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Optimization of the Dyeing Conditions for Wool Fiber with Natural Indigo Using the Argan's Pulp

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Abstract

Herein we report the optimization of the natural dyeing conditions for wool with indigo extracted from the *Indigofera* plant grown in the south - east of Morocco. We are interested in the evaluation of the reduction power of argan's pulp, the variation of the alkaline pH using natural calcium hydroxide and the dyeing duration. Based on spectroscopic analysis, we came to establish an optimal recipe that was easily reproduced.

1. Introduction

Indigo was the first extract used in natural dyeing. The chemical synthesis of Indigo by Adolph Von Baeyer in 1883 has extended the use of indigo as the favored blue used in dyeing [1]. Since it's a non - polluting dye extracted from a natural source that gives deep and solid coloration, it is interesting to think about its reintegration, which should be preceded by studying the environmental impact (when farming the indigo plant), the importance of natural - goods markets and the economic consequence for the countries producing the natural indigo. The total production of natural indigo today cannot be accurately calculated with the largest portion used by local Indian Pakistani weavers. However, natural indigo is exported in small but increasing amounts from India, Mexico, El Salvador and Pakistan to Europe and America, where it is used mainly by hobby dyers [2].

No indigo plant contains the chemical substance indigo, only the preliminary stages thereof. These are, as it were, the two halves of the indigo molecule, but they are not free; rather they are bonded to another molecule, usually glucose. One such preliminary stage is indicant. In the leaves of the indigo plant, there are enzymes that can split the sugar molecule and release the indoxyl (Figure 1). They usually become active in crushed leaves—but not always—with the aid of bacteria that are apparently omnipresent. If these are involved in the process, one speaks of fermentation. This requires many hours or even days [2, 10].

There are many species of plants that contain the preliminary stages of indigo, but only a few of those with the highest content of the preliminary stages of dyestuff are used for dyeing. These are the varieties of the *Indigofera* family, the most important of which is *Indigofera tinctoria*. This plant is probably native to India, but because it can be easily cultivated, it has become widely distributed; under the Arabs, it went as far as Moorish Spain [2]. Furthermore, there are other Indigo plants, such as the varieties of the

brassica family, the most important of which is *Isatis tinctoria*. At first, it was used and cultivated by the Greek and Roman people, and still used by successive civilizations until the appearance of the *Indigofera* plant and synthetic indigo. Actually, some countries in Europe still cultivate and use this plant [3, 4].

The extraction and the fabrication of indigo can be realized by either the fermentation of the dried leaves of the indigo plant during a long time (few days to 3 months, depending on the species and the origin of the indigo plant) [3, 4], or by maceration of the leaves in warm water for 40min, followed by filtration and oxygenation [2, 5, 6]. The obtained extract was a blue powder that was directly used in the dyeing process. Indeed, this last method was used in the present work.

