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**Research Article**

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# Green strategy for Dyeing Wool Fibers by madder Natural Dye

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**ABSTRACT**

*Wool fibers were dyed with natural dye extracted from Madder plant using conventional and microwave heating technique. Wool fibers pretreated with chitosan and tannic acid . The dyeing properties were affected with some factors such as dye extraction, dye concentrations, pH values and the time of dyeing. Color strength was measured for dyed wool fibers. The fastness properties including washing, rubbing, perspiration and light of dyed fibers were measured. The color strength of dyed wool fibers and dye extraction indicates that microwave heating is more effective method than conventional heating. The treated samples exhibited higher results than the untreated. The results illustrated that the pretreatment by chitosan dissolved in citric acid is higher than chitosan in acetic acid compared to untreated dyed wool fibers Dyes Microwaves technique are economic, saving time of dyeing and energy, eco-friendly and enhance the dyeing properties. The results show also good fastness properties. It is necessary to promote non-polluting natural dyes, which involve inexpensive equipment and small scale operations.*

**Keywords:** Madder dye, pretreatment, chitosan, acetic acid, citric acid

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## 1. INTRODUCTION

Natural dyes are colorants that are extracted from various parts of plants such as roots, barks, leaves, flowers, and fruits as well as from insects [1,2]. They have been primarily used for coloring food, leather, and textiles made from natural fibers as silk, wool, cotton[3, 4].

Recently, interest has grown in natural dye applications in the textile industry as a result of the urgent demand for eco-friendly and biodegradable products [5, 6,7]. Although natural dyes are viewed as a safer alternative to synthetic dyes, they have the following disadvantages: low color yield, poor reproducibility, and inferior color fastness properties.

Natural dyes were used as substitute of synthetic dyes due to environmental conditions. They are non-polluting, non-carcinogenic and eco-friendly. Synthetic dyes are broadly disparaged in the world because, they cause water pollution and waste disposal problems. Natural dyes are environmental friendly, biodegradable and non-toxic. They are attracting the awareness of people [8]. Some of natural dyes are anti-allergic and proved to be safe for body contact.

The textile industry must go towards developing of new technologies to reduce the energy and water consumption. The use of microwave in textile wet processing is one way for this purpose. The advantages of microwaves are; using less liquid, exhaust less amount of dyes and leave no waste of liquid dye compared to conventional methods.

Microwave dyeing has other advantages such as less power consumption, easy production of desired shades, and quick dyeing.

In this article, our aim is to show the feasibility of producing high quality dyeing with natural dye extracted from Madder. Dyeing using microwave heating are economically, saving time and energy. This improves our environment and gives opportunities to the fibers industry to catch up with the current consumer trends towards more aesthetic fibers with natural dyeing.

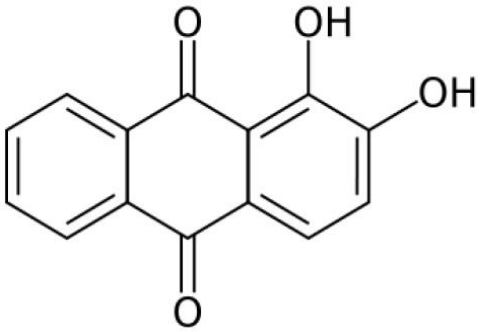
## 2. EXPERIMENTAL SECTION

### 2.1. Materials

**2.1.1. Fibers**: Wool fibers 10/2, supplied by El Mahalla company-Egypt.

**2.1.2. Dyestuffs:**

**Dyestuff Madder dye:** It is a natural coloring matter. Coloring substance used was extracted from Madder(figure 1).




**Chemical structure of Madder dye** **Madder dye**

**Figure1: Chemical structure of Madder dye**

**2.1.3. Extraction of natural coloring matter**

Madder were crushed to the powder form, and then the coloring matter was extracted using (20-80 g of the powder in 1000 ml water) at the boiling for one hour. At the end, the solution was filtered off and left to cool down.

