# INVESTIGATION OF NUTGALL AND SOME NATURAL DYES WITH MORDANTS COTTON DYEING AND FASTNESS LEVEL IN THE CONTEXT OF THE ECOLOGICAL TEXTILE PRODUCTION

**Fatma Nur Başaran[1], Hakan Sarıkaya[2]**

[1] Assoc. Prof., Textile Design and Production Department, Faculty of Art and Design, Gazi University, Ankara
[2] Expert, Textile Design Department, Institute of Fine Arts, Gazi University, Ankara

## ABSTRACT

Humans have been using plant-based dyestuff for centuries as the main provider of dyes for industrial products such as textiles, food, leather, etc. Specifically in Turkey, such plants have been used for the dyeing of the fibre and yarn of cotton, wool and silk used in handicraft products, such as carpets, rugs, fabrics, etc. With the discovery of synthetic dyestuffs in the mid-19th century, natural dyes, and thus, natural dyeing, gradually lost importance, although today, plant-based dyestuffs are again gaining popularity as a result of the rising popularity of the natural and the sustainable concept. The purpose of the present study is to investigate the dyeing effect of solutions obtained from gallnut dyestuffs and mordants from whey, yeast, lye and mushroom extract, with the intention being to identify light, crock and wash fastness levels and examine their usability in today's textile sector. Dyeing cotton fabric samples with gallnut dyestuffs and natural mordants has allowed some important conclusions to be drawn with respect to human and environmental health.

**Keywords:** Gallnut dyestuff, Light Fastness, Crock Fastness, Wash Fastness, Natural mordants

## 1 INTRODUCTION

Natural dyestuff and natural dyeing is as least as old as the history of textiles; and it is known that the natural dyeing of textile fibres started in India and Mesopotamia in the 2000s B.C. (1).

A dyestuff analysis of the red colour of the Pazırık carpet, the oldest known carpet in history, dated to the 5th century B.C., and the felt samples found in the same castle, found that Polish cochineal *(Porphyrophora polonica)* and madder were used in dyeing.(2)

It is known that the Chinese-dyed silk fabrics using dyes called plant indigo and Chinese green date back to the period before Christ; and fabrics found in excavations in Egypt show that the Egyptians also used indigo and mordants in the same period. In 1500 A.D., dyeing knowledge and used transferred from the East to the West, arriving along the coasts of the Mediterranean where the skill developed further. The first known book on dyeing was published in Venice, Italy. (3)

At these times and in the periods that followed, tests were made in many regions of the world with different auxiliary substances to identify natural dye-bearing plants, and attempts were made to achieve various colour tones.

Analysing the development of natural dyeing among the Turkish tribes, it is possible to say that dyestuff plants have existed and been cultivated in Central Asia and Anatolia throughout history. The caravan routes and the

caravan trade were always controlled by the Turks, which ensured that high amounts of dyestuff plants were grown and exported to Europe. In addition, the export of alum from regions rich in alum beds had also been the monopoly of Turks until alum was discovered in Europe in 1462.(4)

In the Ottoman period, cities such as Bursa, İstanbul, Edirne, Tokat, Kayseri and Konya were important centres of the art of dyeing,(5) although natural dyeing using traditional methods and materials has been carried out in all regions throughout history in the family or in small enterprises.

Today, madder and buckthorn can be found all over Turkey in madder and buckthorn fields and as weeds lining the gutters. (6)

Another dyestuff plant, saffron, has been cultivated in Anatolia for 3,500 years, and it is known that saffron cultivated in Tokat was exported even to India. (7)

Gallnut, which has long held an important place among natural dyestuffs in the world, has been in use in Turkey since ancient times. Gallnut shellac is known to have been used in various applications, especially in dyeing and tanning, since the Sumerian era, although manuscripts written with gallnut ink may become yellowed over time or the ink may burn into the paper. Gallnut is collected for dyeing and for tannin. In Turkey, the collected gallnut is ground and then boiled together with the wool, and the obtained colour is between dirty yellow and brown. This method of dyeing is known as "*tetre*" in the local language, and the *tetre* dyed wool is mordanted using ferric alum or iron mud to obtain a black colour. The black colouring in Turkish carpets and rugs is created in this way.(8)

In parallel to the development of natural dyeing, there are also a variety of substances used as mordant in Turkey.

