



## Microwave-assisted dyeing of wool fabrics with natural dyes as eco- friendly dyeing method: part II. The effect of using different mordants

Morsy A. El-Asasery<sup>1,\*</sup>, Abdelhaleem M. Hussein<sup>2</sup>, Nahed M. Nour El-Din<sup>3</sup>, Mohamed O. Saleh<sup>2</sup>, and Abu-Bakr A. El-Adasy<sup>2</sup>

<sup>1</sup> Dyeing, Printing and Auxiliaries Department, Textile Industries Research Division, National Research Centre, Cairo 12622, Egypt

<sup>2</sup> Department of Chemistry, Faculty of Science, Al Azhar University, Assiut 71524, Egypt

<sup>3</sup> Ecology and Range Management Department., Ecology and Dry Lands Agriculture Division, Desert Research Center, 1 Mathal El-Mataria St, 11753 Matariya, Cairo, Egypt



CrossMark

### Abstract

Our current study aims to study the effect of different metallic mordants on the new natural dyes that we were able to extract in a previous study. The extent to which the intensity of colour is affected by the presence of metallic mordants have been studied and also the effect of the concentration of each mordant on the natural dyes was investigated. The changes that took place on the colour axes were screened, and then the fastness of the new dyes were studied after applying them to the wool fabric.

### 1. Introduction

Mordants are mineral salts added to the dyeing process in order to provide chemical bonds between the natural dye and the fabric. They improve the color fastness of the fabrics to be dyed due to easily bonding with natural dye [1-4]. There are some transitional elements that were used in the past as a mordant, such as Cu<sup>2+</sup> and Cr<sup>6+</sup>, but they are not currently recommended due to their environmental damage. Therefore, these harmful elements were replaced by other environmentally friendly elements such as aluminum, iron which used as a mordants to reduce potential harm to human health [5]. Alum usually refers to a hydrated double sulfate of potassium hydrogen sulfate (KAl (SO<sub>4</sub>)<sub>2</sub>.12H<sub>2</sub>O), which confers evenness and brightness on dyed fabrics; iron saddens or dulls the color. Tin is particularly used to achieve brighter colors; however, it might damage wool by making it brittle and rough to the touch [6]. The mordant can be applied before, after or simultaneously with dyeing, forming methods

known as pre-mordanting, post-mordanting and simultaneous mordanting [7, 8]. Another agent used to improve the color fastness of natural dyes on textiles is tannic acid [9-11]. The use of tannin compounds dates back to the 18th century when the quality and utility of animal skins were enhanced through the tannin process [12]. Regarding the dyeing of cotton or wool fabrics, tannic acid forms a color lake with dye inside the fiber which fixes dye to fibers more strongly [6]. It improves the color stability of the tissues to be dyed due to the ease of its association with the natural dye.

### Experimental

#### ▪ Metallic mordants.

The wool fabrics dyed with D1, D2 and D3 dyes at the optimum values obtained as described before in our previous paper [13] were mordanted with different mordant by simultaneous mordanting method. Ferrous sulphate, stannous chloride, potassium alumm. sulphate and copper sulphate were used as a metallic mordants.

\*Corresponding author e-mail: [elapaserym@yahoo.com](mailto:elapaserym@yahoo.com).; (Morsy Elapasery).

EJCHEM use only: Received date here; revised date here; accepted date here

DOI: 10.21608/EJCHEM.2021.72139.3589

©2021 National Information and Documentation Center (NIDOC)

### ▪ Substrates

Scoured and bleached wool fabric was purchased from Misr for Spinning and Weaving Company, Mahalla El-Kobra, Egypt.

### ▪ Microwave assisted extraction

- The process of extraction was carried out by using microwave. A Samsung oven model MS404MADXXBB.

### ▪ Microwave dyeing method

The extracted dye were accompanied with metallic mordant in different concentration and then applied to wool fabrics with M: L ratio 1:100 and the dyeing time was 1 hour.