**2.1.4. *Pretreatment with chitosan***

Chitosan (high molecular weight) solution was freshly prepared by dissolving (2.0 g/l) of chitosan in distilled water containing acetic acid (4g/l) and (2.0 g/l) of chitosan in distilled water containing citric acid (4g/l) [9,10] . The wool fibers were immersed in this solution at a liquor ratio 20:1 and treated at microwave for 5 minuets.. Fibers were then squeezed. and air dried

**2.1.5. *Pretreatment with Tannic acid***

The wool fibers were immersed in tannic acid solution (conc. 5%) at a liquor ratio 20:1[11 ]. The samples were treated at microwave for 5 min. Fibers were then squeezed and air dried.

**2.1.6. Dyeing Procedure**

In a dye bath containing different concentrations (20-80 g/l) of madder dye with a liquor ratio 1:100, the wool fabric was dyed by microwave heating at pH (5) for different time periods(1-5 minutes).The dyed samples were rinsed by warm water and then cold water, washed in a bath containing 5g/l non-ionic detergent at 50ºC for 30 minutes, then rinsed and dried in air at room temperature.

2.1.7.**Measurements Color strength (K/S value)**
An Ultra Scan PRO spectrophotometer was used to measure the reflectance of the samples and hence, the K/S was measured spectrophotometrically at wave length 500 nm. The K/S of untreated and pretreated wool fabrics with chitosan and tannic acid was evaluated.

2.1.8. **Fastness properties**
The dyed samples were washed-off using 2 g/l nonionic detergent at 80°C for 30 minutes, and tested according to ISO standard methods. The specific tests were ISO 105-X12 (1987), ISO 105-C02 (1989), ISO 105-E04 (1989), and ISO 105-B02 (1989), corresponding to colour fastness to rubbing, washing, perspiration and light, respectively. The color changes of the samples were assessed against an accurate Gray scale.

## 3. RESULTUS AND DISCUSSION

### 3.1. Dye Extraction

#### 3.1.1. Effect of Dye Amount

The comparative extractability of Madder dye at different concentrations (20- 80 g/l) using microwave heating at 5 minute and conventional heating for one hour at the boiling temperature was measured .Figure 2 illustrates that as the concentration of the dye increases, color strength (K/S) of the dyed Wool fibers increases when using either microwave heating or conventional heating. The figure also illustrates that 40 g/l is the beast concentration of extraction by microwave heating. The obtained values of color strength (K/S) of Wool fibers indicate that microwave heating is more effective method than conventional heating.

