

# Extraction of Dyestuff From Madder Plant (*Rubia tinctorum L.*) and Dyeing of Wool, Feathered-Leather and Cotton

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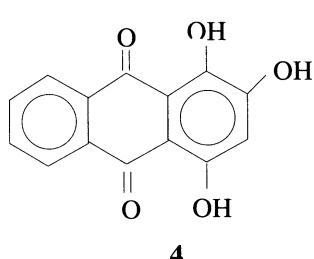
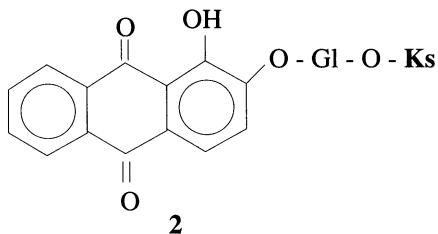
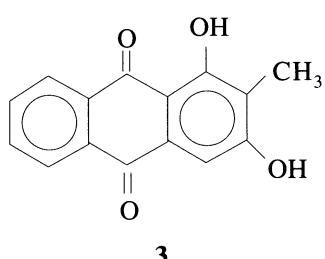
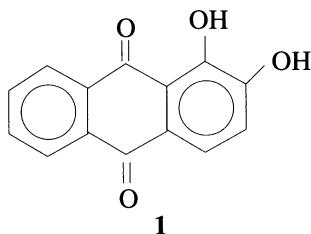
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In this study, the total amount of dyeing substances present in *Rubia tinctorum L.* was found by using the extraction method and then separated in column. All of the dyeings were carried out with red component alizarin (1,2-dihydroxyanthraquinone). Dyeing conditions and other characteristics were determined and in most cases high fast colours were obtained.

## Introduction

Madder plant (*Rubia tinctorum L.*) is an important dye-plant and is known as Turkish Red. Turkish people had used this plant for the first time in 1519. Since 1871, the production and consumption of this plant has decreased. Now, it is used in some regions of Anatolia and in some Asian countries such as Iran, India<sup>1,2</sup>.

*Rubia tinctorum L.* contains mainly alizarin (1,2-dihydroxyanthraquinone) (1) which is produced the hydrolysis of ruberythric acid (2). Rubiadine (1,3-dihydroxy-2-methylanthraquinone) (3) and purpurine (1,2,4-trihydroxyanthraquinone) (4) are other dyestuffs which are present to a lesser extent in the roots of the madder plant<sup>3</sup>.



The amount of red component increases in October and is darker than it is in May and June. Thus, the amount of dyestuff changes in summer and autumn. However, the dyestuffs in *Rubia tinctorum L.* have oxochrome groups such as OH, COOH and C=O. In this study 168 wool, 40 feathered-leather and cotton samples were dyed using mordantation methods and the best dyeing conditions were determined. In addition, a new mordant mixture was obtained.

## Experimental

### General

Soxhlet apparatus was used to extract the dyestuff from *Rubia tinctorum L.* The solvent was evaporated using the Buchi RE 111 Rotary evaporator at Gaziosmanpaşa University. Analytical balance was used for determining the total dyestuff. Light fast were made with a Fadeometer ( Xenotest), washing fasts were carried-out with an Alas Launderometer LHTP model, crocks fast were made with a Crockmeter 225 model in the Merinos factory. Bursa and the Ihlas-Se-Na textile factory in Tokat.

### Plant Material

Madder plant (*Rubia tinctorum L.*) belongs to the Rubiaceae family. It was picked in the center of Tokat in May 1995. The plant was kept in the herbarium of the Plant Protection Department, Faculty of Agriculture, Gaziosmanpaşa University. It was examined and identified by Prof. Dr. Zeki Özer. The herbarium number of *Rubia tinctorum L.* is A.E.F 19571.

### Extraction of Dyestuff from *Rubia tinctorum L.*

Dried and powdered roots (100 g) of *Rubia tinctorum L.* were extracted with 1000 ml of distilled water. The solvent was evaporated and the dark residue was weighed. The total dyestuff was found to be 29.30 %. 20 g of dyestuff was put into columns prepared from silicagel. Distilled CHCl<sub>3</sub> and methanol were used in the mobile phase. Two components were separated (red and yellow). The red one was determined as alizarine (m. p. 289°C) which was already known<sup>4</sup>. The yellow component containing purpurine and rubiadine was not used in the dyeing. All of dyeings were carried out with red component. The amount of alizarine was found to be 60.29% and the other components were found to be 39.71 % (in June)

### Dyeing of Wool (General Procedure)

Dyeing procedures of wool were applied with alizarine using the mordantation methods as given below.:

- 1-Pre-mordantation
- 2-Together mordantation
- 3-Last mordantation

**1-Pre-mordantation:** White woollen strips (1.8 g) were heated in 100 ml 0.1M of mordant solution at 80°C for 1h. After cooling, it was filtered, dried and put into 180 ml of dye-bath (flotte ratio=1/100). After heating for 1h at 80°C, it was allowed to cool. The dyed product was washed with distilled water and dried. This method was applied at pH: 8,6,4 and 2 for each mordant.

