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# Microwave Assisted Green Isolation of Laccaic Acid from Lac Insect (Kerria lacca) for Wool Dyeing

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## ABSTRACT

*The utilization of sustainable products in the applied field is the recent demand of the global community. In this work, the colouring behavior of laccaic acid as a source of natural red dye obtained from lac insect has been studied for the dyeing of wool fabric under microwave treatment. The extract was prepared in acidic medium and stimulated through MW treatment up to 7 minutes and used to dye fabrics. For developing new shades, eco-friendly green mordants such as extracts of acacia and turmeric were employed along with chemical mordants for comparative studies. The obtained results reveal that the extract made in acidic medium after exposure for 5 minutes has given high colour strength onto un-irradiated fabric when employed for 65 minutes at 75 °C. The rating of fastness properties shows that the bio-anchors have given good ratings under optimal conditions. It is inferred that this eco-friendly tool has not only increased the coloring yield of natural anthraquinone dye from lac but also the inclusion of bio-mordants has made the natural coloration process more sustainable. Prog. Color Colorants Coat. 14 (2021), 293-299© Institute for Color Science and Technology.*

## 1. Introduction

The current world is using the huge amount of synthetic dyes for various walks of life [1]. Their synthesis requires such intermediates which not only shed heavy polluted load during their application but also cause big damage to the ecosystem, agriculture, water bodies, etc. [2]. These dyes have also become major threats for human health due to their acute mutagenic, toxic and carcinogenic properties [3]. Now the world is moving towards green products which not only improves the health of the global ecosystem but also plays an additional role as a medicine [4]. Out of these green products, natural dyes are nowadays being welcomed by global community [5]. Bio-colourant such as natural dyes obtained from biological sources such as insects, plants, animals, and microbes can be considered as the alternative sources to synthetic dyes [6]. These are not only eco-friendly and ayurvedic in nature but also have more compatibility with the environment due to having excellent biological properties [7, 8] such as their antioxidant, anti-allergic, anti-fungal, anti-microbial properties [9, 10]. Due to the health benefits attached towards the use of natural dyes and increased consciousness of people towards their health, the use of natural dyes has gained a momentum in various field of life [11, 12].

In the current era of awareness regarding health and

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environmental benefits of the natural dyes, presence of the wide range of colourants in these bio-colourants also increased their use in textile industry [13]. There are different sources to obtain bright red colour from the natural animal sources such as cochineal [14], kermes [15] and Lac [16]. The recent work has been undertaken to explore the potential of the lac dye as a natural dye source. Lac dye is obtained from the female insect known as *Kerria lacca* [16], which is anthraquinone based structure (Figure 1) natural red colourant (Laccaic acid, CI Natural Red 25) [17]. Lac dyes being acidic in nature and only soluble in water, have a strong affinity for the protein substrate such as silk and wool [18].

Although natural colourant is safe and blessing of nature, yet their isolation yield and colour characteristic is under strict observations because conventional methods used for improving their dyeing behavior are taking a lot of time, money, solvent and labor [19]. Now modern methods are being used for the improvement in extraction yield and colouring properties of natural dyeing along with biomordants and sustainable chemical mordants for enhancing the colour fastness properties [20]. Among these modern tools, microwave radiations (MW) are a commercially viable source of heating for the purpose [21]. It is the most effective source for isolation because being leveled and uniform heating source; these rays cause effective mass transfer kinetics through powder-solvent interaction [22]. These rays via rapture of animal cell wall add value in the interaction of powder with solvent via mass kinetics to raise the yield through a short period. Previously, it has also been observed that microwave treatment (MW) enhances colour strength and colourfastness properties by tuning the fabrics physically without causing any chemical change in the functional site of fabric [23].