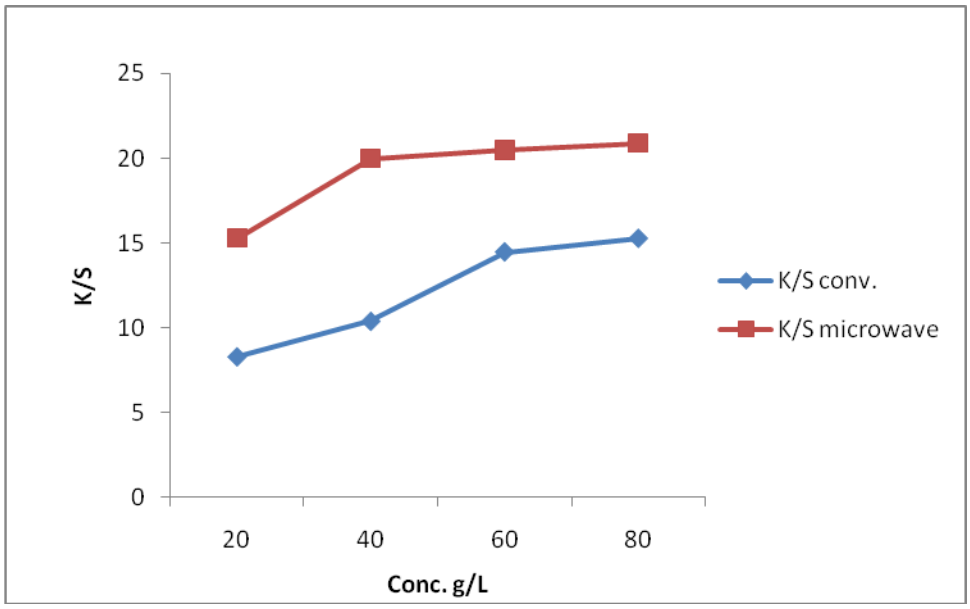



**Figure .2 Effect of Dye Amount conc. on dye extraction by using conventional and microwave heating**

#### 3.1.2. Effect of Extraction Time

The extraction of Madder dye, concentration (40g/l) by microwave heating was carried out for different time in (1-6) minutes and conventional heating for two hours at the boiling .The obtained results from Figure.3,4 indicate that dye extraction by microwave heating is higher than that obtained by traditional heating and the maximum values were obtained after 4 and 60 minutes respectively. This can be attributed to microwave irradiation producing an intensive movement in the liquor due to the wave guides. The resulting distribution of microwaves gives a uniform exposure to which any material moves through.

