Dyeing wool and cotton fibres with acidic extract of *Hibiscus rosa sinensis* flower

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**ABSTRACT**

The focus of this work is to extract a natural dye for colouring camel wool as a substitute for synthetic dyes used in the Sadu House of Kuwait. Their target is to keep the tradition of tent and rug production natural in all its manifestations. Therefore, our task was to find an abundant source that provides a colour preferably red to purple. *Hibiscus rosa sinensis* (HRS) is an abundantly available plant in Kuwait that was explored for extraction of the red dye to colour camel wool permanently. The powdered petals of red flowers of HRS was extracted with 5% acetic acid which yielded a deep red colour that showed a great potential for woollen fibre dyeing. The use of mordants like alum and some metal salts manifested a wide range of fixed colours which intensified at 85 °C. The colours produced had excellent fastness and was accepted by the Sadu House.

**Abbreviations:** GAA: Glacial acetic acid; AA: Acetic acid; HRS: *Hibiscus rosa sinensis*

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1. Introduction

The red flower of *Hibiscus rosa sinensis* (HRS) also known as China rose belongs to Hibisceae tribe of family Malvaceae. It is an evergreen shrub and is abundantly available throughout the Middle East and eastern Asia. Although HRS is not a native plant of Kuwait, it was introduced in 1970s and its flowers are available in Kuwait all year round (Jadhav et al. 2009). In this work the scarlet red coloured flower of HRS was used. Internationally, the flower is used for a wide range of applications including hair care, herbal shampoo, antifungal cream as well as an emollient agent. The flowers are rich in anthocyanin (Jadhav et al. 2009 and Ruban & Gajalakshmi 2012). Anthocyanins are natural colorants with a wide range of colours. As a dye, orange, pink, red, violet and blue can be obtained by either changing the pH of the extracts or fixing the pH of the extract and changing the mordants. The resultant colour of anthocyanin and the mordant complexes can be intensified by heat application.

Anthocyanins as food element are safe for human consumption because of their therapeutic values as described by Jadhav et al. (2009) and Ruban and Gajalakshmi (2012). These uses indicate that the colour as free or bound to a mordant such as stannous chloride (SnCl₂) can be added to food as an additive, where it serves as natural safe colour retention agent and antioxidant with no side effects (Wikipedia 2015). Sharma et al. (2004) reported the remarkable skin anticancer property of HRS flower extract, and was supported by Abdel Ghaffar and El-Elaimy (2012) who had shown that the (HRS) ethanol/water extract, scavenge free radicals and inhibit lipid peroxidation that may lead to carcinogenesis. A 90% ethanol extract of HRS by Sidram et al. (2011) functioned as an anti-solar agent by absorbing ultraviolet radiation. Despite the great potential of anthocyanins used in food, pharmaceutical and cosmetic industries, its application for textile dyeing was limited simply because of either the lack of affinity to the fabrics or low wash fastness. In the current study, the extraction of HRS with methanol resulted in a dark brown colour, yet extraction with ethanol yielded very light red colour compared to deep red colour obtained with 5% acetic acid (AA). The cold 5% AA and ethanol extracts of HRS were tested for visible and UV light absorption. These properties indicated that 5% AA extract could be an ideal dye for individual and industrial use. The properties of the above dye were investigated on demand of Kuwait Sadu House (a society that protect the interests of Bedouins and their ethnic heritage).

The process provided for dyeing the fibres was natural and risk free. The extraction of the colour was achieved by an environmental friendly process, as reported by Gasmelseed et al. (1989), yielding a permanent dark red/purple colour that neither changed nor faded upon exposure to heat or light.