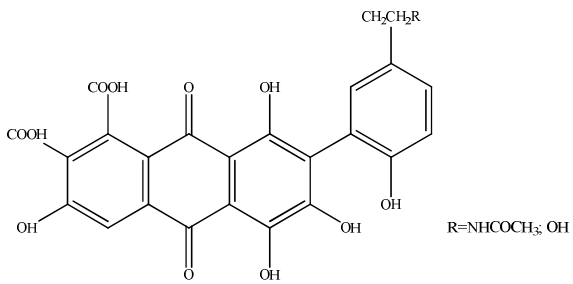


**Figure 1:** Structure of Laccaic Acid.

In view of the advantages of microwave for improving isolation yield of colourant and bio-mordanting for improving fastness ratings, this study has been aimed to explore the colourant potential of anthraquinone based colour from lac insect on the wool fabric under the influence of Microwave irradiation and to utilize bio-mordants for improving colour strength and colourfastness properties.

## 2. Experimental

### 2.1. Collection of material

Lac dye powder was obtained from the Dharma Trading Company, whereas wool fabric (70 g/m$^2$) was purchased from the local textile industry. Two sources of bio-mordants such as Acacia bark and Turmeric rhizomes were purchased from supermarket Faisalabad, Pakistan. The salts of Al (Alum) and Fe (ferrous sulfate) as chemical anchors were purchased from Merck Germany.

### 2.2. Isolation and irradiation

The extraction of anthraquinone based natural red colourant was carried out by the microwave-assisted extraction (MAE) method. During the extraction process owing to the colourant solubility and the nature of fabric two solvents such as acidic and acidified methanolic media were used. The extracts were obtained by refining the crude powder (4 g) with 100 mL of the given solvent for 45 minutes keeping the powder to a solvent ratio of 1:25. After filtration respective extracts and fabrics were treated with MW irradiation at high power for 3-7 minutes [11, 18]. To observe the colour strength (K/S), irradiated and un-irradiated respective extracts were used to dye irradiated and un-irradiated fabrics at 80 °C for 45 minutes keeping extract to fabric ratio of 25:1.

### 2.3. Mordanting conditions

After finding the best medium for the extraction, the dyeing of un-irradiated fabrics was carried using irradiated acid solubilized lac extract. For improving the colour strength and enhancing dye-ability of fabric, two bio-mordant (*Acacia*, *Turmeric*) and two eco-friendly chemical mordants (salts of $Al^{3+}$ and $Fe^{2+}$) were used [17, 21]. For developing new shades with enhanced fastness ratings, mordanting (pre & post) was done for 65 minutes at 75 °C keeping mordanted to fabric ratio of 25:1 [7, 17,18].

### 2.4. Assessment of colour strength and fastness properties

For assessment of colour characteristics of dyed fabrics, Spectra-flash spectrophotometer (SF 600, USA) was used for computing colour characteristics by fallowing Kubelka–Munk equation. However, for observation of the role of bio-mordanting in comparison with chemical bio-mordants ISO standards for colourfastness to light (ISO 105 BO2), washing (ISO 105 C03) and rubbing (ISO 105 X12) were employed to rate these characteristics.

## 3. Results and Discussion

### 3.1. Effect of microwave irradiation on dyeing

Microwave radiations as ecofriendly sustainable tool have increased the isolation yield due to unique caution mechanism [23]. It has been found from the results narrated in the Figure 2 that the acid solubilized extract (RE) irradiated for 5 min. has given excellent results onto un-irradiated fabric (NRF). Upon changing medium (Figure 3), the acidify methanol solubilized extract (RE) irradiated for 7 min. has given above mentioned good results onto irradiated fabric (RF). Low irradiation time does not help to rupture animal cell wall for isolation of laccaic acid, whereas too much irradiation may involve isolation of other by-product along with colourant, which upon dyeing affects the shade. Hence using acid solubilized extract, excellent yield is obtained onto irradiated fabric. This is because the irradiation for 5 min, the powder to solvent interaction becomes significant and through leveled heating, the mass transfer kinetics is enhanced, and excellent extraction yield is obtained onto fabrics [24]. It also reduces the size of clusters of dye molecules into small size molecules to make excellent interaction within the fabric to the greater extent [25, 26]. Here irradiation of fabric is also important, where; the previous studies show that the MW irradiation has nothing to do with the functional site of woolen fabrics. However, these rays tune the fabric physically in such a way that the fabric substantivity towards the dye is enhanced. Hence it is recommended that the utilization of laccaic acid extracted from lac powder in acidic medium followed by MW treatment for 5 minutes should be done to dye un-irradiated fabric.

