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Dyeing of cellulose-based fabrics using dyes extracted from plants.

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Abstract

Unbleached and bleached woven cotton fabrics were scoured with non-bleach washing powder. The first set of fabrics were mordanted once in a mixture of alum [1:120 (w/v)] and vinegar [1:100 (v/v)] while the second set was mordanted thrice (tri- mordanted); first with alum [1:120 (w/v)], then with tannin [1:500 (w/v)] and finally with alum in water. Cooking overnight soaked inflorescences of *Tagetes minuta* in water in the ratio 1:25 (w/v) and straining, provided the muchneeded dye-bath. Dyeing was accomplished by soaking the mordanted fabrics in the dye-bath and simmering for 1 hour at various temperatures viz: room temperature, ranges of 50—60 ^{o}C and 80—90 ^{o}C. Tests performed confirmed the dye produced as a reactive dye.

Cotton fabrics that were tri-mordanted produced brighter golden yellow shades vis á vis those that were singly mordanted in alum and vinegar. The dyeing temperature range of 80—90 ^{o}C produced uniform dyeing of fabric articles hence the optimal dyeing temperature. A khaki (olive green) shade was produced on fabric articles when 38^{o} Bé sodium hydroxide solution (8 mL/L) was added to the dye bath. The resultant golden yellow shade obtained from *Tagetes minuta* inflorescences is a big achievement given that it is a primary colour which can be used to generate other colours for dyeing.

Keywords: *Tagetes minuta*, inflorescence dye-bath, golden yellow shade, khaki shade.

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Introduction

Dyes extracted from plant materials offer alternatives to petrochemical ones that hitherto, have posed serious issues relating to health and the environment (Permalink, 2006). Their suitability is based on the fact that they are natural hence environmentally friendly. The advocacy for their use is in line with goal number 7 of the Millennium Development Goals (UNDP, Web article).

Some of the widely used colours for dyeing include indigo that dyes fabrics blue and is obtained from plants of the genus *Indigofera* (Web article, Indigo). Shades of yellow, gold, rust and green are known to be extracted from *Rhododendron* leaves when used with different mordants (Web article, *Rhododendron*—Dye plants). Natural red dyes have been produced from madder plants (Web article, Madder dye plant). The woad plant (*Isatis tinctoria*), produces blue dyes principally used in Europe (Web article, Woad). *Eucalyptus* spp. and *Allium cepa* skins have been used to produce yellows (Teresinha, Web article).

It has been intimated that natural dyes are vastly superior to synthetic dyes. They age well and develop a patina[2] and an abrash[3] as the older textiles are exposed to sunlight and normal use. The patina is a mellowing of the colours into an eye-pleasing sheen and the abrash is the slightly uneven hues that emerge as different dye lots, even of the same colour, fade at different rates as they age. All of these factors combine in making natural dyes the ideal choice for use as organic fabric dyes (Thurston 1972; Murphy, 2006).

Tagetes minuta is an erect annual herb that has found numerous uses such as a beverage, a medicinal tea, and a condiment[4] since pre-contact times (Rees, 1817). It is commercially grown and harvested for its essential oils (Leung, 1980), as well as secondary metabolites used for pesticides purposes (Steiner, 1941). The herb is finding more uses including production of organic fabric dyes.

Materials and Methods

Tagetes minuta (Mexican Marigold) plant materials were collected from gardens and farms around the Rift Valley Textile (Rivatex) milling factory. Sampling sites included maize farms in Kapseret, and Kipkaren areas as well as gardens close by the milling factory. All measurements were carried out at the Rivatex chemical laboratory, Moi University, Eldoret. Detailed procedures are summarized belew.

Dyeing fabric

Cotton (cellulose-based) fabric articles 002A and 053A in both bleached and non- bleached (loom state or grey cloth) conditions were obtained from the processing department within Rivatex for experimental dyeing. Article 002A at Rivatex is constructed as follows: warp/weft count: 43/43 nm; 13 picks/cm and 25 ends/cm while article 053A has its construction as warp/weft count 18/18 nm; 16 picks/cm and 23 ends/cm.

Fabric Scouring

The cotton fabric articles, cut into small size pieces whose total weight was 100 grams (g), were first washed in hot water for about 10 minutes using a non-bleach washing powder. A stainless steel pot was filled with 5 litres (L) of water in which 35 g soda ash (about ¼ cup) were carefully added. The wet fabric articles were then put into the water and swished around using a stirring rod. The glass rod was left inside the pot, to prop the lid slightly open to prevent the liquid from boiling over. The water was eventually brought to the boil.