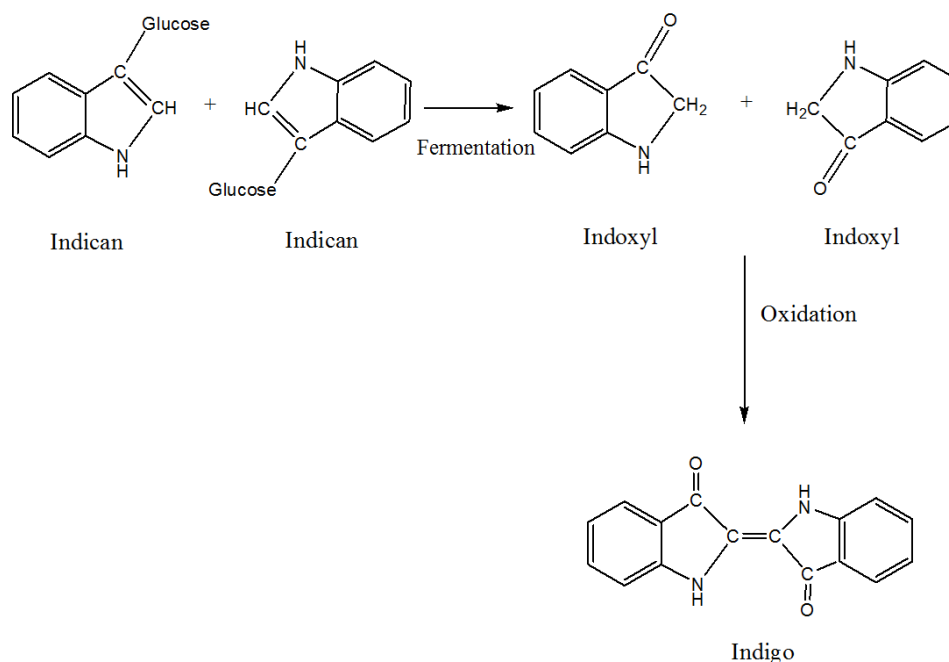


Figure 1. Creation process of indigo from the preliminary stage.

2. Experimental

2.1. Material

Features of wool fiber

The wool fiber used was provided from the Boujaâd city region of Morocco. White fleece was compacted and homogenized to a medium weight of fleece 1. 5–3kg and the fineness of fiber was 50–60 using the Bradford scale.

Natural dye

The dye used in the present study was from a natural source and was extracted from the *indigofera* plant, which grows in the south - east of Morocco [1, 8]. The extraction method was based on the maceration of 200g of dried leaves in water at 40°C for 40min, which transforms the indican into indoxyl that can be oxidized to form indigo. The proportion of pure indigo in this extract was 20 g.

Argan's pulp

In order to master the different factors in the dyeing process of wool fiber with natural indigo, we have to study these factors. Since it's so hard to control all the factors involved in the dye bath, we will study some of the important factors among them. In this work, we are interested in three principal factors: the concentration of the natural reducer, the pH of the solution and dyeing time. The measurement of the effect of all these factors was fulfilled depending on the quantity of the dyestuff absorption.

So, we started by varying the reducer concentration in order to assess its reducing strength. We used argan's pulp as a local plentiful product. This natural source was composed of 20% reducer sugar, 13% cellulose, 6% protein, 2% fat and 4% latex (composed from 86% of cis - poly isoprene: rubber) [7, 9].

The reducer agent, argan's pulp, was collected from straight around the argan tree from the Essaouira city region of South Morocco.

Alkali agent

The alkali agent used, calcium carbonate, was of technical grade and obtained from a customary magazine.

Spectrophotometer

The ultraviolet - visible (UV - vis) spectrophotometer used in this study was a Thermo, model Helios Epsilon. The wavelength range was 325–1100nm with a spectral bandwidth of 1nm.

pH meter

The pH meter used was a Henne, model AD1000. It is the multimeter with professional banc for pH, redox (oxydo - reduction potential) and temperature measurements.

Vat

The vat used was a 2 L stainless steel tin. Heating was

provided using a thermostat hotplate, Scilogex MS - H280 - Pro.

2.2. Dyeing Process

The preparation vat

In the beginning, natural indigo was reduced using argan's pulp. The reducing conditions are presented below:

- Dye: 1% (owf)
- Argan's pulp: X g/L
- Calcium hydroxide: Y g/L
- Temperature: 40°C
- pH: 11
- Liquor ratio

The full solubility of indigo was indicated by the observation of an oily layer with a blue color on the vat surface. The solution was homogeneous with a green – yellow color.

Dyeing conditions

The yarn of wool (10g) was soaked and wrung before being involved in the dyeing vat as described hereafter:

- Insert the yarn into the dyeing vat three times at: 5min, 10min and 15min. After each inserting time, the yarn was removed and left open to air for 5 min.
- The temperature was fixed at 45°C.
- The global dyeing time was 30 min.

Oxidation and rinsing

The oxidation was realized in the open air for 5 min after every insertion time.

The rinse was realized at the end of the dyeing process as presented below:

- 1st rinse in cold water.
- 2nd rinse with neutralization in citric acid to pH=7.

Drying

Drying may be carried out open to air or in a sterile environment at a temperature between 60°C and 80°C.

2.3. Optimization of the pH Dyebath

Dyeing recipe

The weight of yarn used was 5g and the liquor ratio was 1/100.