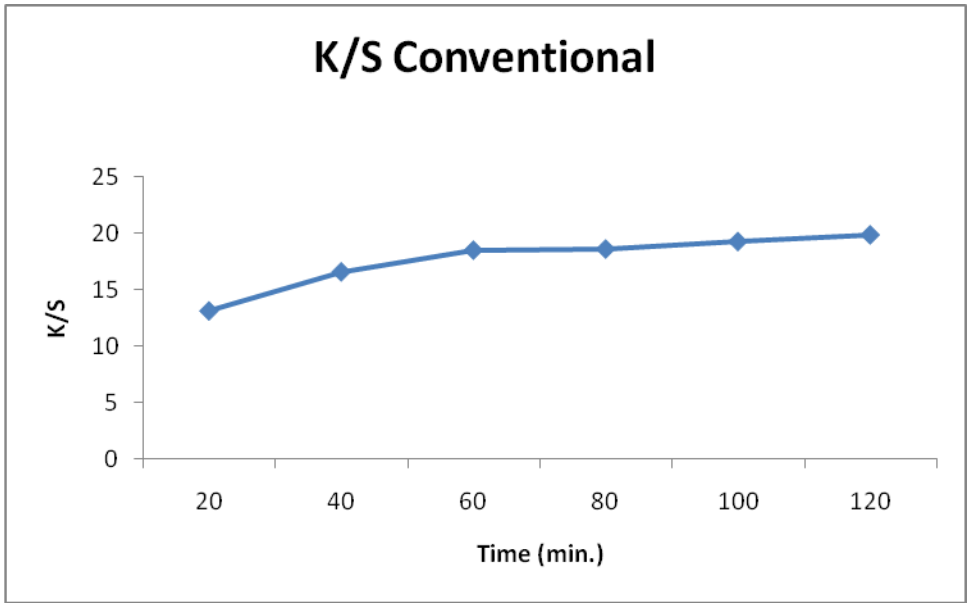



**Figure 3: Effect of time on the extraction of dye by using conventional heating**

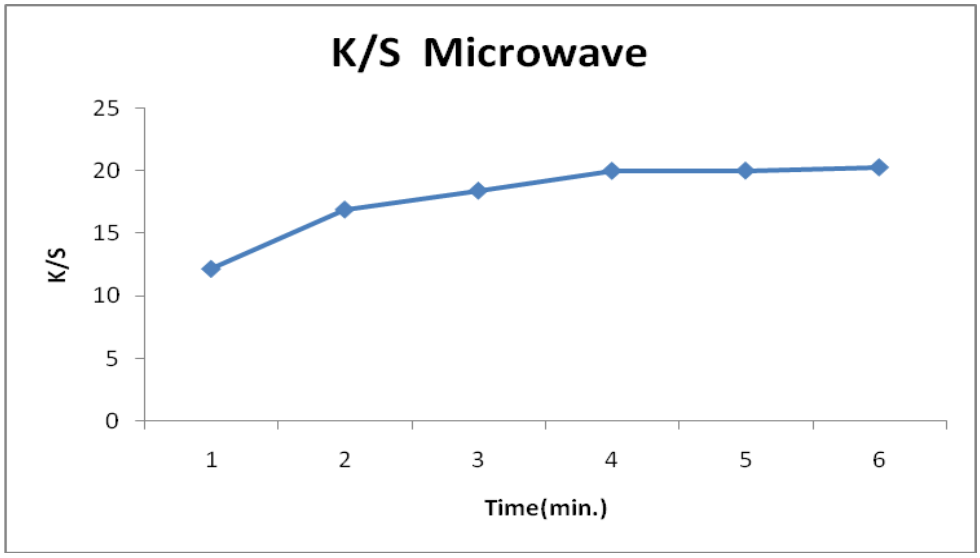



**Figure 4: Effect of time on the extraction of dye by using microwave heating**

### 3.1.3. Effect of pH Level of Dye Bath

The obtained results from figure 5 indicate that ,the pH values (2-11) of the dye bath have considerable effect on the Wool Fibers dye ability when using either dyeing techniques (expressed as K/S).The results illustrate that microwave irradiation and traditional heating enhances dye ability at pH 3. The effect of the dye bath s pH level can be attributed to the waves correlating dye molecules and Wool Fibers. Since Madder dye contains terminal hydroxyl groups, it interacts ionically with the terminal amino groups of Wool Fibers at the acidic condition via the ionic exchange reaction. The weak hydroxyl anion of dye present at an acidic medium replaces that of the acid due to its higher affinity. The anion of dye has a complex nature and when it is bound to the fiber, another kind of interaction takes place together with the ionic force. This ionic interaction leads to an increase in the Wool Fibers dye ability as shown in figure5.

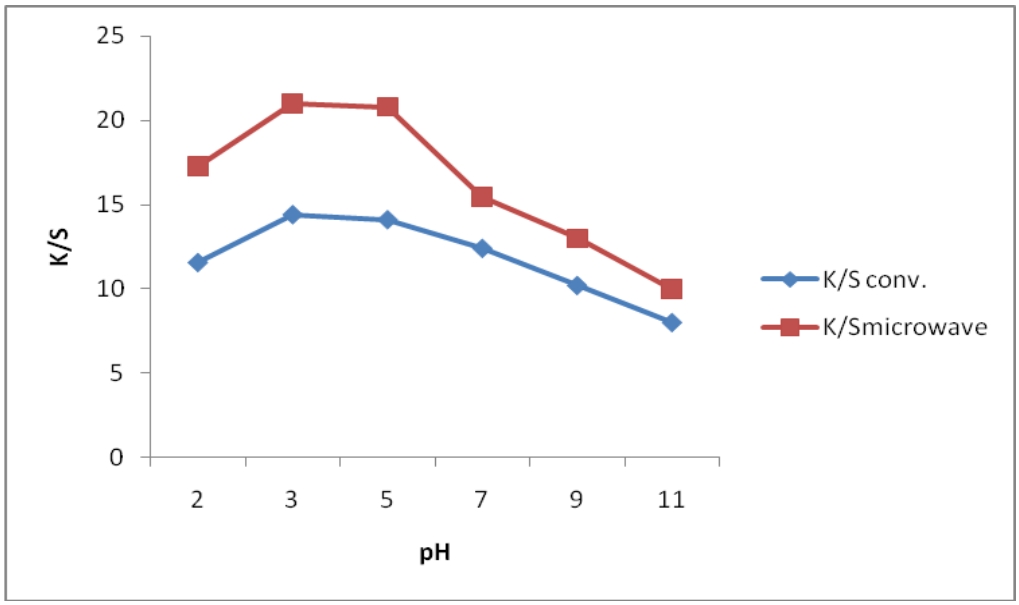



**Figure 5: Effect of dye bath pH on dyeing by conventional and microwave methods on color strength (K/S) of dyed Wool Fibers**

### 3.1.4. Effect of concentration of dye in Dyeing processes

Wool Fibers dyed by natural dye extracted from Madder at different concentrations (20- 80 g/l) of a liquor ratio1:100. Wool fibers were dyed by conventional heating at boiling for one hour and microwave heating for 5 minutes. The results show that the K/S was increased by increasing the concentration of the dye till 60g/l in conventional method. In case of microwave method the K/S increased till 40g/l of the dye. Fig. 6 illustrate that the microwave dyeing is more effective than conventional dyeing.