2. Results and discussion

Extraction of anthocyanins from Hibiscus was preceded with different methods for different applications. Vankar and Shukla (2011) extracted the anthocyanin from HRS using 4% citric acid in methanol to give a better yield and used it to dye cotton and silk. The Sadu House requested extraction of a preferably red colour from a natural source for colouring the fibres used for weaving rug and tents. They insisted on a dyeing process that conformed to Bedouin traditions that is natural and safe for human health. Bedouins commonly use vinegar in their diet; therefore a method was optimised for the extraction of HRS with a dilute solution of AA.
When powdered HRS was extracted once with glacial AA (GAA) (step 1a, Figure 1) and secondly with 5% AA (step 1b, Figure 1). Each experiment resulted in deep red-coloured solutions. The extraction with 5% AA produced a better yield than the GAA extraction. The extracts from steps 1a and 1b were tested for their potential to dye camel wool and cotton fibres, which were mordanted/non, at room temperature and 85 °C.

The 5% AA was the solvent of choice based on Table 1 as it produced the same colour intensity as GAA with a higher yield and was less corrosive.

The scoured camel wool and cotton fibre samples were treated with various mordants. The dyeing process was carried out with 5% AA extract followed by washing with tap water.

![Diagram](image)

**Figure 1.** Scheme for extraction of (HRS).

| Colorant                          | Mordant          | Dyeing at 85 °C | Dyeing at room temperature |
|----------------------------------|------------------|-----------------|---------------------------|
| GAA extract from step 1a (Figure 1) | Non mordanted    | Cotton          | Wool                      |
| 5% AA extract from step 1b (Figure 1) | Non mordanted    | Cotton          | Wool                      |

**Table 1.** Dyeing the stannous chloride mordanted/non, wool and cotton fibres with GAA and 5% AA extracts from step 1a & 1b from Figure 1, at room temperature/85 °C.
The different shades of colours (depending on the mordant used) obtained in this process are shown in Table 2. Dyeing with 5% AA extract was accomplished initially without premordanting the fibres. A dark peach colour was obtained after heating wool at 85 °C. The elevated temperature of dyeing always intensified the colour in mordanted and non mordanted fibres. The intensity of anthocyanin colours, obtained with mordanted fibres especially the wool, increased when heated with 5%AA extract at 85 °C (Table 2). That increase with heat might be due to the formation of a stable anthocyanin–stannous complex (A–S). Its stability was referred by Vankar and Shukla (2011) to the interaction of the OH groups of cellulose in cotton or the NH2 groups in wool fibre with (A–S complex). Also they reported molecular transformation of anthocyanin at variable pH, leading to changes in colour to red (pH 2–3), purple (pH <7) and blue (pH <8).

The A–S complex extends the shelf life of the dyed product because of the free radical scavenging properties (Abdel Ghaffar & El Elaimy 2012). It also functioned as an antibiotic for a range of micro-organisms, as reported by Ruban and Gajalakshmi (2012). Dyeing the fibres of woollen rugs with (A–S) complex imparts protecting characteristics against microbial

**Table 2.** Colours obtained with cotton and camel wool, treated with different mordants and impregnated with 5% AA (HRS) flower extract under heat (85°C) or room temperature.

| Mordant                | Dyeing at 85 °C Cotton | Dyeing at 85 °C Wool | Dyeing at room temperature Cotton | Dyeing at room temperature Wool |
|------------------------|------------------------|----------------------|-----------------------------------|---------------------------------|
| Non mordanted          |                        |                      |                                   |                                 |
| Stannous chloride      |                        |                      |                                   |                                 |
| Alum                   |                        |                      |                                   |                                 |
| Cobalt chloride        |                        |                      |                                   |                                 |
| Ferric chloride        |                        |                      |                                   |                                 |
| Potassium dichromate   |                        |                      |                                   |                                 |
| Magnesium sulphate     |                        |                      |                                   |                                 |
| Nickel chloride        |                        |                      |                                   |                                 |
| Tartaric acid          |                        |                      |                                   |                                 |
| Chrome alum            |                        |                      |                                   |                                 |
damage. Anthocyanins being powerful antioxidant may be considered as an anti-solar agents absorbing harmful UV radiation from the sun leading to carcinogenesis (Sharma et al. 2004).