Mordants can be classified into two types, being chemical and natural mordants. Chemical mordants are metal salts that dissolve in water, with commonly used chemical mordants including alum (KAI(SO4)2-12H2O), ferrous sulphate (Fe SO4-7 H2O), copper sulphate (CuSO4-5H2O) and potassium bichromate, all of which are used locally with different names. The natural mordants cited in literature include substances such as salt, salt of lemon, cattle urine, vinegar, sour grape, citrus juice, washing soda, mole milk, bread yeast, ash, clay, pelite, moss, dried yogurt and lime. All of these natural mordants have at some time been used in different proportions for dyeing in Anatolia. (9)

Ensuring these mordant substances are used together with natural dyes raises their economic value.

Considering the importance of natural dyestuffs for producers seeking to produce ecological textiles, identifying the usability of dyestuffs obtained from gallnut, known in literature for its dyeing characteristics, and the mordant characteristics of such substances as whey, mushroom extract, lye and yeast, and detecting the fastness values and standards on cotton fabrics, will be beneficial to both producers and researchers who use such dyestuffs and mordants.

Studies of natural mordants in Turkey are limited, and there has also been no research into the usability of substances such as whey, mushroom extract, lye and yeast as a mordant. It is expected that the present study will contribute to filling this gap in knowledge, and serve as a guide for future studies.

## 2 DISTRIBUTION OF GALLNUT AND FIELDS OF USE IN TURKEY

*Quercus infectoria* (Cyprus oak) is a tree with a wide top that can grow to heights of 12 m and a diameters of 80 cm. It is found most predominantly in Turkey in the Marmara and the Black Sea regions. The Cyprus oak does not always shed all its leaves in the autumn, as in years when the winter is not excessively harsh, the leaves continue falling until spring. In June-July, the female gallnut bee lays its eggs on the buds of the Cyprus oak, and a nutritional layer, galls, rich in fatty substances, sugar and protein, and surrounded by a thin membrane, forms around the eggs. This nutritional layer is protected by a hard outer layer, and these two layers make up the gallnut, which is round and has a diameter of 1.5–2 cm. Since this mostly occurs on this oak, the species is called the Cyprus oak. (10)

The gallnuts from this plant are very valuable, containing high amounts of tannin. The gallnuts formed on other oak species lack a similar economical value since they contain lesser proportions of tannin, and so are mixed with gallnuts from *Q. infectoria* Oliv. and traded. It is also an important raw material used for the creation of tannic acid in the chemical sector. Since it contains 60–70 percent tanning material, it is used as a fixator in the manufacture of dye, ink and indigo, as well as in the secondary tanning of leather, in which ground gallnut is spread over leather tanned using sumac leaves, which have a tanning property. In the pharmacological sector, tannic acid obtained from tannin is in common use in veterinary practices in the composition of anti-

diarrheic drugs, and is used, although rarely, as a coagulant to stem bleeding. Aside from being used in animal husbandry, it is also used in the dyeing of fabrics and wool.(11)

## 3 NATURAL MORDANT SUBSTANCES USED IN THE STUDY

### 3.1 Lye

Ash, obtained from burning plants and in frequent use as a dye, is an alkaline substance that contains high proportions of iron, as well as magnesium, manganese, copper and zinc. Iron and copper are the active substances in the mordant effect. It is used in the bleaching of wool and cotton in place of soap, and especially in the painting of earthenware jars. (12)

### 3.2 Whey

Whey accumulates at the bottom of buckets during cheese production, and has been in use throughout historic in Asian countries and by the people of the Mediterranean. It finds use as a mordant and as a beverage as a result of its calcium content.

### 3.3 Yeast

Yeast is one of the most important microorganisms used by the human race throughout its history. In the past, it has found use in natural dyeing, containing such metals as cobalt, copper, iron, magnesium, manganese, nickel, zinc and chromium. Today, it is used in natural dyeing due to its high mordant effect, which is attributed to its iron, chromium and copper content.