### Color measurements

Reflectance Spectrophotometer is the device used to measure the intensity of color on dyed wool fabrics and light reflection technology has been applied for that. The dye yield was determined on the dyed fabric using a spectrophotometer. Kubelka-Munk's Equation (1). Equation was applied to find out the intensity of the color of dye, which is symbolized by the symbol K/S [14].

$$K/S = [(1 - R) / 2R] - [(1 - R_0) / 2R_0] \quad (1)$$

Where R is the reflectance of dyed samples, K is the absorption coefficient, S is the scattering coefficient, and  $R_0$  = decimal fraction of the reflectance of the undyed fabric.

### ▪ Fastness properties

#### 1- Color fastness to washing

The color stability to washing was determined by method of ISO 105-C02:1989 [15]. A specimen of the dyed fabric was placed between two bleached slices, one of them is cotton fabric and the other of the wool fabric, and attached with them by hand stitching, and then it was soaked in an aqueous solution consisting of 5 g/L of nonionic detergents at a liquor ratio of 1:50 for 30 minutes at of 60 °C and then sample was rinsed thoroughly with manual squeeze, and then sample was allowed to dry. Gray scale was applied to assess the color fastness to wash.

#### 2- Color fastness to perspiration

Using L-histidine monohydro-chloride monohydrate (0.5 g), sodium chloride (5 g), and sodium dihydrogen orthophosphate dihydrate (2.2 g) in one liter of distilled water, a solution of artificial sweat was obtained. For have acidic sweat pH was adjusted to 5.5 by 0.1 N of NaOH and by the same

way an alkaline sweat was obtained by adjusting pH of solution at 8.0. The fastness test was performed according to the following procedure: Between two different parts of uncolored patterns a sample of dyed fabric (5×4 cm) was sutured. The samples were soaked in both solutions undergoing agitation and pressing for 15-30 min to have perfect wetting. Samples undergo a load of 4-5 kg while the sample was placed between two plates of plastic or glass. Then these plates were placed at a temperature of 37 °C vertically for four hours, and then a gray scale change technique was used to assess the color fastness to perspiration.

#### 3- Color fastness to light

The method of measuring the stability of color fastness to light is ISO 105-B02:1988 test technique where the dyed sample placed for 35 hours to a carbon arc lamp and then using the blue color scale to investigate color change of the tested samples.

#### 4- Color fastness to rubbing

By applying the technique of ISO 105-X12:1987 test the color fastness to crocking was determined. When dyed fabric undergoes rubbing, it is can be estimate if dye move from dyed fabric to another one or not. The wet and dry rubbing had been estimates.

## 3. Results and discussion

### Optimization of mordant concentrations

Different concentrations from metallic mordants were applied to dyeing process of wool by D1, D2 and D3 (from 0.02 gm/L to 0.1 gm/L) and then K/S was tested. Data of table 1 and Fig.1 show that the color strength increase by mordant concentration increasing and by using ferrous sulphate as a mordant color shift slightly toward darkness where (L) values decreased and from negative values of (a) and low positive values of (b) color shift from yellow zone to green zone. Stannous chloride gives new dye where (L) values refer that color move to darkness zone and high positive values of (b) low positive values of (a) indicate that dye go to yellow color.

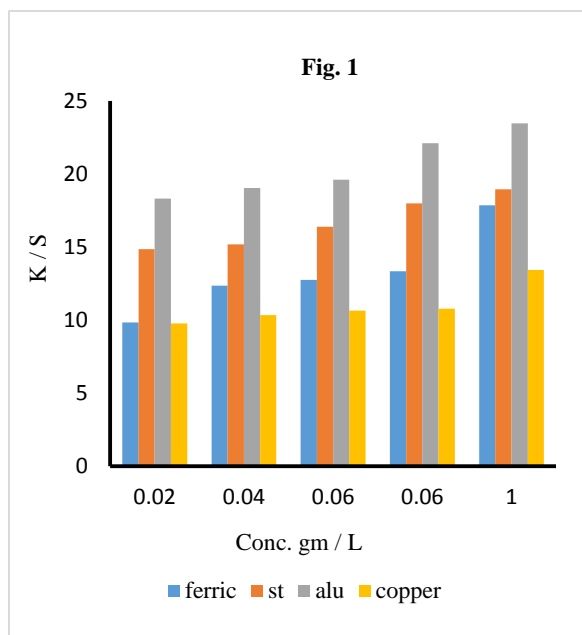


Fig. 1 Effect of mordants concentration on D1.

