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# Influence of Selected Mordants and Mordanting Techniques on Eco-Friendly Dyeing of Cellulosic Fabric using Natural Flavonoid Dye Extracted from Onion Outer Scale


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***ABSTRACT***

*The study investigated the influence of selected mordants and mordanting techniques on dyeing of cellulosic fabric with natural flavonoid dye extracted from onion outer scale. It was an effort to utilize the waste material in an efficient manner which could minimize the cost of dyeing. Aqueous extraction technique was used for the dye extraction. The selected mordants used are alum, potassium dichromate, ferrous sulphate and a mango bark. The three mordanting techniques which includes, chrome mordant process, metachrome process and after-chrome process were adopted. The dye obtained was characterized by colour, melting point, pH, UV-visible and Fourier Transform Infra-red (FTIR) spectrophotometry. The dye was applied on cotton cellulose and the results obtained show multiple colours owing to different mordants used. In all cases, after-chrome process gave the best colour characteristics with the most brilliant colours on fabrics and good colour fastness with all mordants.*



***Keywords:*** *Mordants, Flavonoid, Aqueous, Spectrophotometry and Colours*


## INTRODUCTION

Textile dyeing industry at present uses excessive amount of synthetic dyes to meet the required colouration of global consumption of textiles due to cheaper prices, wider range of bright shades, and considerably improved fastness properties in comparison to natural dyes (El-Nagar *et al.*, 2005; Iqbal *et al.*, 2008). However, the production of these synthetic dyes is dependent on petrochemical source, and some of these dyes contain carcinogenic amines (Hunger, 2003). The application of such dyes causes serious health hazards and influences negatively the eco-balance of nature (Bruna and Maria, 2013; Goodarzian and Ekrami, 2010; Jothi, 2008).

Recently, dyes and colours of natural origin are gaining worldwide recognition as substitute for synthetic dyes in textile manufacture, for colouring of food products, cosmetics and pharmaceutical products (Nilani *et al.*, 2008). This renewed interest in the use of natural dyes is primarily due to increase health concern of many of the synthetic dyes because of their toxic nature and adverse environmental impacts.

Natural dyes comprise those colorants (dyes and pigments) that are obtained from animal, vegetable or mineral matter without chemical processing (Siva, 2007). They are indeed eco-friendly and exhibit better biodegradability with higher environment compatibility (Shahid *et al.*, 2013; Guesmi *et al.*, 2012).Natural dyes are mostly non-substantive and must be applied on textile material with the help of mordants, usually a metallic salt, having affinity for both colouring matter and substrate (Prabu and Bhute, 2012).Metal ions of mordants act as electron acceptors for electron donors to form co-ordination bonds with the dye molecules, making them insoluble in water (Mongkholrattanasit *et al.*, 2011). This leads to improve dye uptake and retention, which results in a greater depth of shade and colour fastness properties. Common mordants are alum, chrome, stannous chloride, copper sulphate, ferrous sulphate, and so forth (Kulkarni *et al.*, 2011).

In the present study, we have selected onion outer scale (Alliumcepa) as a source of Quercetin, a natural flavonoid dye (Mongkholrattansit *et al.*, 2012) which exhibits anti-carcinogenic, antioxidant and good dyeing properties due to its auxochrome (-OH group) and other chromogen present in the structure (Kavak and Onal, 2010; Patil*et al.*, 1999). The structure of the colouring pigment in onion outer skin (Quercetin) is shown in Figure 1.

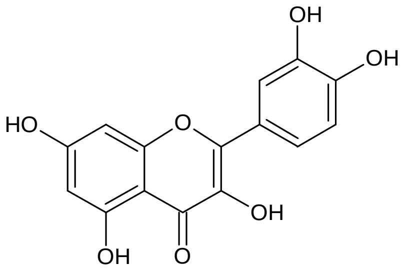


Figure 1: Structure of the colouring pigment (Quercetin) in onion outer scale.

## MATERIALS AND METHODS

### Materials

Red onion skin was gotten from Danmagaji market, Zaria; while the mineral mordants and mango bark were collected from NARICT chemical store and farm respectively. A desized, scoured and bleached plain weave cotton fabric was obtained from African Textile Manufacturers, Kano.

### Extraction

The onion skins were dried under direct sunlight until constant weight was achieved and ground into small pieces with the help of pestle and mortal. The wastages were removed using a fine strainer and the final weight was taken. The colour component was extracted from the skins in aqueous extraction process. This was achieved by extracting fixed quantity of crushed skins under pH 5 with a material to liquor ratio of 1 : 10 (weight of crushed skins in g : amount of water in mL) at boil for 60 min. In each process of extraction, the mixture was cooled down and finally the dye extracts were filtered accurately. Similar procedure was adopted for the extraction of mango bark which was used as mordant.