- Dye: 1% (owf)
- Argan's pulp: 20 g/L
- Calcium hydroxide: Xg/L
- Temperature: 40°C
- Time: 30min
- pH of dye bath: -

The values of X studied are given in Table 1.

Table 1. The pH value and related concentration of calcium hydroxide.

X (g/L)	pH
0.5	10.5
1.5	11
8	12
20	12.5

It should be noted that the optimal quantity of calcium hydroxide could not be set since this product was sensitive to

carbon dioxide that exists in the air. This can react with calcium hydroxide to produce calcium carbonate.

The dyeing process was realized as described above.

Spectral analysis

Calibration of the spectrophotometer

Calibration of the spectrophotometer was realized with a standard solution prepared according to the concentration of argan's pulp and the four concentrations of calcium hydroxide (X: 0.5, 1.5, 8, 20 g/L) that were used in the previous dye bath. Therefore, we prepared four standard solutions with the four preceding concentrations of calcium hydroxide and one with the same concentration of argan's pulp.

Measurement of the exhaustion rate of indigo

We removed 1mL of the solution from each of the four dye baths to be measured. Each sample was diluted to 10 mL using the prepared standard solutions. The absorbance measurements are shown in Figure 2.

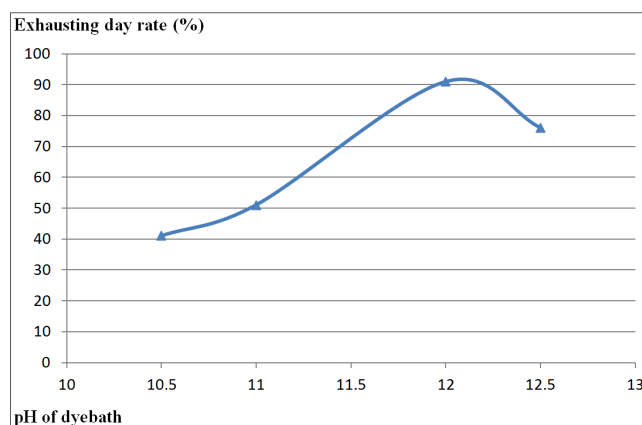


Figure 2. The dye absorbance measurements as a function of dye bath pH.

2.4. Optimization of the Reducer Concentration

Eight samples of Boujaad wool were dyed with several concentrations of argan's pulp. The others parameters of the recipe remained the same without any modification.

Dyeing recipe:

The weight of yarn used was 5g with a liquor ratio 1/100.

- Dye: 1% (owf)
- Argan's pulp: X g/L
- Calcium hydroxide: 2g/L
- Temperature: 40°C
- Duration: 30min
- pH: 11.5
- X: 5, 7, 9, 12, 16, 20, 25 and 35g/L.

The dyeing process was realized as indicated above.

Spectral analysis

Calibration of spectrophotometer

The standard solutions were prepared according to the eight reducer concentrations (X: 5, 7, 9, 12, 16, 20, 25 and 35g/L) and the same calcium hydroxide concentration (2g/L, pH 11.5) was added as described in the last dyeing recipe.

Measurement of the exhaustion rate of indigo

For each of the eight dye baths we removed 1mL of the solution to be measured. Each sample was diluted to 10 mL using the prepared standard solutions. The absorbance measurements are shown in Figure 3.

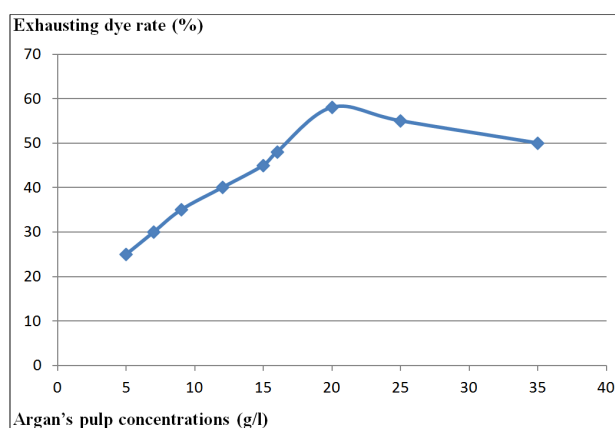


Figure 3. The dye absorbance measurements as a function of argan's pulp concentrations.