In the same behavior potassium alumm. sulphate has low positive values of (a) and high positive value of (b) then dye found in yellow zone but it becomes darker and that was clear from low positive values of (L).

Copper sulphate as a mordant with D1 produced new dye darker than original dye and dye which produced from ferrous sulphate and D1 that was clear from (L) values and negative values of (a) and low positive values of (b) show that dye found in green color zone.

Fig. 2 refers that K/S values increased by increasing concentration of metallic mordants. That is clearly from (L), (a) and (b) values.

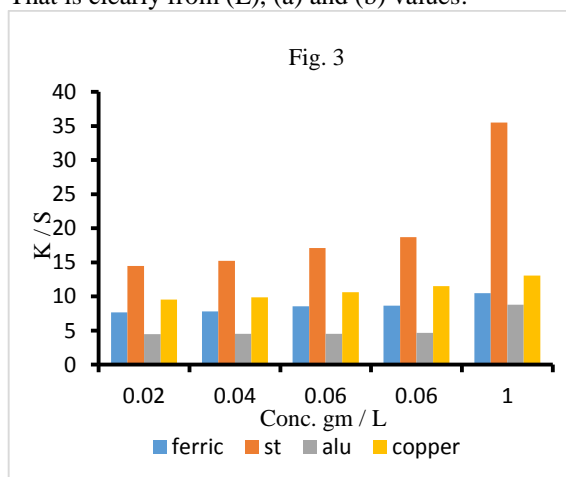


Fig. 3 Effect of mordants concentration on D3.

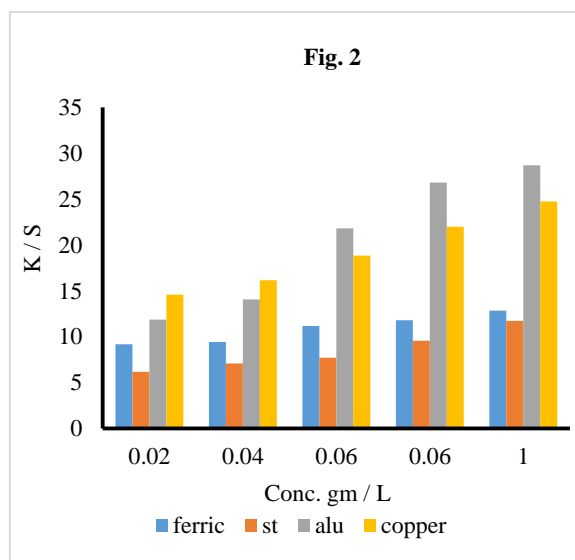


Fig. 2 Effect of mordants concentration on D2.

Values of (L), (a) and (b) in table 2 indicate that mixing of ferrous sulphate with D2 color shift slightly toward darkness and color found green zone. Stannous chloride with D2 gives a dye present in darkness zone and go to yellow color due to (L), (a) and (b) values. Combination of potassium alumm.sulphate and D2 gives dye has low positive values of (a) and high positive value of (b) then dye found in yellow zone but it becomes darker and that was clear from low positive values of (L). The same behavior of the Copper sulphate as a mordant with D1 is seen when it combined with the D2 where new dye is darker than original dye. That is clearly from (L), (a) and (b) values. Fig.3 shows that increasing concentration of metallic

Fig.3 shows that increasing concentration of metallic mordants when they correlate with D3 leads to increasing in color intensity. Information of table 3, show that the selected metallic mordants have the same behavior with D3 when then mixed with it where dye which produced from mixing D3 with ferrous sulphate is dark and found in green zone. Mixture of D3 and stannous chloride produced dye found in dark area and yellow zone and dye found in green color zone. Mixture of potassium alumm. sulphate and D3 gives dye present in yellow zone and dark area. Dye of Copper sulphate as a mordant with D3 is seen when it combined with the D3 is darker than original dye and dye found in green color zone.