The heat was adjusted to low boil/hard simmer and allow the fibre to boil half covered for 2 hours (h). The fabric articles were stirred every 15 minutes to ensure adequate scouring. The saucepan was removed from the heat source after 2 h and the fabric articles allowed to cool down until they could safely be removed from the water. The fabric articles were finally rinsed in clean cold water (Web article, Mordanting Cotton).

Fabric Mordanting

Mordanting was done in two sets:

a) Single mordanting in an Alum/Vinegar bath

An alum [hydrated aluminum potassium sulphate—$KAl(SO_4)_2.12H_2O$] mordant bath was prepared for this procedure. A solution containing 25 g of alum, 30 millilitres (mL) of vinegar (3% acetic acid) in 3 L of water was prepared. The solution was brought to boil and then let to cool. The fabric articles were immersed into the cool mordant bath whose temperature was eventually brought to the range of 80—90 ^oC by heating. This was simmered for 1 h, removed and then let to cool (Storey, 1978; Allfiberarts[a]).

b) Tri-mordanting in three successive mordant baths of Alum, Tannin and Alum

Bath 1 (Alum mordant):

The dye pot was filled with 3 L water and nearly brought to boil. Alum (25 g) were first dissolved in a small container with boiling water and slowly added to the pot and stirred well. Soda ash [Na_2CO_3] (6 g) were then weighed out and added slowly and carefully to the water in the pot. The cleaned and scoured fabric articles were then added to the pot, heated to simmering point and simmered for 1 h. The fabric articles were stirred every fifteen minutes during simmering and eventually left in the pot for 24 h. The fabric article pieces were wringed well, dried in the open air and left to age for a week. The pieces were washed well before treating them in the second bath.

Bath 2 (Tannin mordant):

The dye pot was filled with 3 L of hot water. Tannic acid (6 g) were dissolved in a small container with boiling water and added to the pot to which the cotton fabric articles were added. Simmering was done for 1 h and left for 24 h. The cotton pieces were then wringed well, dried and left to age for a week. The pieces were washed well before treating them in the third bath.

Bath 3 (Second Alum Bath):

Alum mordanting was repeated as described in Bath 1 (Web article, Mordanting Cotton).

Dye extraction from *Tagetes minuta*

Inflorescences, borne on the apical parts of the plants, were carefully removed. Two hundred grams of the inflorescences were weighed and immersed in 5 L of water. These were brought to boil and simmer for 2½ h. The dye bath obtained was let to cool prior to straining the liquid (Web article, Allfiberarts[b]).

Fabric dyeing

The mordanted fabrics were separately added to the dye-bath so obtained. The dye- bath was returned to heat and simmer for 1 h at various temperatures viz: room temperature, ranges of 50—60 ^oC and 80— 90 ^oC then removed from the heat source and let to cool. The fabrics were later rinsed in warm water—ideally water of temperatures similar to the dyebath—to avoid shocking the fabrics and causing felting to occur (Web article, Allfiberarts[c]).

Testing dyed fabric

a) Fastness to washing

A soap solution made up of 5 g of soap and 2 g of anhydrous sodium carbonate (Na_2CO_3) in 1 L of distilled water in a beaker free from fluorescent brightening agents was prepared and heated to 60 °C. Specimens of dyed cotton fabrics measuring 10 cm x 4 cm were cut out. They were separately placed between two pieces of undyed cloths of plain weave free from size and other finishing chemicals of cotton measuring 5 cm x 4 cm and each of the now three pieces held together by stitching round the edges, leaving 5 cm x 4 cm exposed. The specimens were then placed in a beaker for 30 minutes with constant stirring while maintaining the temperature at 60 °C by steady heating. They were removed and rinsed in cold water then in cold water running from a tap for 10 minutes. The specimens were then squeezed and dried in air not exceeding 60° C (Trotman, 1970).

b) Bleaching test on dyed fabrics

Pieces of dyed fabric were immersed into a 3.85% v/v solution of sodium hypochlorite (NaOCl) and left to stand for a period of 1 h.