### Dyeing procedure

The dye extract was applied to cotton fabric with mordant and without mordant under identical conditions. Control dyeing (i.e. without mordant) was performed to assess the affinity between the dye and the fibre.

**Control dyeing (without mordant):**1g of the pre-treated cotton fabric was dyed using the onion outer skin dye. Control dyeing was carried out without mordant. Dyeing was conducted at a material to liquor ratio of 1:50 using a shade of 5% on the weight of fabric (o.w.f.). The dyeing temperature was raised by 2.50°C/min and dyeing was carried out at 85°C for 1 hour in standard laboratory dye master. After half of the dyeing time, 3 g/l sodium sulphate ($Na_2SO_4$) was added as an exhausting agent. At the end of the dyeing time, the samples were removed, washed and dried.

### Mordanting

Three mordanting techniques which include; chrome mordant process, metachrome process and after-chrome process were adopted. It is worthy of note that all the mordants were used separately in other to assess which of them has a better results.

**Chrome mordant process:** Fabrics were mordanted separately using 5% o.w.f prior to dyeing. It was carried out at $60^oC$ for 30 minutes with material to liquor ratio of 1:40.

**Metachrome proess:** In this method, the cotton fabric was treated with both the dye and the mordant simultaneously using 5% o.w.f. at $60^oC$ for 30 minutes with material to liquor ratio of 1:40.

**After-chrome process:** The fabrics in this case were dyed after which the dyed samples were taken out of the dye bath at the expiration of the dyeing time. The samples were squeezed and then treated with 5% o.w.f. of the mordants using liquor ratio of 1:40 at $60^oC$ for 30 minutes (Chandramohan *et al.,* 2012).

At the end of the dyeing process, the samples were washed with water and then dried in open air. Finally the dyed samples were soaped with 2g/L soap solution at $50^oC$ for 10 min, followed by rinsing and drying under the sun (Kumerasan*et al.,* 2011).

### UV/Visible spectrophotometry

The UV/visible spectra of the dye specimen were recorded with the Pye unicam 5625 UV/visible spectrophotometer.

### Fourier Transform Infrared spectrophotometry (FTIR)

The FTIR spectrum of the dye was measured in order to determine the functional groups present in the dye. This was done using KBr on Carry 630 FTIR Spectrophotometer.

### Melting point determination

Small quantity of the concentrated dye was placed in the capillary tube used for melting point determination. The temperature was raised gradually and observed keenly until it starts melting. This was recorded as the melting point of the dye.

**Determination of dye exhaustion**

Before dyeing, the dye solution from the dye bath was taken and the optical density was determined using UV spectrophotometer and the result obtained was taken as $OD_1$and after dyeing, the fabric was removed from the dye bath and the solution was left to cool after which part of it was taken and the optical density determined to give $OD_2$. Percentage (%) exhaustion value was calculated as follows:

$$\text{Percentage exhaustion} = \frac{O.D1 - O.D2}{O.D1} \times 100$$

where $O.D_1$ = optical density of the dye before dyeing. $O.D_2$ = optical density of the dye after dyeing.

**Evaluation of colour fastness properties**

Wash fastness of the dyed samples were tested according to ISO 105-CO3 test method. The samples were washed in standard soap solution at 60°C for 30 min., keeping material to liquor ratio at 1:50. Dry and wet rubbing fastness of the dyeings was tested according to ISO 105-X12 test method using crock meter. Light fastness was tested according to ISO 105-BO2 test method. The dyed fabrics were exposed to xenon arc lamp for 48 hours alongside the blue wool standard fabrics at standard testing conditions. The fastness was assessed by comparing the fading of the specimen with that of blue wool standard (Anon, 1990).