Table 1. Effect of metallic mordant on D1.

| mordant           | Mordant conc. | K / S | L     | a      | b     | c     | h     |
|-------------------|---------------|-------|-------|--------|-------|-------|-------|
| Without mordant   |               | 12.28 | 64.02 | 5.04   | 25.22 | 25.72 | 78.70 |
| Ferric sulphate   | 0.02          | 9.83  | 51.52 | - 0.54 | 10.12 | 10.13 | 93.03 |
|                   | 0.04          | 12.35 | 44.03 | - 0.89 | 9.76  | 9.80  | 95.22 |
|                   | 0.06          | 12.75 | 45.32 | - 1.24 | 19.17 | 9.26  | 97.69 |
|                   | 0.08          | 13.34 | 43.33 | - 0.77 | 10.06 | 10.09 | 94.40 |
|                   | 0.10          | 17.85 | 44.43 | - 1.20 | 9.02  | 9.10  | 97.59 |
| Stannous chloride | 0.02          | 14.85 | 56.69 | 2.28   | 29.84 | 29.93 | 85.63 |
|                   | 0.04          | 15.19 | 52.08 | 1.71   | 29.92 | 29.97 | 86.73 |
|                   | 0.06          | 16.38 | 51.40 | 1.20   | 29.21 | 29.23 | 87.65 |
|                   | 0.08          | 17.98 | 52.07 | 0.59   | 28.14 | 28.15 | 88.81 |
|                   | 0.10          | 18.94 | 52.11 | - 0.49 | 27.27 | 27.28 | 91.03 |
| Pothach. Aluom    | 0.02          | 18.31 | 72.34 | 6.68   | 45.37 | 45.45 | 86.62 |
|                   | 0.04          | 19.03 | 71.77 | 5.25   | 55.26 | 55.50 | 84.57 |
|                   | 0.06          | 19.6  | 71.29 | 6.02   | 58.80 | 59.10 | 84.16 |
|                   | 0.08          | 22.10 | 71.84 | 6.80   | 63.86 | 64.22 | 83.92 |
|                   | 0.10          | 23.47 | 72.94 | 6.27   | 62.42 | 62.73 | 84.27 |
| Copper sulphate   | 0.02          | 9.77  | 69.21 | - 0.04 | 32.15 | 32.15 | 90.07 |
|                   | 0.04          | 10.33 | 70.01 | 0.19   | 33.50 | 33.50 | 89.67 |
|                   | 0.06          | 10.65 | 70.23 | 0.21   | 35.46 | 35.46 | 89.66 |
|                   | 0.08          | 10.78 | 71.65 | - 0.02 | 35.48 | 35.48 | 90.04 |
|                   | 0.10          | 13.42 | 72.06 | 0.20   | 33.71 | 33.71 | 89.66 |

Table 2. Effect of metallic mordant on D2.