**2-Together mordantation:** Mordant (equivalent to a 0.1 M concentration value), 180 ml of dyestuff solution and 1.8 g of woollen strips were mixed in 250 ml of erlenmeyer. This mixture was heated at 80°C

for 1h. After cooling, it was filtered, washed with distilled water and dried. This method was also carried out at pH: 8,6,4 and 2 for each mordant.

**3-Last mordantation:** First 1.8 g of woollen strips were heated in 180 ml of dye-bath at 80° for 1h. After cooling, the woollen strips were filtered. Washed with distilled water and dried. Secondly it was put into 100 ml 0.1 M of mordant solution and heated at 80°C for 1 h. Finally, it was filtered, washed with distilled water and dried. The last mordantation was carried out at pH. 8,6,4 and 2 for each mordant.

The woollen strips dyed according to each of these three methods were kept in 3%  $NH_3$  solution to increase the fasts of the colours.

### Dyeing of Feathered-Leather (General Procedure)

These processes were carried out for the first time using the Pre mordantation and Together mordantation methods.

**1-Pre-mordantation:** The white feathered-leather treated with potassium bichromate (approx. 15 cm<sup>2</sup>) was heated in 100 ml 0.1M of mordant solution at 35 – 40°C for 1h in 300 ml of erlenmeyer. After cooling, it was washed with distilled water, dried and put into 100 ml of dye-bath. It was shaken at frequent intervals for one hour at 35 – 40°C. Finally, the dyed feathered-leather was filtered, washed with distilled water and dried.

**2-Together Mordantation:** The feathered-leather 100 ml of dyestuff solution and mordant agent were mixed in 250 ml of erlenmeyer. This mixture was shaken at frequent intervals for 1 h at 35 – 40°C. Finally, the dyed feathered-leather was filtered, washed with distilled water and dried.

The length of the feather of the feathered-leather used in this work was approximately 1-1.5 cm for each of the two methods, pH was selected as 4.5 and 2.

### Dyeing of Cotton (General Procedure)

Pre mordantation and together mordantation were carried out in dyeing of cotton.

**1-Pre-mordantation:** The cotton (1.5 g 50 cm<sup>2</sup>) was heated at 90°C for 1h in 100 ml of mordant solution. After cooling, it was taken out, washed with distilled water and dried. Then, it was put into 100 ml of dye-bath and heated at 90°C for 1h. After cooling and filtering, the dyed-cotton was washed with distilled water and dried.

**2-Together mordantation:** The cotton (1.5 g, 50 cm<sup>2</sup>) and mordant solution (equivalent to a 0.1 M value) were added to 100 ml of dyestuff solution and heated at 90°C for 1h. After cooling, it was taken out and washed with distilled water and dried.

The pH was selected as 7 and 4. pH was applied as 7 and 2 together dyeing with  $CoCl_2$  and  $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ .

## Results and Discussion

The results for the fast analyses <sup>4,5</sup> and colour codes<sup>6</sup> of dyed wool were given in Table 1., the fast analyses of dyed feathered- leather were given in Table 2, and the analysis of dyed-cotton were given in Table 3.