## RESULTS AND DISCUSSION

**Physical characteristics of the dye**

The physical characteristics of the dye are displayed in Table 1. It is seen from the table that the colour of the dye is brownish red with fine sticky texture when concentrated. The pH of the dye was found to be 5.90 which indicate acidity. The dye when concentrated was sparingly soluble in cold water and readily soluble in hot water with melting point of about 194 - 196°C.Table 1:

Table 1: Physical characteristics of onion dye extract

| **Dye plant** | **Yield (%)** | $\lambda_{max}$ | **pH** | **M.pt** | **Colour of aqueous extract** | **Solubility** | |
|---|---|---|---|---|---|---|---|
| | | | | | | Cold water | Hot water |
| Onion scale | 22.45 | 372 nm | 5.90 | 194 - 196 | Brownish red | Sparingly soluble | Readily soluble |

**Dyeing behavior of the extract**

The onion extract was found to have little substantivity for cotton fabrics when applied without mordant. The substantivity was however improved when mordants were applied in the dyeing process. However, the dye uptake was found to be better off in after-chrome process as it was observed in the shade obtained after the dyeing and mordanting process.

**Influence of mordants and mordanting techniques on dye-ability of the substrate**

Mordants play very important role in the substantivity of coloured fabrics dyed with natural dyes. The mordants used in this case gave varying shades. Better colour strength results are dependent on the metal salt used (Kamel*et al.*, 2009). The strong co-ordination tendency of iron enhances the interaction between the fibre and the dye, resulting in high dye uptake (Jothi, 2008). In contrast, aluminum salts formed weak coordination complexes with the dyes. This tends to form quite strong bonds with the dye molecule rather than with the fibre (Cotton and Wilkinson, 1972). Thus, they covered the dye and reduce its interaction with the fibre. This therefore could result in low colour strength when compared with that of ferrous sulphate mordant.

It was observed that after-chromeprocess gave rise to the highest colour strength and dye uptake in comparison with metachrome and chrome mordant process. This is seen from the deeper shade exhibited by the substrate dyed by after-chrome process. It is believed that in metachrome process, the onion dye molecules and the mordant may have formed insoluble co-ordination compounds and thus precipitated from dyeing bath, resulting in the decrease of effective dye uptake and colour strength while in the case of chrome mordant process, the mordant ions absorbed on the fibres could desorbed from the fibres and form insoluble co-ordination compounds with the onion dye molecules. This also could be the reason for the lower dye uptake and colour strength, cum, lighter shade of the dyed substrate. This is in agreement with the findings of (Kulkarni*et al.*, 2011).

**Results of dye exhaustion**

The results of dye exhaustion of onion outer scale on cotton fabrics are shown in Table 3. It was seen that cotton fabric dyed without mordant showed the least exhaustion. However, on application of mordants, the dye exhaustion improved greatly with ferrous sulphate showing the best exhaustion on cotton fabrics.

Table 3: Maximum exhaustion of onion scale dyed cotton fabrics

| Fabric dyed with onion scale | Dye Exhaustion (%) |
|---|---|
| Cotton without mordant | 48 |
| Cotton afterchromed with mango bark | 54 |
| Cotton afterchromed with alum | 65 |
| Cotton afterchromed with potassium dichromate | 80 |
| Cotton afterchromed with ferrous sulphate | 84 |

**Infra-red spectra of the dye**

The extract shows a strong signal corresponding to the aliphatic -C-H stretch around 2958 $cm^{-1}$. A wide band from -OH group was manifested in the range of 3421 $cm^{-1}$ and this may be due to the presence of phenols in the extracted dye. Absorptions also exist in the regions of 1624 $cm^{-1}$ for the C=O group which may be due to the presence of carboxylic acids. The absorptions at 1070 $cm^{-1}$ may indicate the presence of C-O group of alcohols and esters. This is in agreement with the findings of (Osabohien, 2009).

**Wash fastness**

The results of wash, rubbing and light fastness of the dyed cotton fabrics are shown in Table 4.The unmordanted dyed fabrics showed colour change rating of 3 which is fairly good. This can be attributed to affinity of the colouring component through the intermolecular H-bonding and van der Waals forces. On application of mordants, varying shades of colour were obtained and the colour change ratings were found to be within 4 to 5. A rating of 5 (excellent) was found when Ferrous sulphate was used in after-chrome process and this can be as a result of strong co-ordination tendency of iron which enhances interaction between the dye and the fibre thus, resulting in the formation of co-ordination complex which makes it difficult for the dye to come out of the fibre during washing, hence, the high wash fastness rating obtained (Jothi, 2008). Using potassium dichromate and alum mordant, the ratings were found to be 4-5 and 4 respectively for the change in colour of the dyeings and after-chrome process still gave a better results than other mordanting techniques adopted. It can be said that the overall ratings of colour change were good and this can be as a result of the influence of the rate of diffusion of dyemolecules and state of dyes inside the fiber. The tendency of the dye to aggregate inside the fiber which results in increased molecular size brings about good wash fastness results (Uddin, 2014). On the other hand, the colour staining ratings were found to be from 4 and 4-5 for all the dyed fabrics which is an indication of slight staining of the adjacent fabric and a good colour fastness of the dyeings.