| mordant           | Mordant conc. | K / S | L     | a      | b     | c     | h     |
|-------------------|---------------|-------|-------|--------|-------|-------|-------|
| Without mordant   |               | 8.86  | 65.96 | 4.59   | 24.49 | 24.92 | 79.37 |
| Ferric sulphate   | 0.02          | 9.19  | 52.48 | 0.45   | 12.63 | 12.64 | 87.94 |
|                   | 0.04          | 9.44  | 45.72 | 0.98   | 12.90 | 12.93 | 85.67 |
|                   | 0.06          | 11.19 | 50.21 | 0.64   | 13.43 | 13.45 | 87.28 |
|                   | 0.08          | 11.79 | 52.28 | 1.70   | 15.94 | 16.04 | 83.91 |
|                   | 0.10          | 12.84 | 50.08 | 1.87   | 16.16 | 16.26 | 83.41 |
| Stannous chloride | 0.02          | 6.18  | 71.67 | - 1.66 | 28.18 | 28.20 | 91.74 |
|                   | 0.04          | 7.11  | 71.63 | - 1.08 | 27.53 | 27.55 | 92.26 |
|                   | 0.06          | 7.73  | 72.77 | - 0.68 | 30.61 | 30.61 | 91.27 |
|                   | 0.08          | 9.57  | 75.59 | - 2.08 | 28.18 | 28.26 | 94.22 |
|                   | 0.10          | 11.74 | 70.09 | 1.22   | 31.18 | 31.20 | 87.75 |
| Pothach. Aluom    | 0.02          | 11.87 | 76.40 | - 1.17 | 38.46 | 38.48 | 91.74 |
|                   | 0.04          | 14.08 | 76.05 | 2.88   | 45.92 | 46.01 | 86.41 |
|                   | 0.06          | 21.81 | 74.18 | - 0.75 | 39.30 | 39.31 | 91.09 |
|                   | 0.08          | 26.81 | 76.59 | - 0.01 | 28.28 | 28.28 | 90.02 |
|                   | 0.10          | 28.70 | 72.71 | - 0.50 | 35.76 | 35.76 | 90.79 |
| Copper sulphate   | 0.02          | 14.60 | 52.83 | 4.05   | 29.46 | 29.74 | 82.18 |
|                   | 0.04          | 16.15 | 49.57 | 2.13   | 28.60 | 28.68 | 85.74 |

|  |      |       |       |      |       |       |       |
|--|------|-------|-------|------|-------|-------|-------|
|  | 0.06 | 18.86 | 50.37 | 0.46 | 26.47 | 27.47 | 89.01 |
|  | 0.08 | 21.98 | 44.91 | 1.94 | 26.67 | 26.75 | 85.83 |
|  | 0.10 | 24.73 | 43.94 | 0.86 | 24.23 | 24.25 | 87.96 |

Table 3. Effect of metallic mordant on D3.

| mordant           | Mordant conc. | K / S | L     | a      | b     | c     | h     |
|-------------------|---------------|-------|-------|--------|-------|-------|-------|
| Without mordant   |               | 16.89 | 65.03 | 5.58   | 26.36 | 26.95 | 78.06 |
| Ferric sulphate   | 0.02          | 7.66  | 51.80 | 0.06   | 11.07 | 11.07 | 89.69 |
|                   | 0.04          | 7.82  | 50.20 | 0.01   | 11.60 | 11.60 | 89.95 |
|                   | 0.06          | 8.57  | 4.77  | 0.03   | 11.82 | 11.82 | 90.18 |
|                   | 0.08          | 8.65  | 49.69 | 0.65   | 14.05 | 14.07 | 87.36 |
|                   | 0.10          | 10.48 | 44.19 | - 0.04 | 11.08 | 11.08 | 90.18 |
| Stannous chloride | 0.02          | 14.47 | 75.35 | 0.03   | 28.90 | 28.90 | 89.95 |
|                   | 0.04          | 15.21 | 77.50 | - 2.02 | 38.68 | 38.73 | 92.99 |
|                   | 0.06          | 17.09 | 75.24 | - 0.04 | 37.48 | 37.48 | 90.07 |
|                   | 0.08          | 18.71 | 76.74 | 0.39   | 44.43 | 44.43 | 89.90 |
|                   | 0.10          | 35.49 | 68.18 | 4.29   | 37.01 | 37.26 | 83.39 |
| Pothach. Aluom    | 0.02          | 4.46  | 69.10 | 3.65   | 25.11 | 25.37 | 81.72 |
|                   | 0.04          | 4.51  | 69.74 | 3.44   | 25.53 | 26.76 | 82.33 |
|                   | 0.06          | 4.54  | 69.71 | 3.50   | 26.13 | 26.36 | 82.37 |
|                   | 0.08          | 4.65  | 69.77 | 3.06   | 26.40 | 26.58 | 83.40 |
|                   | 0.10          | 8.81  | 63.52 | 5.40   | 28.40 | 28.91 | 79.23 |
| Copper sulphate   | 0.02          | 9.54  | 56.47 | 4.86   | 24.21 | 24.70 | 78.66 |
|                   | 0.04          | 9.88  | 54.12 | 5.44   | 25.17 | 54.12 | 77.82 |
|                   | 0.06          | 10.62 | 53.13 | 5.55   | 24.77 | 25.38 | 77.37 |
|                   | 0.08          | 11.53 | 53.19 | 4.98   | 25.09 | 25.35 | 78.74 |
|                   | 0.10          | 13.06 | 52.14 | 4.79   | 24.89 | 25.35 | 79.11 |