Extraction of Dyestuff From Madder Plant (*Rubia tinctorum L.*)..., A. ÖNAL.,

**Table 1.** Results of fast analyses and colour codes of dyed woolen strips

Dyeing method	Mordant	Flotte ratio	pH	Bath temp. (°C)	Dyeing period (hr)	Colour code*	light fast	Croc- king moist	fast dry	Washing fast	Hypo-chloride fast
Together-mordant.	FeSO <sub>4</sub>	0.01	8	80	1	18-1341 TB	5(darken)	3-4	4	3-4	-
"	"	"	6	"	"	17-1422 TB	5(darken)	3-4	4	4	-
"	"	"	4	"	"	18-1321 TB	5(darken)	4	4-5	4	-
"	"	"	2	"	"	17-1322 TB	5(darken)	4	4-5	4-5	-
"	Pb(NO <sub>3</sub> ) <sub>2</sub>	"	8	"	"	18-1325 TB	3-4	3-4	4	4-5	-
"	"	"	6	"	"	18-1320 TB	4	3-4	4	4-5	-
"	"	"	4	"	"	15-1231 TB	4	4	4	4	-
"	"	"	2	"	"	16-1235 TB	4	4	4	4-5	-
"	Al(NO <sub>3</sub> ) <sub>3</sub>	"	8	"	"	16-1325 TP	4	4	4	4	-
"	"	"	6	"	"	17-1446 TB	5	4	4	4	-
"	"	"	4	"	"	17-1353 TB	4-5	4	4	4	-
"	"	"	2	"	"	18-1248 TB	5	4-5	4-5	4-5	-
"	CoCl <sub>2</sub> .6H <sub>2</sub> O	"	8	"	"	13-181616 TB	3	3-4	3-4	4	-
"	"	"	6	"	"	18-1421 TP	3	3-4	4	4	-
"	"	"	4	"	"	17-1340 TP	3-4	4	4	4	-
"	"	"	2	"	"	17-1336 TP	3-4	4	4	4-5	-
"	(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>4</sub> O <sub>24</sub> .4H <sub>2</sub> O	"	8	"	"	18-1421 TB	5(darken)	3-4	4	4	-
"	"	"	6	"	"	19-1436 TB	5(darken)	3-4	4	4-5	-
"	"	"	4	"	"	18-1320 TB	5(darken)	4	4	4	-
"	"	"	2	"	"	18-1326 TB	5(darken)	4	4	4-5	-
"	KAl(SO <sub>4</sub> ) <sub>2</sub> .12H <sub>2</sub> O	"	8	"	"	16-1350 TB	4-5	4	4	4-5	-
"	"	"	6	"	"	16-1448 TB	4	4	4	4	-
"	"	"	4	"	"	16-1454 TB	4	4	4	4-5	-
"	"	"	2	"	"	16-1448 TB	4-5	4	4	4	-
"	MnSO <sub>4</sub>	"	8	"	"	17-1516 TB	3-4	3-4	4	4	-
"	"	"	6	"	"	18-1421 TB	4	3-4	4	4	-
"	"	"	4	"	"	17-1436 TB	4	4	4	4	-
"	"	"	2	"	"	16-1332 TB	4	4	4-5	4-5	-
"	CuCl <sub>2</sub>	"	8	"	"	12-0808 TB	4	3-4	4	4	-
"	"	"	6	"	"	18-1112 TB	4	4	4	4	-
"	"	"	4	"	"	17-1418 TB	3-4	4	4	4	-
"	"	"	2	"	"	17-1322 TB	4	4	4-5	4	-
"	Na <sub>2</sub> CrO <sub>4</sub>	"	8	"	"	18-1436 TB	4	4	4-5	4	-
"	"	"	6	"	"	18-1340 TB	4-5	4	4	4	-
"	"	"	4	"	"	17-1142 TB	4-5	4	4-5	4	-
"	"	"	2	"	"	18-1433 TB	4	4	4	4	-
"	K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	"	8	"	"	18-1433 TB	4	4	4	4	-
"	"	"	6	"	"	18-1330 TB	4	4	4-5	4-5	-
"	"	"	4	"	"	18-1142 TB	4-5	4	4-5	4-5	-
"	"	"	2	"	"	18-1436 TB	4	3	4	4	-
"	Cd(NO <sub>3</sub> ) <sub>2</sub>	"	8	"	"	18-1436 TB	4	3	4	4	-
"	"	"	6	"	"	18-1244 TB	4-5	3	4	4	-
"	"	"	4	"	"	17-1046 TB	4	3-4	4	4	-
"	"	"	2	"	"	16-1054 TB	4-5	4	4	4	-
"	AgNO <sub>3</sub>	"	8	"	"	19-0303 TB	5(darken)	3	3	3	-
"	"	"	6	"	"	19-0608 TB	5(darken)	3	3	3	-
"	"	"	4	"	"	19-0000 TB	5(darken)	4-4	3-4	3	-
"	"	"	2	"	"	19-0903 TB	5(darken)	5	3-4	3-4	-
"	ZnSO <sub>4</sub>	"	8	"	"	18-1447 TB	4	4	4-5	4-5	-
"	"	"	6	"	"	18-1629 TB	4	4	4-5	4-5	-
"	"	"	4	"	"	16-1342 TB	4	4-5	4-5	4-5	-
"	"	"	2	"	"	16-1142 TB	4-5	4-5	5	4-5	-
"	SnCl <sub>2</sub>	"	8	"	"	16-1344 TB	4-5	4-5	4	4-5	-
"	"	"	6	"	"	16-1356 TB	4-5	4-5	4	4-5	-
"	"	"	4	"	"	16-1343 TB	5	4-5	4-5	4-5	-
"	"	"	2	"	"	15-1263 TB	5	4-5	5	5	-
Pre-mord.	Fe <sub>2</sub> SO <sub>4</sub>	"	8	"	"	17-1417 TB	5(darken)	4	4	4	-
"	"	"	6	"	"	18-1321 TB	5(darken)	4	4	4	-
"	"	"	4	"	"	17-1044 TB	5(darken)	4	4	4-5	-
"	"	"	2	"	"	17-1327 TB	4	3-4	3-4	3-4	-