In the case of the mango bark which is a natural mordant, the result of the change in colour was found to be good (3-4 and 4) with after-chrome process having the best result.

**Light fastness**

The result of the light fastness as displayed on Table 4 show that natural dyes generally have poor light fastness (Samanta and Agarwa, 2009). For unmordanted cotton fabrics, the light fastness rating was found to be 1. Using mordants, there was an improvement in the light fastness rating. In the case of metallic mordant, ferrous sulphate after-chromed samples had the highest light fastness which is 4 and this can be attributed to formation of complex with transition metals which protected the chromophore from photolytic degradation. The photons sorbed by the chromophoric group dissipated their energy by resonating within the six-membered ring formed, thus, protecting the dyes. Ferrous sulfate can bind with more dye molecules than alum or chromium. During exposure to light, the fabrics mordanted with ferrous sulfate, alum, or chromium may have the same number of dye molecules destroyed. But as the fabrics mordanted with ferrous sulfate had deeper shades due to bonding with more number of dye molecules, it seemed to fade less compared to the fabric mordanted with alum or chromium (Uddin, 2014).

In the case of the mango bark mordant, the light fastness properties were very poor which is common with natural dyes and mordants (Samanta and Agarwa, 2009).

**Rubbing Fastness**

The results of the rubbing fastness are displayed in Table 4. From the Table, it is seen that good rub fastness was exhibited by the fibers dyed using the dye extracted from the onion outer scale. Complexing the fiber with mordant has the effect of insolubilizing the dye, making it colour fast.

Table 4: Fastness properties of dyed samples

| **Mordants** | **Method of application** | **Wash fastness** | | **Rubbing fastness** | | | | **Light fastness** |
|---|---|---|---|---|---|---|---|---|
| | | | | Dry | | Wet | | |
| | | CC | CS | CC | CS | CC | CS | |
| Alum | Chrome mordant process | 4 | 3-4 | 5 | 4-5 | 4-5 | 4 | 2-3 |
| | Metachrome process | 4 | 3-4 | 5 | 4-5 | 4-5 | 4 | 2-3 |
| | Afterchrome process | 4-5 | 4 | 5 | 4-5 | 5 | 5 | 3 |
| Ferrous Sulphate | Chrome mordant process | 4-5 | 4 | 5 | 4-5 | 4-5 | 4 | 3 |
| | Metachrome process | 4-5 | 4 | 4-5 | 4 | 4-5 | 4 | 3-4 |
| | Afterchrome process | 5 | 4-5 | 5 | 4-5 | 4-5 | 4 | 4 |
| Potassium dichromate | Chrome mordant process | 4-5 | 4 | 5 | 4-5 | 4-5 | 3-4 | 3-4 |
| | Metachrome process | 4-5 | 4 | 5 | 4-5 | 4-5 | 4 | 3 |
| | Afterchrome process | 4-5 | 4 | 5 | 4-5 | 4-5 | 4 | 3-4 |
| Mango bark | Chrome mordant process | 3- 4 | 3 | 4 | 3-4 | 4 | 3-4 | 2 |
| | Metachrome process | 3 | 2-3 | 4 | 3-4 | 3-4 | 3 | 2 |
| | Afterchrome process | 4 | 3-4 | 4 | 3-4 | 3-4 | 3 | 3 |
| | No mordant | 3 | 2-3 | 4 | 3-4 | 3-4 | 3 | 1 |

## CONCLUSION

The result obtained from this research shows the potentials of using a natural flavonoid compound which in this case is onion outer scale as colourant for cellulosic substrates. The wash, light and rubbing fastness properties on cellulose when applied without any mordant were found to be moderate. However, the use of mordants in textile dyeings was found to be essential as there was tremendous improvement in shades and fastness properties of the dyeings. From all the results obtained, dyeings obtained by after-chrome process with ferrous sulphate gave the best results.

Although, the metallic salt mordant proved to be better but mango bark mordant which is natural and eco-friendly has also proved to be an ideal mordant for the dyeing of cellulosic fabrics owing to the acceptable fastness results obtained from the dyeings.

Finally, the process is promising as it has proved the efficacy of using onion outer skin as a dye for cellulosic substrates e.g. cotton fabric. It can therefore be popularized among the local natural dyers in order to obtain an eco-friendly product with value added properties.

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