### 3.2. Effect of chemical and bio-mordant on dyeing

Mordanting is an essential step in the colouration of natural fabrics using natural dyes because, through this method, the poor fastness ratings can be improved [28]. It has been found from Figure 4 that 7% of the extract of Turmeric having curcumin and 5% of acacia extract having quercetin have given excellent results when employed before and after onto un-irradiated fabric (NRF) using irradiated acid solubilized extract (RE). The results given in Figure 5, show that 5% of Al, 3% of Fe as pre, 3% of Al and 5 % of Fe as post mordants have given excellent results. Mordants such as a salt of Al and Fe employed through the formation of coordinate covalent bond interact with active structure (-C=O and -OH) of colourant and –CONH- group of wool to give metal dye onto fabric, resulting in excellent colour depth with enhanced fastness characteristics [28-30].

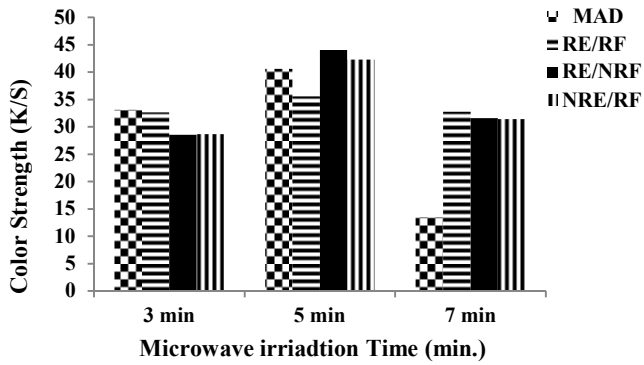


**Figure 2:** Dyeing of irradiated (RE)and un-irradiated wool (NRF) using irradiated (RE) and un-irradiated (NRE) acid solubilized extract of lac powder.

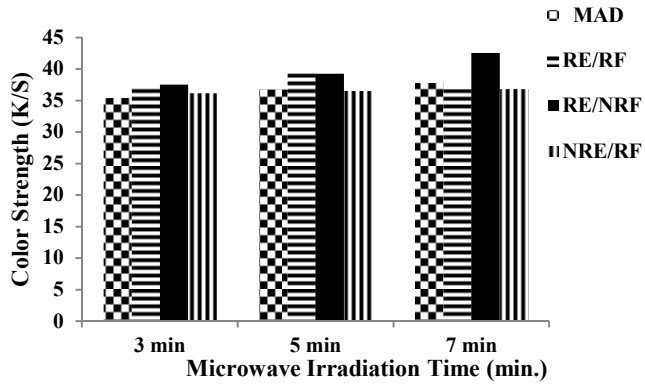


**Figure 3:** Dyeing of irradiated(RF) and un-irradiated wool (NRF) using irradiated (RE) and un-irradiated (NRE) acidify methanol solubilized extract of lac powder.

Upon utilization of bio-mordants, the addition of benzene ring and conjugation system, the presence of excess -OH moieties form extra H-bonding with -C=O and -OH of colourant and -CO and -$NH_2$ of wool. This extra bonding also adds value in rating from good to excellent. This is because of the addition of extra H-bonding due to the presence of -OH of Bio-source, with -C=O and -OH of lac dye with amide linkage of wool which gives firm interaction to given new shades and enhanced colouring variables [9, 21, 23, 31]. This is because of stable metal dye complex formation onto fabric before and after dyeing. However, iron having low reduction power has given good colour strength than Al. Overall bio-mordants have given good results when the irradiated solubilized extract is employed for bio-dyeing of un-irradiated fabric.