Extraction of Dyestuff From Madder Plant (*Rubia tinctorum L.*)..., A. ÖNAL.,

	<i>Pb(NO<sub>3</sub>)<sub>2</sub></i>	"	8	"	"	18-1130 TB	4	3-4	3-4	3-4	-
"	"	"	6	"	"	17-1422 TB	4	3-4	4	4	-
"	"	"	4	"	"	14-1122 TB	4	4	4	4	-
"	"	"	2	"	"	16-1314 TB	4	4	4	4	-
"	<i>Al(NO<sub>3</sub>)<sub>3</sub></i>	"	8	"	"	15-1263 TB	4-5	4-5	4-5	5	-
"	"	"	6	"	"	16-1135 TB	4-5	4-5	4	5	-
"	"	"	4	"	"	13-1022 TB	4-5	4-5	4-5	5	-
"	"	"	2	"	"	15-1263 TB	4-5	4-5	5	5	-
"	<i>CoCl<sub>2</sub>.6H<sub>2</sub>O</i>	"	8	"	"	14-1122 TB	3	3	4	4	-
"	"	"	6	"	"	16-1336 TB	4	4	4-5	4	-
"	"	"	4	"	"	12-0807 TB	4	3-4	4	4	-
"	"	"	2	"	"	14-1336 TB	4	3-4	4	4	-
"	<i>(NH)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>4H<sub>2</sub>O</i>	"	8	"	"	14-0108 TB	5(darken)	4	4	4	-
"	"	"	6	"	"	14-6305 TB	5(darken)	4	4	4-5	-
"	"	"	4	"	"	18-0521 TB	5(darken)	4	4-5	4	-
"	"	"	2	"	"	17-0517 TB	5(darken)	4	4	4-5	-
"	Urea+NH <sub>3</sub> + Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	"	8	"	"	17-1718 TB	5	4-5	5	5	-
"	<i>MnSO<sub>4</sub></i>	"	8	"	"	16-1343 TB	3-4	4	4	3-4	-
"	"	"	6	"	"	16-1338 TB	3-4	3-4	4	4	-
"	"	"	4	"	"	16-1341 TB	4	4	4	4	-
"	"	"	2	"	"	12-1048 TB	4	4	4	4	-
"	<i>CuCl<sub>2</sub></i>	"	8	"	"	17-1045 TP	3-4	4	4-5	4	-
"	"	"	6	"	"	17-1044 TP	4	4	4-5	4	-
"	"	"	4	"	"	17-1327 TP	4	4	4-5	4	-
"	"	"	2	"	"	17-1340 TP	4-5	4	4-5	4	-
"	<i>Na<sub>2</sub>CrO<sub>4</sub></i>	"	8	"	"	18-1434 TP	4	4	4	4	-
"	"	"	6	"	"	18-1537 TP	4	4	4-5	4	-
"	"	"	4	"	"	18-1433 TP	4	3-4	3-4	4	-
"	"	"	4	"	"	18-1230 TP	4-5	4-5	4-5	4-5	-
"	<i>K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub></i>	"	8	"	"	18-1451 TP	4	4	4-5	4-5	-
"	"	"	6	"	"	18-1443 TP	4	3-4	4	4	-
"	"	"	4	"	"	18-1339 TP	3-4	4	4	4-5	-
"	"	"	2	"	"	18-1441 TP	4-5	4-5	4-5	4-5	-
"	<i>Cd(NO<sub>3</sub>)<sub>2</sub></i>	"	8	"	"	16-1334 TP	4	4	4-4	4-5	-
"	"	"	6	"	"	17-1045 TP	4	4	4	4	-
"	"	"	4	"	"	16-1332 TP	4	4	4	4	-
"	"	"	2	"	"	17-1436 TP	4-5	4-5	4	4-5	-
"	<i>AgNO<sub>3</sub></i>	"	8	"	"	18-1314 TP	5(darken)	3	3-4	4	-
"	"	"	6	"	"	18-1222 TP	5(darken)	3	3	3-4	-
"	"	"	4	"	"	19-1121 TP	5(darken)	3	3	4	-
"	"	"	2	"	"	19-1420 TP	5(darken)	4	4	4	-
"	<i>ZnSO<sub>4</sub></i>	"	8	"	"	17-1929 TP	5	4	4-5	5	-
"	"	"	6	"	"	17-1740 TP	5	4	4-5	5	-
"	"	"	4	"	"	16-1331 TP	4-5	4	4-5	5	-
"	"	"	2	"	"	15-1231 TP	5	4	5	5	-
"	<i>SnCl<sub>2</sub></i>	"	8	"	"	16-1361 TP	4-5	4-5	5	5	-
"	"	"	6	"	"	16-1360 TP	4-5	4-5	5	5	-
"	"	"	4	"	"	16-1362 TP	4-5	4-5	5	5	-
"	"	"	2	"	"	16-1462 TP	5	5	5	5	-
Last Mord.	<i>FeSO<sub>4</sub></i>	"	8	"	"	18-1142 TP	5(darken)	3-4	3-4	3-4	-
"	"	"	6	"	"	17-1340 TP	5(darken)	3-4	3-4	3-4	-
"	"	"	4	"	"	17-1137 TP	5(darken)	4	4	4	-
"	"	"	2	"	"	17-1330 TP	5(darken)	4	4	4	-
"	<i>Pb(NO<sub>3</sub>)<sub>2</sub></i>	"	8	"	"	16-1331 TP	3-4	3-4	3-4	3-4	-
"	"	"	6	"	"	17-1225 TP	3-4	3	4	3-4	-
"	"	"	4	"	"	17-1228 TP	4	4	4	4	-
"	"	"	2	"	"	18-1244 TP	4	4	4	4	-
"	<i>Al(NO<sub>3</sub>)<sub>3</sub></i>	"	8	"	"	14-1224 TP	4-5	4	4	4	-
"	"	"	6	"	"	15-1334 TP	4-5	4	4	4	-
"	"	"	4	"	"	16-1338 TP	4-5	4	4	4	-
"	"	"	2	"	"	16-1317 TP	4-5	4-5	4-5	4-5	-
"	<i>CoCl<sub>2</sub>.6H<sub>2</sub>O</i>	"	8	"	"	16-1708 TP	4	4	4	4	-
"	"	"	6	"	"	17-1511 TP	4	4	4	4-5	-
"	"	"	4	"	"	18-1512 TP	4	4	4	4	-
"	"	"	2	"	"	19-1331 TP	4-5	4	4-5	4-5	-

