            | 1.27614                       |

Table 2: Selection of MLR at λ max 486nm.

| Material liquor ratio of leaves to water | Absorption (A) (dilute by 1:21) |
|----------------------------------------|-------------------------------|
| 0.1111111111                           | 0.24469                       |
| 0.1284722222                           | 0.31408                       |
| 0.145033333                           | 0.24333                       |
| 0.163194444                           | 0.17384                       |
| 0.180555556                           | 0.19081                       |
| 0.197916667                           | 0.17448                       |
| 0.215277778                           | 0.16346                       |

Table 3: Optimization of time.

| Sample no. | Extraction time | % of dye yield | Absorption (A) |
|------------|-----------------|----------------|---------------|
| 1          | 30 min.        | 58             | 0.31408       |
| 2          | 45 min.        | 65             | 0.34331       |
| 3          | 60 min.        | 69             | 0.35125       |

Dyeing process

The dyeing process was carried out using ECO IR dyeing machine with the optimized recipe obtained from section 3.1. Different concentrations of dyes were tested by varying the MLR on dyeing of the half bleached cotton fabric sample and their K/S value obtained (Figure 5).

As observed from Figure 5, 1:30 MLR ratio had the highest K/S value (1.3594) as compared with other MLR values. As the K/S value increased the color depth of dyed fabric also increased. For this reason, the optimum dyeing condition of 1:30 MLR, 45 minutes at a temperature of 80°C was selected.

Effects of salts

To check the effect of addition of salts on the use of the extracted dye, three samples were dyed. The first sample had no salt, second had 5g/l of salt added and third had 10g/l of salt added. The dyed fabric K/S value was evaluated and the results are shown in Figure 6.
From Figure 6, it can be seen that the K/S value (1.359) of dyed sample without addition of salt was the highest as compared to dying with addition of salt. Therefore this kind of natural dye is suitable for dyeing without addition of salt because it has good exhaustion in cotton fabric.

**Pre-mordanting methods**

Table 4 shows the effect of pre-mordanting of the natural dye on the cotton fabric. It can be seen that copper sulphate had the highest K/S value. This shows that with the application of copper sulphate as a pre-mordant, a deep color can be obtained with the extracted natural dye.

**Simultaneous mordanting methods**

Similarly, it can be clearly seen from Table 5 that copper sulphate will give higher K/S if used as a simultaneous mordant when dyeing with the extracted natural dye.

**Post mordant methods of dyeing**

For the use of post mordants, it can be seen from Table 6 that its application reduced the K/S value when compared with no addition of post mordants.

The use of mordant changes the color of the dyed fabric as shown in Table 7 and increases the costs of the dyeing process, as well as the mordants are not environmental friendly. But without addition of mordants, it is possible to obtain good shade of the dyed fabric with good water and rubbing fastness. For this reason, this dye is economical and environmentally suitable for carrying out dyeing without addition of any auxiliary or mordants.

**Table 4: Testing of the effect of pre-mordanting method.**

| Mordants used | K/S   | Color of dye solution |
|---------------|-------|----------------------|
| Aluminum sulphate | 1.3128 | Light Red            |
| Potassium dichromate | 1.4235 | Yellow               |
| Stannous chloride | 1.7002 | Dark Red             |
| Copper sulphate | 4.0059 | Yellow               |
| No mordant     | 1.3594 | Red                  |

**Table 5: Testing the effect of Meta- mordanting method.**

| Mordants used | K/S   | Color of dye solution |
|---------------|-------|----------------------|
| Aluminum sulphate | 1.567  | Red                  |
| Potassium dichromate | 1.1612 | Yellow               |
| Stannous chloride | 1.2516 | Red                  |
| Copper sulphate | 5.9986 | Yellow               |
| No mordant     | 1.3594 | Red                  |

**Table 6: Testing the effect of post mordanting method.**

| Mordants used | K/S   | Color of dye solution |
|---------------|-------|----------------------|
| Aluminum sulphate | 1.0258 | Red                  |
| Copper sulphate | 1.1169 | Red                  |
| No mordant     | 1.3594 | Red                  |

**Testing of durability**

From Table 8, the grey scale showed that this dye had good dry and wet rubbing fastness. Because to obtain above grade 4/5 indicates that during wearing, the fabric will resist any kind of rubbing from fading out of the dyed part of the fabric. Table 8 also shows that this dye does not fade during washing because after washing ten times, grade 5/5 was still obtained which indicates excellent washing fastness. The light fastness was checked by solar box and the result obtained was grade 4/5. This means the dyed cotton fabric had average resistant to light.
**Table 7: Different shades caused by using mordants.**

| Mordants            | Shades                  |
|---------------------|-------------------------|
| Potassium dichromate| ![Potassium dichromate](image1) |
| Aluminum sulphate   | ![Aluminum sulphate](image2) |
| Stannous chloride   | ![Stannous chloride](image3) |
| Direct dyeing without mordanting | ![Direct dyeing without mordanting](image4) |
| Copper sulphate     | ![Copper sulphate](image5) |

**Table 8: Testing of durability.**

| Parameters used | Results          |
|-----------------|------------------|
| No. of Wash Cycles | 1      5       10    Not Wash |
| Washing Fastness | K/S 1.5438       1.3995   1.3994  1.3594 |
| Grey scale      | 5  5  5  5      |
| Dry grey scale  | 5  5  5  5      |
| Wet grey scale  | 4/5 4/5 4/5 4/5  |

**Conclusion**

This paper deals with extraction of natural dye from *Osyris quadripartita* plant and its application in dyeing cotton fabric. This plant is widely available in east Africa and has a potential to be a source of natural dye, for dyeing textile materials. The dye from *Osyris quadripartita* plant can be well extracted with dye material concentration of 1:125 MLR (8gpl) by boiling the material at 80°C for 60 minutes using aqueous extraction methods. Dyeing with extract was optimized with 1:30 MLR and dyeing for 45 minutes at 80°C. Better color strength was achieved with copper sulphate with meta-mordant dyeing but copper sulphate is not environmentally friendly. Direct dyeing of cotton without any addition of a mordant obtained better fastness properties for this kind of natural dye.

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**Conflict of Interest**

None.

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