Table 4:Fastness properties of dyed fabrics with D1 and selected metallic mordants

| D1 & mordants             | Washing fastness |    |    | Perspiration fastness |    |    |          |    |    | Light fastness |     |
|---------------------------|------------------|----|----|-----------------------|----|----|----------|----|----|----------------|-----|
|                           | Alt              | SC | SW | Acidic                |    |    | Alkaline |    |    |                |     |
|                           | Alt              | SC | SW | Alt                   | SC | SW | Alt      | SC | SW |                |     |
| Ferrous sulphate          | 5                | 5  | 5  | 5                     | 5  | 5  | 5        | 5  | 5  | 5              | 4-5 |
| Stannous chloride         | 5                | 5  | 5  | 5                     | 5  | 5  | 5        | 5  | 5  | 5              | 5   |
| Potassium alumm. sulphate | 5                | 5  | 5  | 5                     | 5  | 5  | 5        | 5  | 5  | 5              | 5   |
| Copper sulphate           | 5                | 5  | 5  | 5                     | 5  | 5  | 5        | 5  | 5  | 5              | 4-5 |

Table 5:Fastness properties of dyed fabrics with D2 and selected metallic mordants.

| D2 & mordants             | Washing fastness |    |    | Perspiration fastness |    |    |          |    |    | Light fastness |     |
|---------------------------|------------------|----|----|-----------------------|----|----|----------|----|----|----------------|-----|
|                           | Alt              | SC | SW | Acidic                |    |    | Alkaline |    |    |                |     |
|                           | Alt              | SC | SW | Alt                   | SC | SW | Alt      | SC | SW |                |     |
| Ferrous sulphate          | 5                | 5  | 5  | 5                     | 5  | 5  | 5        | 5  | 5  | 5              | 5   |
| Stannous chloride         | 5                | 5  | 5  | 5                     | 5  | 5  | 5        | 5  | 5  | 5              | 4-5 |
| Potassium alumm. sulphate | 5                | 5  | 5  | 5                     | 5  | 5  | 5        | 5  | 5  | 5              | 5   |
| Copper sulphate           | 5                | 5  | 5  | 5                     | 5  | 5  | 5        | 5  | 5  | 5              | 4-5 |

Table 5: Fastness properties of dyed fabrics with D3 and selected metallic mordants.

| D3&mordants               | Washing fastness |    |    | Perspiration fastness |    |    |          |    |    | Light fastness |     |
|---------------------------|------------------|----|----|-----------------------|----|----|----------|----|----|----------------|-----|
|                           | Alt              | SC | SW | Acidic                |    |    | Alkaline |    |    |                |     |
|                           |                  |    |    | Alt                   | SC | SW | Alt      | SC | SW |                |     |
| Ferrous sulphate          | 5                | 5  | 5  | 5                     | 5  | 5  | 5        | 5  | 5  | 5              | 4-5 |
| Stannous chloride         | 5                | 5  | 5  | 5                     | 5  | 5  | 5        | 5  | 5  | 5              | 4-5 |
| Potassium alumm. sulphate | 5                | 5  | 5  | 5                     | 5  | 5  | 5        | 5  | 5  | 5              | 5   |
| Copper sulphate           | 5                | 5  | 5  | 5                     | 5  | 5  | 5        | 5  | 5  | 5              | 4-5 |