Extraction of Dyestuff From Madder Plant (*Rubia tinctorum L.*)..., A. ÖNAL.,

"	$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$	"	8	"	"	18-1314 TP	5(darken)	4	4	4	-
"	"	"	6	"	"	18-1451 TP	5(darken)	4	4	4	-
"	"	"	4	"	"	18-1536 TP	5(darken)	4	4	4	-
"	"	"	2	"	"	18-1434 TP	5(darken)	4	4-5	4	-
"	$KAl(SO_4)_2 \cdot 12H_2O$	"	8	"	"	13-1023 TP	4	4	4-5	4	-
"	"	"	6	"	"	14-1231 TP	4	4	4	4	-
"	"	"	4	"	"	15-1242 TP	4	4	4	4	-
"	"	"	2	"	"	16-1257 TP	4-5	4-5	4-5	4-5	-
"	$MnSO_4$	"	8	"	"	15-1607 TP	3	3	3-4	4	-
"	"	"	6	"	"	15-1611 TP	3	3	3-4	4	-
"	"	"	4	"	"	15-1906 TP	4	4	4	4	-
"	"	"	2	"	"	13-2803 TP	4	4-5	4	4	-
"	$CuCl_2$	"	8	"	"	15-0522 TP	4	3-4	4	4	-
"	"	"	6	"	"	15-0318 TP	4	3-4	4	3-4	-
"	"	"	4	"	"	16-0730 TP	3-4	4	4	4	-
"	"	"	2	"	"	17-1045 TP	4	4	4	4	-
"	$Na_2CrO_4$	"	8	"	"	13-1021 TP	4	4	4-5	4	-
"	"	"	6	"	"	14-1230 TP	3-4	4	4-5	4	-
"	"	"	4	"	"	18-1451 TP	4	3-4	3-4	3-4	-
"	"	"	2	"	"	16-1323 TP	4	4	4	4	-
"	$K_2Cr_2O_7$	"	8	"	"	16-1126 TP	3-4	4	4	4	-
"	"	"	6	"	"	15-1132 TP	4	4	4	4	-
"	"	"	4	"	"	17-1036 TP	4	4	4	4	-
"	"	"	2	"	"	16-1432 TP	4	4	4-5	4-5	-
"	$Cd(NO_3)_2$	"	8	"	"	14-1316 TP	3-4	3	3-4	4	-
"	"	"	6	"	"	15-1523 TP	3-4	3	3-4	4	-
"	"	"	4	"	"	15-1327 TP	4	4	4	4	-
"	"	"	2	"	"	16-1439 TP	4	4	4	4	-
"	$AgNO_3$	"	8	"	"	19-1118 TP	5(darken)	2-3	2-3	3	-
"	"	"	6	"	"	19-1217 TP	5(darken)	3	3	3	-
"	"	"	4	"	"	19-1230 TP	5(darken)	3	3	3	-
"	"	"	2	"	"	18-1031 TP	5(darken)	3	3-4	3	-
"	$ZnSO_4$	"	8	"	"	16-1520 TP	4-5	4	4	4	-
"	"	"	6	"	"	17-1518 TP	4-5	4	4-5	4	-
"	"	"	4	"	"	17-1524 TP	4-5	4	4	4	-
"	"	"	2	"	"	15-1515 TP	4-5	4-5	4-5	4-5	-
"	$SnCl_2$	"	8	"	"	14-1128 TP	4-5	4	4	4	-
"	"	"	6	"	"	14-1051 TP	4	4	4	4	-
"	"	"	4	"	"	15-1345 TP	4	4	4	4	-
"	"	"	2	"	"	14-1054 TP	4-5	4	4-5	4	-
"	$NaOH$	"	8	"	"	15-1515 TP	4	3-4	4	4	-
"	"	"	6	"	"	15-1523 TP	4	4	4	4	-
"	$CH_3COOH$	"	4	"	"	14-1311 TP	4-5	4	4	4-5	-
"	"	"	2	"	"	16-1235 TP	4-5	4	4	4-5	-

\* Colour codes were determined using the "Pantone Textile Color Guide"

**Table 2.** Results of fast analyses and colour codes of dyed feathered-leathers

Dyeing method	Mordant	Flotte ratio	pH	Bath temp. (°C)	Dyeing period (hr)	Colour code*	light fast	Croc- king	fast dry	Washing fast	Hypo-chloride fast
Together mordant.	$FeSO_4$	0.01	4.5	35-40	1	13-1014 TB	5(darken)	4	4-5	4-5	-
"	"	"	2	"	"	13-1011 TB	5(darken)	4	4-5	4-5	-
"	$SnCl_2$	"	4.5	"	"	14-1225 TB	4-5	4	4-5	5	-
"	"	"	2	"	"	14-1224 TB	4-5	4-5	5	5	-
"	$AgNO_3$	"	4.5	"	"	17-1328 TB	5(darken)	3-4	3-4	3	-
"	"	"	2	"	"	16-1432 TB	5(darken)	3-4	3-4	3-4	-
"	$K_2Cr_2O_7$	"	4.5	"	"	16-0954 TB	5(darken)	4-5	4	4-5	-
"	"	"	2	"	"	16-0948 TB	5(darken)	4-5	4	4-5	-
"	$KAl(SO_4)_2 \cdot 12H_2O$	"	4.5	"	"	13-0917 TB	4-5	4-5	4-5	4-5	-
"	"	"	2	"	"	13-1018 TB	4-5	4-5	4-5	4-5	-
"	$MnSO_4$	"	4.5	"	"	14-1220 TB	4	4	4	4	-
"	"	"	2	"	"	13-1030 TB	4	4	4-5	4	-
"	$NiCl_2$	"	4.5	"	"	13-1014 TB	3-4	4	4	4	-
"	"	"	2	"	"	13-1027 TB	4	4-5	4	4	-