### 3.4 Mushroom

Turkey is rich in natural mushrooms due to its endemic flora and climatic conditions, which has resulted in the use of mushrooms in various fields. Mushrooms, which contain calcium, iron, phosphorous, potassium and copper minerals, has found use in natural dyeing for many years. The essential minerals contained within mushrooms that have a fixative effect, ensuring the dye attaches to the fibre, are iron and copper.

## 4 METHOD

In this study, the plant dyestuff used was the gallnut obtained from the Kale Naturel establishment, which is a producer of plant dyestuffs in Turkey. In the trials made using this dyestuff, whey, mushroom extract, lye and yeast were used as natural mordants. Different dye recipes were prepared for the dyestuff, with each mordant and dyeing trial conducted in the dye laboratory of Berteks Tekstil. The wash, crock (wet, dry) and light fastness tests of the trials were conducted and assessed in accordance with TS ISO standards.

## 5 PREPARATION OF DYE RECIPES

The dye recipes prepared for the dyeing cotton fabric using lye, yeast, whey and mushroom extract were applied in accordance with pre-mordanting and simultaneous mordanting methods, and fabric specimens were obtained. Since the amounts of dyestuff and mordant used in all dyeing methods are the same, both methods were included in the dyeing method section in the recipe. However, in pre-mordanting, the cotton fabric was treated with only mordants at 90°C for 60 minutes, after which the dyeing process was carried out.

Table 1 Cotton fabric dyeing prescription, prepared using gallnut dyestuff and mordants

| | | |
|---|---|---|
| **Dyestuff** | **Type** | **Gallnut** |
| | **Amount** | 5 g/lt |
| **Fabric** | **Type** | Cotton |
| | **Weight** | 5 g |
| **Mordants** | **Type** | Mushroom extract, lye, whey, yeast |
| | **Amount** | 20 g/lt |
| **Dyeing** | **Temperature** | 90°C |
| | **Time** | 60 min |
| | **Method** | Pre-mordanting and simultaneous mordanting |

## 6 PREPARATION OF FABRIC SPECIMENS AND SOLUTION

Each cotton fabric specimen weighed 6 g, and a total of 15 fabric specimens were prepared, ready for dyeing.

In the dyeing trials with simultaneous mordanting, four solutions were prepared using different mordants, alongside the gallnut dyestuff. For the preparation of the solutions, 100 ml Erlenmeyer flasks were used, and the raw materials amounts put into these flasks were as follows:

- 1st Solution: 20 g/lt lye and 5 g/lt gallnut dyestuff
- 2nd Solution: 20 g/lt yeast and 5 g/lt gallnut dyestuff
- 3rd Solution: 20 g/lt whey and 5 g/lt gallnut dyestuff
- 4th Solution: 20 g/lt mushroom extract and 5 g/lt gallnut dyestuff

In the pre-mordanting process, on the other hand, each specimen was treated with only mordants at 90°C for 60 minutes, after which the dyeing process was carried out. Each prepared solution was stirred for 30 minutes using a magnetic stirrer and the pH values were measured. These measurements are given in the dyeing recipes.

Figure 1. Solution and magnetic stirrer

## 7 DYEING THE FABRIC SPECIMENS

After checking the cleanliness of the thermal dyeing machine, the dyeing containers are made ready. The fabric specimens are put into the prepared test containers, and 100 ml of each aqueous solution prepared for

dyeing is placed into the test containers. The test containers are placed in the machine and treated at 90°C for 60 minutes. After the dyeing process is completed, the dyed cotton fabrics are taken out and ventilated for some time. Then, they are rinsed with cold water, spread onto the drying paper and placed into the drier. The specimens are treated for 15–20 minutes at 60°C, after which they are taken out and ventilated.