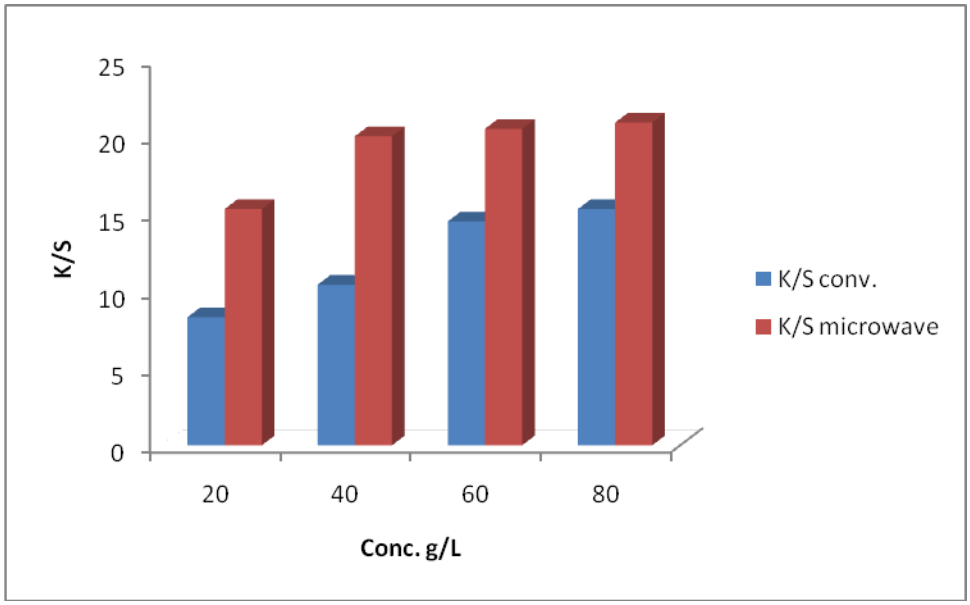



**Figure 6: Effect of conc. of dye on dyeing Wool Fibers by conventional and microwave methods on color strength (K/S)**

### 3.1.5. Effect of Dyeing Time in Dyeing processes

The effect of dyeing time was also studied. Figures 7 and 8 illustrate the effect of dyeing time on the color strength. From figures, it can be observed that the color strength increases as dyeing time increases when using either microwave or traditional heating. The effect of microwaves is greater as dyeing time increases up to 5 minutes, while it takes 50 minutes to achieve the same effect when using traditional heating. Under microwave heating, the

dyed fiber reveals significantly improved levelness. In case of traditional heating, the absorbed dye is distributed unevenly on individual fibers and is concentrated in only a few parts of the outer areas. The use of microwave heating leads to a greater degree of dye penetration. Microwave heating results in a higher and more uniform concentration of the dyestuff on the fiber surface, making it available for diffusion into the fiber interior.