Extraction of Dyestuff From Madder Plant (*Rubia tinctorum L*)..., A. ÖNAL.,

"	<i>Al(NO<sub>3</sub>)<sub>3</sub></i>	"	4.5	"	"	13-1030 TB	4-5	4	4-5	5	-
"	"	"	2	"	"	13-0922 TB	4-5	4-5	4-5	4-5	-
"	<i>CoCl<sub>2</sub>.6H<sub>2</sub>O</i>	"	4.5	"	"	15-1529 TB	4				-
"	"	"	2	"	"	16-1342 TB	3-4	4	4	4	-
"	<i>(NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>.4H<sub>2</sub>O</i>	"	4.5	"	"	16-1344 TB	5(darken)	4	4-5	4-5	-
"	"	"	2	"	"	16-1329 TB	5(darken)	4	4-5	4-5	-
Pre mordant.	<i>FeSO<sub>4</sub></i>	"	4.5	"	"	14-1313 TB	5(darken)	4	4-5	4-5	-
"	"	"	2	"	"	13-1017 TB	5(darken)	4-5	4-5	5	-
"	<i>SnCl<sub>2</sub></i>	"	4.5	"	"	15-1334 TB	5	5	5	5	-
"	"	"	2	"	"	14-1224 TB	5	5	5	5	-
"	<i>AgNO<sub>3</sub></i>	"	4.5	"	"		5(darken)	4	3-4	3	-
"	"	"	2	"	"		5(darken)	3-4	4	3-4	-
"	<i>K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub></i>	"	4.5	"	"	16-1332 TB	5(darken)	4	4-5	4	-
"	"	"	2	"	"	15-1322 TB	5(darken)	4	4-5	4	-
"	<i>KAl(SO<sub>4</sub>)<sub>2</sub>.12H<sub>2</sub>O</i>	"	4.5	"	"	15-1318 TB	4-5	4	4-5	4	-
"	"	"	2	"	"	14-1113 TB	4-5	4	4	5	-
"	<i>MnSO<sub>4</sub></i>	"	4.5	"	"	14-1220 TB	4	4	4	4	-
"	"	"	2	"	"	14-1311 TB	4	4	4-5	4-5	-
"	<i>NiCl<sub>2</sub></i>	"	4.5	"	"	15-1318 TB	4	4	4	4	-
"	"	"	2	"	"	16-1337 TB	4	4	4	4	-
"	<i>Al(NO<sub>3</sub>)<sub>3</sub></i>	"	4.5	"	"	16-1340 TB	5	4	4-5	4-5	-
"	"	"	2	"	"	14-1225 TB	5	4-5	5	5	-
"	<i>CoCl<sub>2</sub>.6H<sub>2</sub>O</i>	"	4.5	"	"	14-1220 TB	4	4	4	4	-
"	"	"	2	"	"	13-1027 TB	4	4	4	4	-
"	<i>(NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>.4H<sub>2</sub>O</i>	"	4.5	"	"	17-1755 TB	5(darken)	4	4	4-5	-
"	"	"	2	"	"	17-1736 TB	5(darken)	4	4	4-5	-

\* Colour codes were determined using the "Pantone Textile Color Guide"