Figure 2. Dyeing Machine


Figure 3. Drier

Figure 4. Dyed fabric specimen

## 8 RESULTS OF THE FASTNESS ANALYSIS OF THE FABRIC SPECIMENS

Table 2. Results of the fastness analysis of the pre-mordanted fabric specimens

| Mordant | Flotte Ratio | Light Fastness | Crock Fastness | | Wash Fastness |
|---|---|---|---|---|---|
| | | | Wet | Dry | |
| **Lye** | 1/10 | 5 | 4 | 5 | 4 |
| **Yeast** | 1/10 | 3 | 2 | 4 | 4 |
| **Whey** | 1/10 | 2 | 5 | 3 | 4 |
| **Mushroom Extract** | 1/10 | 3 | 4 | 4 | 4 |

From Table 2 it can be seen that with a 1/10 flotte ratio, the light fastness of the cotton fabric specimens dyed with pre-mordanting is good (5) with lye mordant; low (2) with whey mordant; and medium (3) with mushroom extract mordant, in accordance with the blue scale. From this it can be concluded that the light fastness obtained when dyeing cotton fabric with gallnut dyestuff is at a medium level, except when using whey.

Examining the values concerning the wet crock fastness made with the grey scale, the fastness results of dyeing obtained were rather good (4) with lye and mushroom extract; medium (2) with yeast mordant; and very good (5) with whey. These values point to a good level of wet crock fastness when dyeing cotton fabrics with gallnut dyestuff, except when using the yeast mordant.

Examining the values concerning dry crock fastness made with the grey scale, the fastness results of dyeing obtained were rather good (4) with yeast and mushroom extract; good (3) with whey; and very good (5) with lye. From these values it can be understood that dry crock fastness in dyeing cotton fabrics with gallnut dyestuff is at a good level.

Examining the values concerning wash fastness, the fastness results of dyeing obtained with lye, yeast, whey and mushroom extract were rather good (4) according to the grey scale, pointing to an overall good level of wash fastness in the dyeing of cotton fabrics with gallnut dyestuff.

Table 3. Results of the fastness analysis of simultaneously mordanted fabric specimens

| Mordant | Flotte Ratio | Light Fastness | Crock Fastness | | Wash Fastness |
|---|---|---|---|---|---|
| | | | Wet | Dry | |
| **Lye** | 1/10 | 4 | 2 | 4 | 4 |
| **Yeast** | 1/10 | 2 | 4 | 5 | 4 |
| **Whey** | 1/10 | 2 | 4 | 5 | 4 |
| **Mushroom Extract** | 1/10 | 4 | 3 | 4 | 4 |

From Table 3 it can be seen that with a 1/10 flotte ratio, the light fastness with the grey scale of the cotton fabric specimens dyed with simultaneous mordanting is rather good (4) with lye mordant; low (2) with yeast and whey mordants; and rather good (4) with mushroom extract. These values indicate that the light fastness obtained when dyeing cotton fabric with gallnut dyestuff is at a rather good level, except when dyeing with yeast and whey.

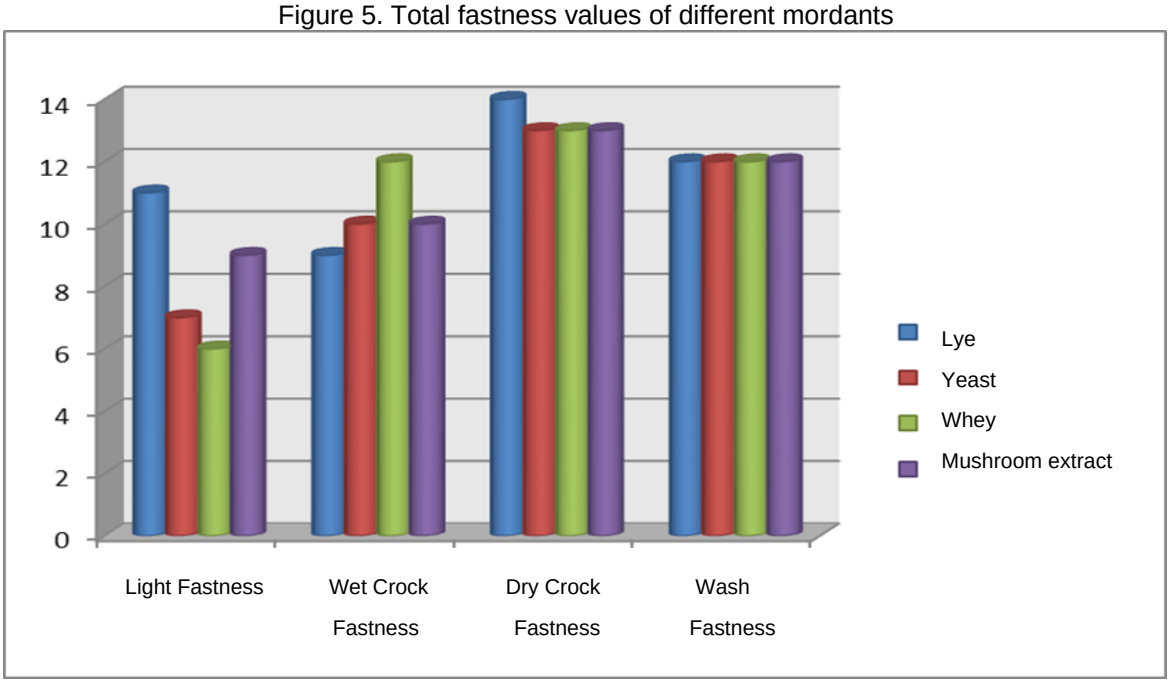
Examining the values concerning wet crock fastness made with the grey scale, the fastness results of dyeing obtained were rather good (4) with yeast and whey mordants, medium (2) with lye, and good (3) with mushroom