### Conclusion

In this study, the effect of the mordants is monitored on the natural dyes, which extracted from natural plants, and the effect of the mordants concentration on the intensity of the color was studied, and it was found that it increases with the increase in the concentration of the mordant. The stability of new dyes resulting from the combination of new dyes with mordants has been studied, and it has very good fastness properties against light and other excellent properties against washing and perspiration.

### References

- (1) Vankar, P. S.; Shanker, R.; Verma, A. Enzymatic natural dyeing of cotton and silk fabrics without metal mordants. *Journal of Cleaner Production*, **15**, 1441-1450, (2007).
- (2) KOH, J. Handbook of textile and industrial dyeing. Book, chapter, Dyeing of cellulosic fibres. Woodhead Publishing Limited, (2011).
- (3) Křížová, H. Textile Dyeing - Theory and Applications. Book. Chapter Natural Dyes. TUL, Vysokoškolský podnik Liberec s.r.o., Studentská 2. Liberec, (2013).
- (4) Bhattacharya, S. D.; Shah, A. K. Metal ion effect on dyeing of wool fabric with catechu. *JSDC*, **116**, 10-12, (2000).
- (5) Cardon, D. Natural Dyes: Sources, Tradition, Technology and Science. Archetype Publications Ltd, (2007).
- (6) Mahmud-Ali, A.; Fitz-Binder, C.; Bechtold, T. Aluminium based dye lakes from plant extracts for textile coloration. *Dyes and Pigments*, **94**, pp 533-540., 2012.
- (7) Gupta, V. K. Chemistry and Technology of Natural and Synthetic Dyes and Pigments, book., chapter Fundamentals of Natural Dyes and Its Application on Textile Substrates. intechopen., ISBN: 978-1-83968-758-7., (2020).
- (8) Yusuf, M.; Shabbir, M.; Mohammad, F. Natural Colorants: Historical, Processing and Sustainable Prospects. *Natural Products and Bioprospecting*, **7**, 123-145. 2017.
- (9) Saxena, S.; Raja, A. S. M. Roadmap to Sustainable Textiles and Clothing. Book, chapter Natural Dyes: Sources, Chemistry, Application and Sustainability Issues., Springer, ISBN 978-981-287-065-0., (2014).
- (10) Singh, S. V.; Purohit, M. C. Applications of Eco-Friendly Natural Dye on Wool Fibers Using Combination of Natural and Chemical Mordants. *Universal Journal of Environmental Research and Technology*, **2**, 48-55, (2012).
- (11) Edwards, L. C.; Freeman, H. S. Synthetic dyes based on environmental considerations. Part 3: Aquatic toxicity of iron-complexed azo dyes. *Color. Technol*, **121**, 260 – 270, (2005).
- (12) Mongkholrattanasit, R.; Kryštůfek, J.; Wiener, J.; Studničková, J. Properties of Wool and Cotton Fabrics Dyed with Eucalyptus, Tannin and Flavonoids. *FIBRES & TEXTILES in Eastern Europe*, **19**, (2011).
- (13) El-Asasery, M. A. ; Hussein, A. M.; Nour El-Din, N. M. ; Saleh, M. O.; El-Adasy, A. A. Microwave-assisted Dyeing of Wool Fabrics with Natural Dyes as Eco-Friendly Dyeing Method: Part I. Dyeing Performance and Fastness Properties. Inpress.
- (14) Bhajan, H.; Kumar, S.; Bharati, A. Handbook of Natural Dyes and Pigments. Book, chapter Mordants and their applications. Woodhead Publishing India, ISBN 978-93-80308-54-8.