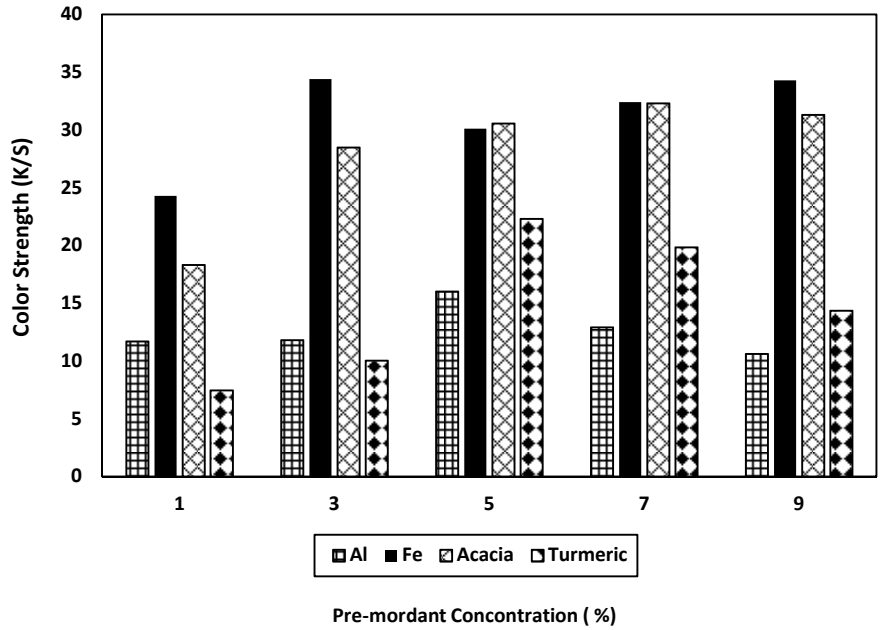


**Figure 4:** Pre-mordanting of un-irradiated wool using irradiated acid solubilized extract of lac powder.

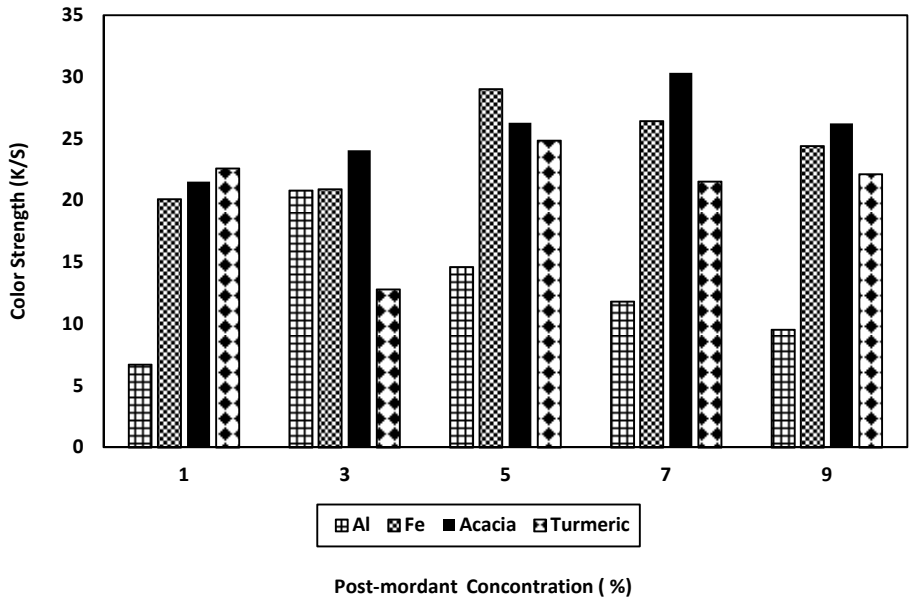


**Figure 5:** Post-mordanting of un-irradiated wool using irradiated acid solubilized extract of lac powder.

### 3.3. Effect of mordanting on fastness properties

The colourfastness ratings are in Table 1 show that in comparison to chemical mordants, bio-mordants have given moderate to good rating to light, washing and rubbing fastness [27]. Similarly, the ratings of colorfastness presented in Table 2 show that by employing chemical mordant such as alum (Al) and iron (Fe) lac dye good washing, light, rubbing fastness properties has been obtained. This may be due to strong complex formation between colourant molecules with mordants [28, 32, 33]. Thus, the application of microwave radiation for the isolation of natural red anthraquinone based colourant from lac insect and the utilization of eco-friendly mordants has made the dyeing process cleaner, nontoxic and optimized for dyeing textile fabric with acceptable fastness properties [23, 34].