2.5. Optimization of the Dyeing Time

Samples of wool fiber were dyed with several dyeing times using the optimal recipe previously described and the same dyeing process.

Dyeing recipe

The weight of yarn used was 10g with a liquor ratio 1/100.

- Indigo: 1% (owf)
- Argan's pulp: 20 g/L
- Calcium hydroxide: 2g/L, pH 11.5
- Temperature: 40°C
- Duration: X min
- pH: 11.5
- The values of X were: 30min, 40min, 50min, 60min and 70min.

Spectral analysis

Calibration of the spectrophotometer

The standard solution was prepared using the same reducer concentration (20g/L) and calcium hydroxide concentration (2g/L, pH 11.5) added in the last dyeing recipe.

Measurement of the exhaustion rate of indigo

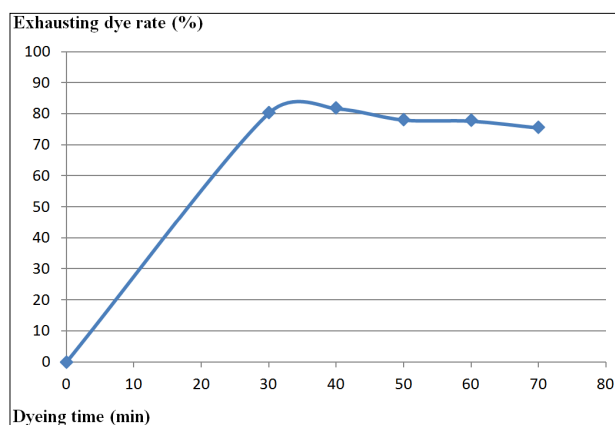


Figure 4. The dye absorbance measurements as a function of dyeing time.

After each dyeing time (Xmin), we removed 1mL of the solution to be measured. Each sample was diluted to 10 mL using the prepared standard solution. The absorbance measurements are shown in Figure 4.

3. Results and Discussion

3.1. Optimization of the Dyebathp

The sample dyed in the dye bath with a pH medium equal to 12 had a maximum intensity of coloration (Figure 2). The exhaustion rate of this sample has a maximum value at 91%.

At pH 12.5, the absorption intensity decreased. This decrease in the affinity of indigo to the fiber was attributed to the high dye solubility.

However, in order to avoid fiber alteration in the high alkaline medium, we should not exceed a pH of 11.5 at 40°C.

3.2. Optimization of the Reducer Concentration

The sample dyed with an argan's pulp concentration of 20g/L produced the maximum dye absorption (highest exhaustion rate). Upon increasing the reducer quantity, the reducer solubility increases and quick diffusion inside the fiber is observed.

In contrast, a reducer concentration exceeding this value (>20g/L) causes a decrease in the dye absorption. This may be explained by over-reduction of indigo that can lead to the dye being destroyed.

3.3. Optimization of the Dyeing Time

A dyeing time of 30min realized the maximum dye absorption. Beyond this value, we observe a slight diminution of the dye absorption. This diminution, may be due to the desorption of the dye after establishing equilibrium. There are many factors contributing to this desorption, such as pH of the medium and the aerial oxidation of indigo. We conclude that the optimal dyeing time for this recipe was 30min.

4. Conclusions

In this study, we tested the possibility of using spectroscopic analysis in a dyeing process with natural indigo. Therefore, we checked the feasibility of this analysis to quantify the indigo in the natural vat during the dyeing process. We ascertained the power of argan's pulp to reduce indigo at an optimal quantity. This natural reducer is a plentiful source from the south of Morocco and is usually used as a food for cattle. Subsequently, we realized the optimal pH using natural calcium hydroxide. Finally, we found the optimal dyeing duration taking into consideration the optimal parameters previously obtained.

Globally, we noted the complexity in realizing the spectroscopic analysis of the indigo vat due to the instability of indigo concentration in the dyeing vat. Indigo in the vat

oxidized quickly upon exposure to atmospheric air, which can effect the initial concentration. Therefore, we had to consider this variation and minimize this oxidation during the dyeing process.

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