c) Testing for type of dye produced by *Tagetes minuta*

i. Preliminary dye testing using 5% w/v NaOH

One half of a litre of 5% w/v caustic soda (NaOH) solution was prepared and put in a saucepan. Pieces of dyed fabrics were added, then heated and brought to boil for a period of 2 minutes. Caution was taken to avoid inhaling the choking NaOH fumes (Trotman, 1970).

ii. Stripping or lightening of dye

A solution containing 15ml/l of 38 °Bé caustic soda, 5g/L of Hydrosulphite Conc. BASF, and 2g/L of Albigen A was prepared. The solution was prepared just before use by adding the alkali to the bath first, followed by strewing in the hydrosulphite powder in small portions with stirring. A piece of the dyed cellulose fabric was added to the solution and the temperature raised by heating and maintaining the temperature at a range of 60—80 °C for a period not exceeding 1 h. The bath was let to cool and fabric articles rinsed intensively in cold water (Technical Leaflet—Hydrosulphite Conc. BASF, 1986).

Results

Dye extraction from *Tagetes minuta*

The dye-bath obtained from the inflorescences had a dark-brown appearance as illustrated in Figure 1.

Figure 1:

Figure 1: Dye-bath preparation from *Tagetes minuta* floret parts. Right: floret dye bath.

Fabric mordanting and dyeing

Conspicuous change of colour on fabrics was observed. The fabric articles took up a golden yellow colour early in the dying process. Fabric articles tri-mordanted and dyed at room temperature and pressure (rtp) and those at the range of 50—60 °C showed inconsistency in colour absorption especially at rtp (Figures 2 and 3).

Figure 2a):

Figure 2b):

Figure 2c):

Figure 2d):

Figure 3a):

Figure 3b):

Figure 3c):

Figure 3d):

Figure 2a): Tri-mordanted cotton fabric (bleached) article 002 dyed at rtp. Control on the left.
Figure 2b): Tri-mordanted cotton fabric (un-bleached) article 002 dyed at rtp. Control on the left.
Figure 2c): Tri-mordanted cotton fabric (bleached) article 053 dyed at rtp. Control on the right.
Figure 2d): Tri-mordanted cotton fabric (un-bleached) article 053 dyed at rtp. Control on the left.

Figure 3a): Tri-mordanted cotton fabric (bleached) article 002 dyed at 50—60 ^{o}C. Control on the left.
Figure 3b): Tri-mordanted cotton fabric (un-bleached) article 002 dyed at 50—60 ^{o}C. Control on the left.
Figure 3c): Tri-mordanted cotton fabric (bleached) article 053 dyed at 50—60 ^{o}C. Control on the left.
Figure 3d): Tri-mordanted cotton fabric (un-bleached) article 053 dyed at 50—60 ^{o}C. Control on the left.

However, all articles dyed in the temperature range of 80—90 ^{o}C exhibited uniform absorption of colour (Figures 4 and 5).


Figure 4a):


Figure 4b):


Figure 4c):


Figure 4d):

Figure 5a):

Figure 5b):

Figure 5c):

Figure 5d):

Figure 4a): Tri-mordanted cotton fabric (bleached) article 002 dyed at 80—90 ^{o}C. Control on the right.

Figure 4b): Tri-mordanted cotton fabric (un-bleached) article 002 dyed at 80—90 ^{o}C.. Control on the left.

Figure 4c): Tri-mordanted cotton fabric (bleached) article 053 dyed at 80—90 ^{o}C. Control on the left.

Figure 4d): Tri-mordanted cotton fabric (un-bleached) article 053 dyed at 80—90 ^{o}C. Control on the left.

Figure 5a): Single-mordanted cotton fabric (bleached) article 002 dyed at 80—90 ^{o}C. Control on the right.

Figure 5b): Single-mordanted cotton fabric (un-bleached) article 002 dyed at 80—90 ^{o}C. Control on the left.

Figure 5c): Single-mordanted cotton fabric (bleached) article 053 dyed at 80—90 ^{o}C. Control on the right

Figure 5d): Single-mordanted cotton fabric (un-bleached) article 053 dyed at 80—90 ^{o}C. Control on the left

Single mordanted articles were dyed in the temperature range of 80—90 °C (Figure 5).