**Table 3.** Results of fast analyses and colour codes of dyed cotton

Dyeing method	Mordant	Flotte ratio	pH	Bath temp. (°C)	Dyeing period (hr)	Colour code*	light fast	Croc- king moist	fasts dry	Washing fast	Hypo-chloride fast
Together mordant.	<i>FeSO<sub>4</sub></i>	"	7	90	1	12-1007 TB	5(darken)	4	4-5	4	-
"	"	"	4	"	"	13-3803 TB	5	4	4-5	4	-
"	<i>SnCl<sub>2</sub></i>	"	7	"	"	12-0817 TB	4	4	5	4	-
"	"	"	4	"	"	12-0722 TB	4	4	5	4-5	-
"	<i>AgNO<sub>3</sub></i>	"	7	"	"	19-1230 TB	5(darken)	4	4	3-4	-
"	"	"	4	"	"	13-1403 TB	5(darken)	4	4	3-4	-
"	<i>K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub></i>	"	7	"	"	12-1305 TB	5(darken)	4-5	5	4	-
"	"	"	4	"	"	12-1304 TB	5(darken)	4	4-5	4	-
"	<i>KAl(SO<sub>4</sub>)<sub>2</sub>.12H<sub>2</sub>O</i>	"	7	"	"	12-1009 TB	4-5	4	4-5	4	-
"	"	"	4	"	"	12-0913 TB	4-5	4	4-5	4	-
"	<i>MnSO<sub>4</sub></i>	"	7	"	"	14-1509 TB	4	4	4-5	4	-
"	"	"	4	"	"	12-1107 TB	4	4	4	4	-
"	<i>NiCl<sub>2</sub></i>	"	7	"	"	11-2511 TB	4	4	4	4	-
"	"	"	4	"	"	12-2905 TB	4	4	4	4	-
"	<i>Al(NO<sub>3</sub>)<sub>3</sub></i>	"	7	"	"	11-1306 TB	4-5	4	5	4-5	-
"	"	"	4	"	"	12-1304 TB	5	4	5	4-5	-
"	<i>CoCl<sub>2</sub>.6H<sub>2</sub>O</i>	"	7	"	"	12-1305 TB	4	4	4	4-5	-
"	"	"	4	"	"	13-1408 TB	4	4	4	4	-
"	<i>(NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>.4H<sub>2</sub>O</i>	"	7	"	"	12-0915 TB	5(darken)	4	5	4-5	-
"	"	"	4	"	"	13-1022 TB	5(darken)	4	5	4-5	-
Pre mordant.	<i>FeSO<sub>4</sub></i>	"	7	"	"	12-1404 TB	5(darken)	4	5	4	-
"	"	"	4	"	"	13-3803 TB	5(darken)	4	5	4-5	-
"	<i>SnCl<sub>2</sub></i>	"	7	"	"	12-0911 TB	4-5	4	5	4-5	-
"	"	"	4	"	"	13-0941 TB	4-5	4	5	4-5	-
"	<i>AgNO<sub>3</sub></i>	"	7	"	"	19-1118 TB	5(darken)	3-4	4	4	-
"	"	"	4	"	"	19-1217 TB	5(darken)	3-4	4	4	-
"	<i>K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub></i>	"	7	"	"	11-2309 TB	4-5	4	4-5	4	-
"	"	"	4	"	"	11-2409 TB	4-5	4	4-5	4	-
"	<i>KAl(SO<sub>4</sub>)<sub>2</sub>.12H<sub>2</sub>O</i>	"	7	"	"	12-0915 TB	5	4	4	4	-
"	"	"	4	"	"	13-1021 TB	5	4	4	4	-

Extraction of Dyestuff From Madder Plant (*Rubia tinctorum L.*)..., A. ÖNAL.,

"	<i>MnSO<sub>4</sub></i>	"	7	"	"	13-2004 TB	4	4	5	5	-
"	"	"	4	"	"	12-1304 TB	4	4	4-5	4-5	-
"	<i>NiCl<sub>2</sub></i>	"	7	"	"	16-1520 TB	4	4	4-5	4	-
"	"	"	4	"	"	14-1312 TB	4	4	4-5	4	-
"	<i>Al(NO<sub>3</sub>)<sub>3</sub></i>	"	7	"	"	12-0917 TB	5	4	5	4-5	-
"	"	"	4	"	"	14-1219 TB	5	4	4-5	4-5	-
"	<i>CoCl<sub>2</sub>.6H<sub>2</sub>O</i>	"	7	"	"	13-1406 TB	4	4	4-5	4	-
"	"	"	4	"	"	12-0822 TB	4-5	4	4	4	-
"	<i>(NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>.4H<sub>2</sub>O</i>	"	7	"	"	12-1009 TB	5(darken)	4	4	4	-
"	"	"	4	"	"	12-0804 TB	5(darken)	4	4-5	4	-
Unmordant	-	"	8	"	"	16-1626 TB	4-5	4	4	4	-

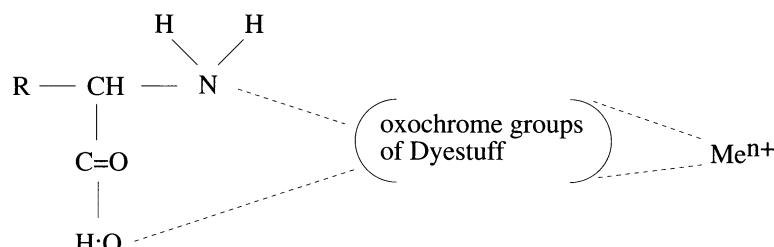
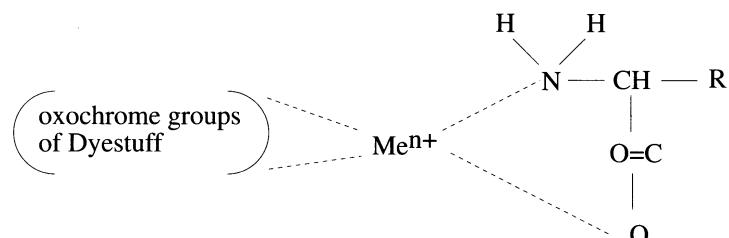
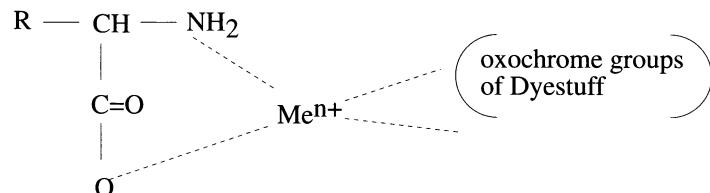
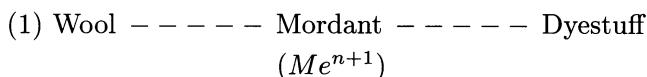
\* Colour codes were determined using the "Pantone Textile Color Guide"

Molecules of wool consist of amino acid units. Proteins are formed by amion acids which have free amino and carboxyl groups. Therefore, wool has an amphoteric formation<sup>6</sup>. In the dyeing of wool, intermolecular hydrogen bonding occurs between the dyestuff and the amino group of wool (scheme 1).