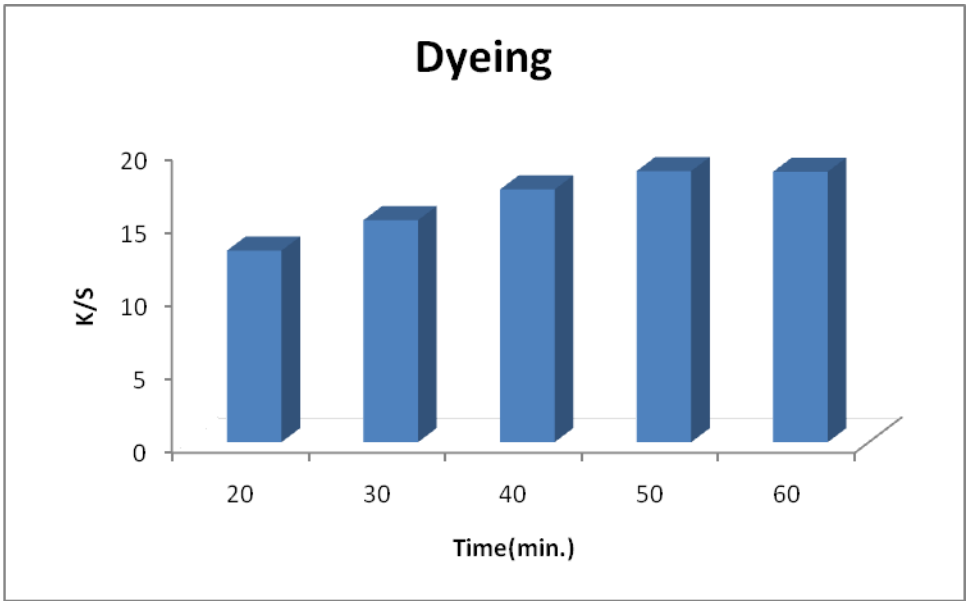



**Figure 7 : Effect of time on dyeing Wool Fibers by conventional method on color strength(K/S)**

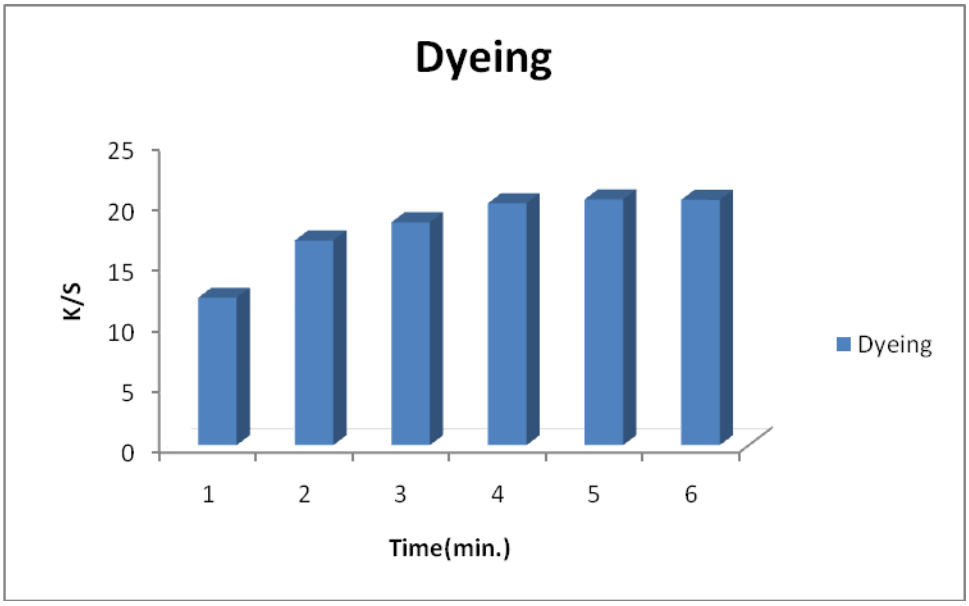



**Figure 8: Effect of time on dyeing Wool Fibers by microwave methods on color strength (K/S)**

### 3.1.6. Effect of The pretreatment on dyeing by microwave:

The pretreatment with chitosan and tannic acid leads to enhancing dyeing of the fiber. It make chemical bonds either to the terminal -NH2 or -COOH groups of the polypeptide chain or to the functional groups present in the side chains of the component amino acids [12,13 ]. Figure 9 showed that the treated samples exhibit higher values of K/S than the untreated samples .Figure 9, also illustrate that the pretreatment by chitosan dissolved in citric acid is higher than chitosan in acetic acid compared to untreated dyed wool fibers Dyes are substantive or adjective.

Tannic acid is a mordant act as chemical bonds between the dye molecules and the functional groups of the fibers, and generally change the color produced by the dye [14,15 ].