extract. According to these values, it is seen that the wet crock fastness in dyeing cotton fabrics with gallnut dyestuff are at a good level, except for the dyeing with lye mordant.

Examining the values of dry crock fastness made with the grey scale, the fastness results of dyeing obtained were rather good (4) with lye and mushroom extract; and very good (5) with yeast and whey mordant. Based on these values, dry crock fastness in dyeing cotton fabrics with gallnut dyestuff can be said to be at a rather good level.

Examining the values concerning wash fastness, the fastness results of dyeing obtained with lye, yeast, whey and mushroom extract were rather good (4) according to the grey scale, indicating that the wash fastness when dyeing cotton fabrics with gallnut dyestuff is at a rather good level.

Figure 5. Total fastness values of different mordants




## 9 CONCLUSION

This study is the first step in the use of gallnut, the dyeing characteristics of which have been well documented in literature, as a dyestuff in the textile sector, in the investigation of the dyeing effects and fastness on different fabrics under industrial conditions and in the transfer of natural dyeing processes to the commercial sector. It can be considered a pre-study of dyestuff production from other plants with dyeing properties.

Within the scope of the study, it was concluded that the light fastness obtained when dyeing cotton fabric specimens with pre-mordanting using lye, yeast, whey and mushroom extract is at a medium level, except for whey. It can be said that for the wet and dry crock fastness values, the results were at a good level with all of the mordants used; although the wet crock fastness of the yeast mordant was lower than the others. It was determined that wash fastness is high in all dyeing processes (Table 2).

It can be seen that when using simultaneous mordanting, the light fastness of the cotton fabric specimens was at a rather good level, except for whey and yeast; and for wet and dry crock fastness values, the results were at a good level with all the mordants used, although the wet crock fastness of the lye mordant was found to be lower than the others. It was determined that wash fastness is high in all dyeing processes (Table 3).

There are 6.5 million hectares of quercus forest in Turkey, with the widest distribution found in the Black Sea and Marmara regions. In this respect, there is no problem in sourcing the gallnut raw material as a dyestuff, which reduces production costs.

In recent years, preferences for ecological products have increased based on health and user-friendliness concerns. Within this context, the importance of textile products produced with natural fibres, dyestuff and natural mordants is increasing. The extension of such studies will no doubt benefit both the manufacturer and consumer, and consequently, the Turkish economy, with respect to production costs.

## NOTES

1. (Karadağ, 2007, p. 21-32).
2. (Şanlı ve Arlı, 2007, p. 55-78).
3. (Öztürk, 1999, p. 16).
4. (Öztürk, 1999, p. 16).
5. (Özbel, 1972, p. 11).
6. (Eşberk, 1947, p. 10-11).
7. (Enez, 1987, p. 9).
8. (OGM, 2014).
9. (Önal ve Kepez 1998, p. 15).
10. (Atalay, 1994, p. 45).
11. (OGM, 2014).
12. (Eşberk, 1947, p. 4).

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