**Scheme 1.**

Mechanism of Pre-mordantation (1), together mordantation (2) last mordantation (3) can be considered as given in scheme 2.



**Scheme 2.**

One hundred and sixty-eight colours and colour tones were obtained from the dyeing the woollen strips. Only dark blue couldn't be obtained. Bright and high fast colours were obtained from pre mordantation and together mordantation methods, respectively. Dark colours were obtained at pH 8 and 6; pale colours were obtained at pH 4 and 2. Pre mordantation was determined as the best dyeing method of all the dyeing processes.

In addition a new mordant mixture was found (50 ml, 3%  $NH_3$  + 0.2 g of urea+ 0.3 g of  $Na_2C_2O_4$ ) for the dyeing of wool in this study unfading colours were obtained using this mordant mixture by the pre mordantation method. Dyed wool did not fade in the strong fadings such as  $NaOCl$  or sunbeam. In this dyeing, the pre mordantation time was applied 24 hours at 20°C and the dyeing time was applied 1 hour at 80°C.

In the dyeing of feathered-leather at the higher pH values (6,7,8) didn't result in chemical bonding because of the low temperature. Last mordantation did not give a positive result because the temperature of the dye-bath must be between 35-40° during the dyeing process. Leather shrinks at higher temperatures (> 40°) and the dyeing process doesn't give a good result at lower temperatures (< 35°). Desirable results were obtained in the dyeing of feathered-leather. This is related to the reather industry.

Cotton has a different structure and properties with respect to wool and leather. It is composed of glucoside units. It can give coordinative and hydrogen bonding with mordant or dyestuff molecules. Cotton is dyed without using any mordant. When mordant is used, lots of colour tones except dark blue can be obtained. The dyeing mechanism is given below (scheme 3).

Cotton ..... Mordant ..... Dyestuff

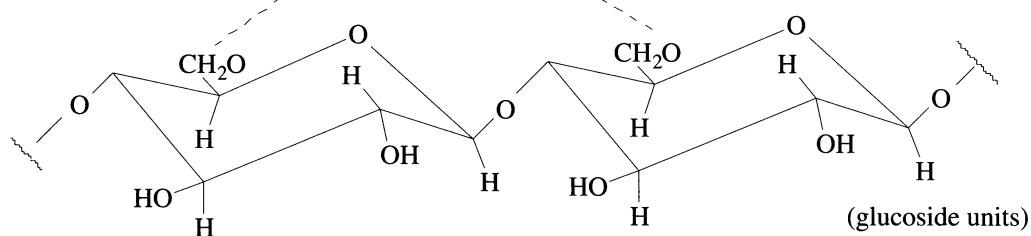
$(Me^{n+})$

Cotton ..... Mordant ..... Dyestuff

$Me^{n+}$

Oxochrome groups  
of Dyestuff

$Me^{n+}$



Scheme 3.

In the dyeing of cotton, the most suitable pH range was determined as 7-2. Colours change according to the pH, mordant and dyeing time.

## Conclusion

1. In this study a lot of colour tones were obtained.

2. There is no problem in finding colour tones such as red, yellow, green, brown, pink and grey.
3. This study is cheap very reliable and repeatable.
4. The colour duration period could be up to 400 years<sup>8</sup> (for carpets and kilims)
5. Studies on wool, leather and cotton are new and important for the leather and textile industries.

## Acknowledgement

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## References

1. M. Ali Kağıtçı, “Boya” İktisat ve Ticaret Ansiklopedisi 3. bsk. s. 90, 1948.
2. T. Baytop, “Farmakognozi” vol: 2, pp. 288, Dilek Matb. İstanbul, 1983.
3. T. C. Sanayi ve Ticaret Bakanlığı, Küçük Sanatlar Sanayi Bölgeleri ve Siteleri Genel Müdürlüğü, “Bitkilerden Elde edilen Boyalarla Yün Liflerinin Boyanması” s. 62. 1991.
4. A. Önal, M. Kepez, **T. J. Chem.**, C13, S3, 299-302 (1989).
5. A. G. Perkin et al. **J. Chem. Soc.**, 67, 497-502, (1985).
6. Pantone “Textile Color Guide”, Pantone Inc., New Jersey, 1992.
7. R. J. Fessenden and J. S. Fessenden, “Organic Chemistry,” 4 th. pp. 1029, 1061, 1087, Cole Publishing Company, California, 1990.
8. Ü. Eyüboğlu, ver Ark., “Doğa Boyalarla Yün Boyama”, Uygulamalı Eğitim Vakfı Yayınları, 12, (1983).