Figure 5a):

Figure 5b):

Figure 5c):

Figure 5d):

Figure 5a): Single-mordanted cotton fabric (bleached) article 002 dyed at 80—90 °C. Control on the right.
Figure 5b): Single-mordanted cotton fabric (un-bleached) article 002 dyed at 80—90 °C. Control on the left.
Figure 5c): Single-mordanted cotton fabric (bleached) article 053 dyed at 80—90 °C. Control on the right
Figure 5d): Single-mordanted cotton fabric (un-bleached) article 053 dyed at 80—90 °C. Control on the left

There seemed to be no clear difference except for brighter colours for the tri-mordanted 002 articles compared with the single mordanted (Figures 4a & b, 5a & b).

Figure 4a):

Figure 4b):

Figure 5a):

Figure 5b):

However, the single mordanted 053 bleached article exhibited a brighter shade compared with the tri-mordanted one (Figures 4c and 5c).

Figure 4c):

Figure 5c):

This could be attributed to other prevailing factors/conditions during the dyeing process that brought about the differences

Visual observation showed that un-bleached fabric articles had a higher intensity of colour as compared to the bleached ones especially for article 002 whereas brightness was greater in the bleached fabrics for both articles 002 and 053 (Figures 2—5).

The characteristic *Tagetes minuta* odour which could have repulsive effects to different people was totally absent after dyeing and drying the fabric articles. This is a very positive attribute indicating that factors contributing to the odour emanating from the plant prior to dye extraction are absent in the dye produced.

Addition of 38^{o} Bé caustic soda (8 ml/l) to the dye bath and dyeing produced a khaki (olive green) shade (Figure 6)

Figure 6a):

Figure 6b):

Figure 6a): Bleached cotton fabric article 002 dyed at 80—90 ^{o}C. Control on the left.
Figure 6b): Un-bleached cotton fabric article 002 dyed at 80—90 ^{o}C. Control on left.

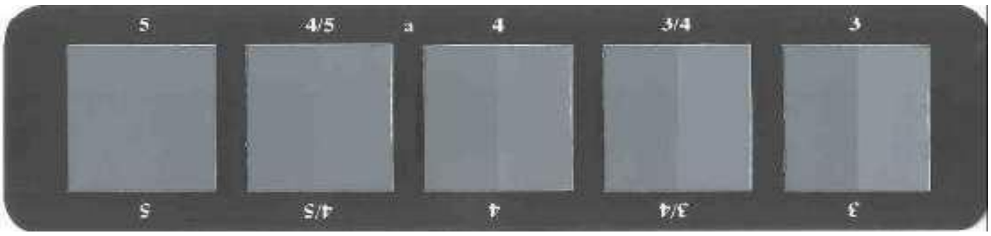
Physico-chemical testing of dyed fabrics

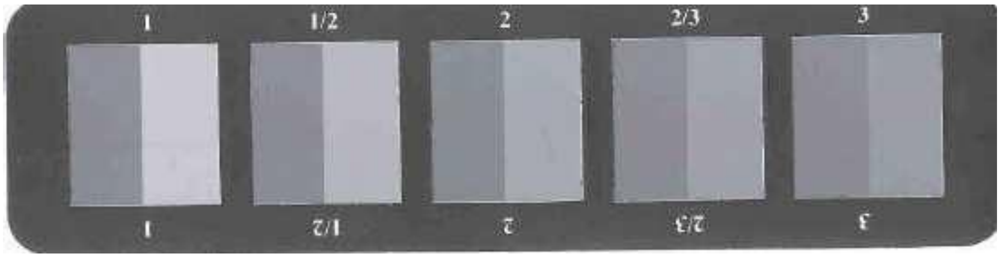
a) Fastness to washing test

The fabric articles showed no fading in the shade acquired during the dyeing process after carrying out the fastness to washing test. Fastness test carried out on the fabric, according to the McLaren (*J.S.D.C.*, 1952, **68,** 203) gave grade 5 (Appendix 1).