Stannous chloride as a mordant resulted in a deep purple colour on the fibre with 5% AA extract of HRS flowers. After drying the dyed fibres, neither the colour washed out (on four sequential rinsing by tap water, Figure 2), nor upon exposure to sunlight for 30 days (Table S1). The anthocyanin extract from HRS showed similar dyeing characteristics as the shikonin-based dye extracted from the desert wild plant *Arnebia decumbens*, previously used by the Sadu House. It must be noted that no mordant was used with the shikonin-based colour to attain its excellent washing fastness (Gasmelseed et al. 1989).

The impact of extracting in different solvents affected the absorbance spectrum of the natural dye. In Figure S1, HRS flower extracted in 5% AA showed maximum absorbance in the UV range from 235 to 280 nm. This result is in agreement with the work done by Nevade et al. (2011). It has moderate absorption in the visible region at 515 nm. In Figure S2, HRS flower extract with cold absolute ethanol resulted in maximum absorption between 244 and 282 nm in the UV range and no absorption in the visible range. The absorption spectrum of ethanol extract of HRS was compared to that of 5% AA (Figures S1 and S2) because most of the works reported previously by researchers are focused on ethanol extract. HRS cold extracted with 5% AA had higher light harvesting efficiency than the HRS extracted with cold absolute ethanol. Comparing the absorption spectra in Figures S1–S3, it is obvious that the UV light absorption in Figure S3 increased dramatically. The camel fibre dyed with A–S complex, though it absorbs mostly in the UV region, the results from Table S1 proved that the colour is fixed. This anti-solar property of the dark red/purple colour distinguishes it as a prime candidate for dyeing fabrics, especially those used for outdoor sports activities, as it might protect the skin from the harmful irritation of the solar radiations that mostly lead to sun burn and skin cancers. Therefore, the UV/visible absorption ratio is important for selecting a natural organic dye to colour fabrics, rugs, tents and dye-sensitised solar cells.

Figure 2. Absorbance spectrum of the coloured camel fibres consecutive rinsing (a) first wash, (b) second wash, (c) third wash and (d) fourth wash.
(Mphande & Pogrebnoi 2014), or for the synthesis of nanoparticles using biological sources (Subbaiya & Selvam 2015).

3. Experimental

All experimental details are given in the supplementary material.

4. Conclusion

Bedouin women prefer dyeing white wool with brilliant red colour, as it stands for joy, prosperity and liven up the monotonous surroundings. The red root of *A. decumbens* is available in Kuwait desert, but was not used in dyeing because the colour extracts with organic materials like ethanol, ethyl ether and acetone. Traditionally other natural dyeing substances, especially madder (*Rubia tinctorum*) red roots, were bought from close markets like Hassa or Zubair in Saudi Arabia. Madder roots were extracted with vinegar to give a colourless solution. If this solution was used to top-up wool fibre mordanted with camel basic urine or alum, the wool will be dyed with reddish colour, pleasant but not joyful. Currently, this is the reason behind using synthetic fast and bright coloured dyes.

This paper focused on finding modern recipes to rediscover traditional methods of dyeing in Kuwait desert. The procedure of extracting red HRS to produce a natural pigment from a local plant fulfilled two targets. Firstly, guaranteed the availability of the red pigment constantly for the Sadu house, from a sustainable shrub relative to uncertainty in attaining it from a periodical plant from the desert, like in case of *A. decumbens*, where the plant will not grow if there is no rain. Secondly, the extraction was achieved using available material which is vinegar and the mordant of choice was stannous chloride, which is used as food additive (E 512), where it serves as a colour retention agent and antioxidant (Wikipedia 2015). In addition, Food Standards Agency listed E 512 as safe food additive which is approved by EU (Food Standards Agency 2010). These points address the safety requirements demanded by the Sadu house to keep the tradition of tent and rug weaving natural in all the steps. The colour extracted has additional virtue of protecting the skin from the dangerous effects of free radical exposures and the carcinogenic effect of the ultraviolet rays of the sun.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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