**Table 1:** Colorfastness ratings of bio mordanted wool fabric sing irradiated acid solubilized extract of lac powder.

| | Mordant conc. (%) | Turmeric | | | | Acacia | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | LF | WF | DRF | WRF | LF | WF | DRF | WRF |
| Pre Mordanting | 1 | 5 | 4 | 3/4 | 4 | 5 | 4 | 5 | 5 |
| | 3 | 5 | 4 | 4 | 5 | 4/5 | 3 | 5 | 4 |
| | 5 | 5 | 4 | 5 | 4 | 5 | 4 | 5 | ¾ |
| | 7 | 4/5 | 4 | 5 | 4 | 4/5 | 3 | 5 | 4 |
| | 9 | 4/5 | 3 | 5 | 5 | 4/5 | 4 | 5 | 4 |
| Post Mordanting | 1 | 4/5 | 3 | 4 | 5 | 4/5 | 4 | 3/4 | 5 |
| | 3 | 4/5 | 4 | 4 | 5 | 5 | 4 | 4 | 5 |
| | 5 | 4/5 | 4 | 4 | 5/4 | 5 | 4 | 4 | 5 |
| | 7 | 4/5 | 4 | 5 | 4/5 | 4/5 | 4 | 4 | 5 |
| | 9 | 5 | 4 | 5 | 5 | 5 | 4 | 4 | 5 |

LF= light fastness, WF= wash fastness , DRF= dry rubbing fastness, WRF= wet rubbing fastness

**Table 2:** Colorfastness ratings of chemical mordanted wool fabric using irradiated acid solubilized extract of lac powder.

| | Mordant conc (%) | Aluminum | | | | Iron | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | LF | WF | DRF | WRF | LF | WF | DRF | WRF |
| Pre Mordanting | 1 | 4/5 | 3 | 4 | 4 | 4/5 | 4 | 4 | 4 |
| | 3 | 4/5 | 4 | 4 | 5 | 4/5 | 4 | 4 | 4 |
| | 5 | 5 | 4 | 4 | 5 | 4/5 | 4 | 4 | 4 |
| | 7 | 4/5 | 4 | 4 | 5 | 4/5 | 3 | 4 | 4 |
| | 9 | 5 | 4 | 4 | 5 | 5 | 4 | 4 | 4 |
| Post Mordanting | 1 | 5 | 4 | 4 | 4 | 4/5 | 4 | 4 | 4 |
| | 3 | 5 | 4 | 4 | 4 | 5 | 4 | 4 | 4/5 |
| | 5 | 5 | 4 | 4 | 4 | 5 | 4 | 4 | 5 |
| | 7 | 5 | 4 | 4 | 4 | 4/5 | 4 | 4 | 5 |
| | 9 | 4/5 | 4 | 4/5 | 5 | 4/5 | 4 | 4 | 5 |

LF= light fastness, WF= wash fastness , DRF= dry rubbing fastness, WRF= wet rubbing fastness

## 4. Conclusions

Green technologies in various walks of life such as textiles, foods, flavors, cosmetics and pharmaceuticals, etc. have always been welcomed due to their cost, time, and energy effective nature. Microwave treatment is one of such technologies which are not only clean and sustainable nature but also via its fast treatment nature isolate the colourant from crude natural product through the short process of time followed by its application onto natural fabrics. In this study extract from lac dye powder obtained in acidic medium was stimulated for 5 minutes and used to dye un-irradiated woolen fabric. The newly developed tool i.e. bio-mordants on account of value addition and sustainability have been employed to improve colour depth and to enhance fastness rating. It has been concluded that such radiation tools should be used to explore other anthraquinone based animal dyes for utilization in applied walks of life keeping because of the global community and health sustainability of the ecosystem.

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