Appendix 1: Geometric scales of testing dyed material for colour fastness

Grade	Contrast before & after test	I.S.O. recommended scale
5	0 N.B.S. units	0 N.B.S. units
4	*n* N.B.S. units	1.5 N.B.S. units
3	2*n* N.B.S. units	3 N.B.S. units
2	4*n* N.B.S. units	6 N.B.S. units
1	8*n* N.B.S. units	12 N.B.S. units





b) Bleaching test on dyed fabrics

There was little effect of NaOCl on dyed fabric (Figure 7),

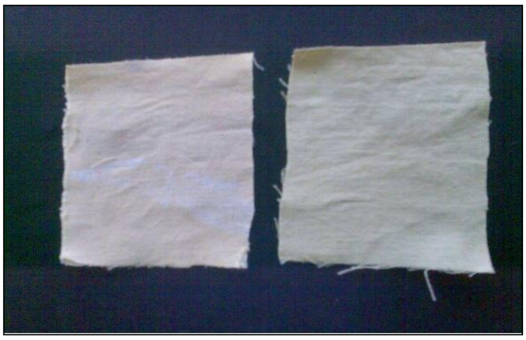


Figure 7:

Figure 7: A Fabric piece that has undergone bleaching. Control on the right

when pieces were immersed in concentrated bleaching solution for 1 hour except for the brightening effect. Thus the dye is not easily bleached by ordinary bleaches.

c) **Testing for dye obtained from *Tagetes minuta***

i. Preliminary dye testing using 5% w/v NaOH

Not so much of the dye was observed in the alkali solution (Figure 8a)

Figure 8a:

Figure 8a): Resulting faint yellow alkali solution after dyed fabric for 2 minutes. Unused alkali solution on the right. Though the fabric showed considerable removal of dye (Figure 8b).

Figure 8b:

Figure 8b): Resultant fabric after boiling with the dye removed. Control on the left

The inference is that the dye was a reactive dye.

ii. Stripping or lightening of dye

There was a considerable stripping of dye from the dyed fabrics. The more the stripping time, the more the amount of dye removed (Figure 9).

Figure 9a:

Figure 9b:

Figure 9a): Fabric stripped for 30 minutes. Note the un-stripped patches on the treated fabric. Control on the left.
Figure 9b): Fabric stripped for 60 minutes. Control on the left.

The stripping of the dye using this test confirmed that the dye was a reactive dye.

Discussion

The results showed that plants are capable of producing viable dyes that can be used for dyeing cellulose-based fabrics. *Tagetes minuta* produces reactive yellow dyes as evidenced in this study (Figures 2a—5d)

Figure 2a):

Figure 5d):

and supported by standard testing procedures of Trotman, (1970). However, different colours could be produced on the fabrics following the type of mordant or combination of mordants used in fixing the colours onto the fabrics (Web article, Allfibrearts[c]). This is well demonstrated in this study when the alum mordants produced a yellow shade as opposed to the khaki (olive green) shade that was produced when the fabric articles were dyed in the dye-bath that had been mixed with 38^{o} Bé caustic soda (Figure 6).

Figure 6a):

Figure 6b):

Figure 6a): Bleached cotton fabric article 002 dyed at 80—90 ^{o}C. Control on the left.
Figure 6b): Un-bleached cotton fabric article 002 dyed at 80—90 ^{o}C. Control on left.

The study revealed that high temperatures are optimal for dyeing. This is well demonstrated in the results obtained at the various temperature ranges (Figures 2a—5d).

Figure 2a):

Figure 5d):

There is a possibility that higher temperatures with a component of pressure could give better results in terms of brightness and fastness. This requires further investigation.
The alum tri-mordanted fabric articles gave brighter colours of yellow for article 002 compared to the singly alum mordanted articles. This suggests that the tannin component in tri-mordanting brings about a brightening effect. Moreover, longer periods of mordanting as was done in tri-mordanting could have added to the brightness effect.

The dyed fabrics were observed not to bleach by sodium hypochlorite (i.e., 3.85%v/v). This is an important attribute and it could be quite appropriate to dye protective attires with *Tagetes minuta* dyes.
The study also revealed that the characteristic pungent smell produced by the plant was totally absent from the dyed fabric articles. It is here concluded that the factors contributing to that foul smell are absent in the active particles of the dye.
Findings of this study points to the fact that dye extraction from *Tagetes minuta* plant is possible. The properties of the dye obtained compare favourably with commercial reactive dyes which are petro-chemically derived. However, the dye produced in this case is natural and environmentally friendly. Of great importance is the fact that the dye produced from *Tagetes minuta* is a primary colour which together with the other two primary colours of red and blue could yield all the other desired colours for dyeing purposes. This study is therefore a milestone in the dyeing industry as the world seeks to protect the environment from synthetic dyes. Moreover, raw materials from *Tagetes minuta* should be easy to avail given that it can easily be propagated. It turns out to be a convenient renewable resource and will authoritatively serve the purpose

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