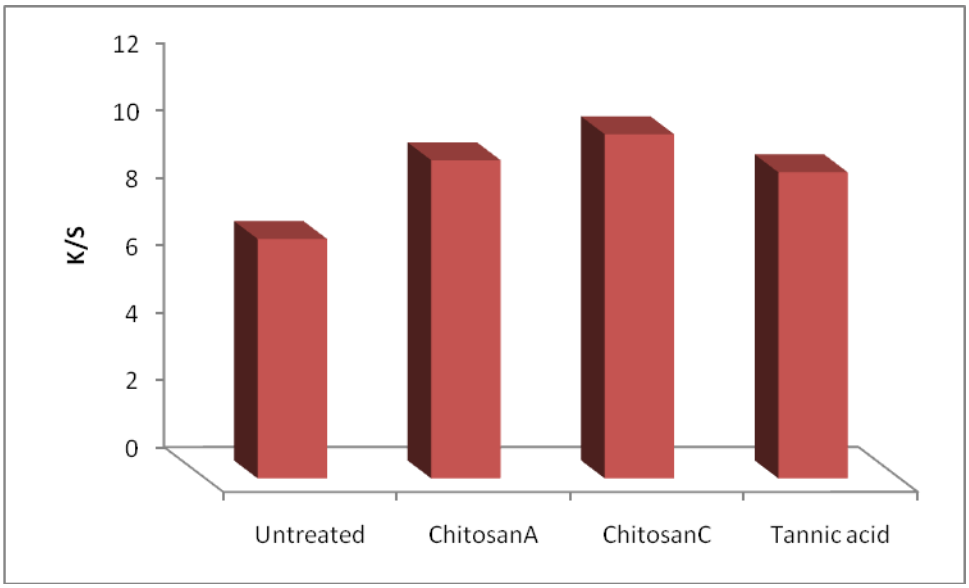



**Figure 9 : Effect of The pretreatment on dyeing by microwave (A: Acetic , C: Citric acid )**

### 3.1.7. Fastness properties

The fastness properties of washing, rubbing, perspiration and light in terms of microwave irradiation and traditional heating are shown in Table 1. The results indicate "good to excellent" fastness properties of the dyed samples when using microwave irradiation and "fair to good" when using traditional heating.

**Table 1.: The color strength and the colorimetric data for wool fibers untreated and pretreated with chitosan in (acitic and citric acid) and tannic acid**

| | K/S | L | A | B | Δ E |
|---|---|---|---|---|---|
| **Untreated** | **7.2** | **51.23** | **26.54** | **33.03** | **6.17** |
| **Chitosan A** | **9.46** | **54.64** | **32.12** | **42.57** | **9.78** |
| **Chitosan C** | **10.23** | **57.91** | **33.10** | **41.58** | **11.52** |
| **Tannic acid** | **9.1** | **60.81** | **31.23** | **35.46** | **0.06** |

*A: Acetic , C: Citric acid*

**Table 2. The fastness properties of wool fibers untreated and pretreated with chitosan in (acitic and citric acid) and tannic acid**

| Conc. | Dyed samples | Fastness to rubbing | | Wash fastness | | Light fastness |
|---|---|---|---|---|---|---|
| | | Wet | Dry | Alt. | Sc. | |
| **0** | **Untreated** | 4 | 4-5 | 4 | 4 | 5 |
| **2g/L** | **Chitosan A** | 4-5 | 4-5 | 5 | 5 | 6-7 |
| | **Chitosan C** | 5 | 5 | 5 | 5 | 7 |
| **5g/L** | **Tannic acid** | **4** | 4 | 4 | 4 | 6 |

*Alt: Altration, Sc: Stainning cotton, A: Acetic , C: Citric acid*

## 4. CONCLUSION

Microwaves heating were found effective in dye extraction from natural dye Madder and dye uptake by Wool Fibers. The enhanced effect was about 50% more than conventional heating. Other additional features about microwaves are that, they are cheaper, more economical, eco-friendly, and produce a higher dye uptake as compared to conventional techniques. The results illustrated that the pretreatment by chitosan dissolved in citric acid is higher than chitosan in acitic acid compared to untreated dyed wool fibers Dyes Madder shows good fastness properties (washing, rubbing, perspiration and light) of Wool Fibers. The fastness properties seem to achieve the best results in the case of microwave heating as compared with traditional heating. Also, microwaves heating use much less liquid,

they can exhaust or save dyes and leave no waste of liquid dye compared to conventional methods. Microwave dyeing has other advantages such as less power consumption, easy production of desired shades and quick dyeing. In the case of microwave dyeing Wool fibers takes only 5 minutes for dyeing , while conventional dyeing requires 50 minutes of heating at boiling. This research proves the feasibility of high-quality natural dye extracted from Madder.

It was found that the use of microwave heating in dyeing processes save energy and time. Dye uptake increased to 50%, dyeing time reduced to 90%, savings energy 90 % and gave Uniform dyeing.

So, the results of this article offer a new technique for dyeing wool fibers by environmentally friendly method and saving time and energy.

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