

Original article

# Extraction and Application of Eco-Friendly Natural Dyes Obtained from Libyan Local Plants on Textiles

Eman Alsaeh<sup>1</sup>, Najmeddin Ellali<sup>2\*</sup>, Amjaad Alseah<sup>1</sup>, Waad Altroak<sup>1</sup>, Nooralhuda Atia<sup>1</sup>

<sup>1</sup>Department of Chemistry, Faculty of Science, Alejelat, University of Zawia, Libya

<sup>2</sup>Faculty of Pharmacy, University of Zawia, Libya

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Corresponding Email. [najmiellali@yahoo.com](mailto:najmiellali@yahoo.com)

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## ABSTRACT

**Aims.** In the present study, experiments were carried out to use an extract isolated from parts of plants (shell of almond, bark, and pods of carob, the flower of *reichardia tingitana* L., and leaves of olive, guava, and malvus with aqueous as a natural dye. **Methods.** The dye potential of the extracts was evaluated by dyeing on cotton, silk, and nylon fabrics under normal dyeing conditions using selected synthetic and natural mordants. The synthetic mordant was ferrous chloride whereas the natural mordants were lemon peel and pomegranate rind and tested for their colour fastness to washing and rubbing properties. **Results.** The results found to be for each mordant was selected for light and dark shades. The washing and rubbing fastness of the dyed samples were also evaluated, giving fair to excellent fastness grades and this evaluation is also useful for textile industries. **Conclusion.** Dye extracted from plants can be used as a textile dye with selected natural mordants.

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## INTRODUCTION

Natural dyes are derived from natural resources. Colouring materials obtained from natural resources such as plants, animals, minerals, and microbial varieties are used to tint various textile materials. Many different regions in the world have their own natural dyeing traditions using the natural resources available in those regions [1]. Natural dyes are clinically safer than synthetic analogs in handling and use because of their non-carcinogenic and biodegradable nature and generally have higher compatibility with the environment [2]. In addition, some synthetic dyes are not environmentally friendly [3]. The use of non-allergic, non-toxic, and eco-friendly natural dyes has become a matter of significant importance due to the increased environmental awareness in order to avoid some hazardous synthetic dyes [4]. Using vegetable waste to extract colour not only gives colour yield but also saves the environment from the hazardous waste produced by artificial colours [5].

*Olea europaea* L., Olive leaves have numerous medicinal uses in addition to being used in the production of natural dyes [6]. The olive tree's leaves are rich in flavonoids including luteolin, kaempferol, and apigenin as well as several other compounds such as oleuropein and sesquiterpenoids. [7].

The guava tree, or *Psidium guava*, is a member of the myrtaceae family [8]. Quercetin, a substance found in guava leaves that gives textiles their colour, is also used in the preparation of natural dyes and textile dyeing processes [9].

*Malva sylvestris* L. plant, is an annual herbaceous medicinal plant belonging to the *Malvaceae* family and usually known as common mallow. It is commonly used as a vegetable and for medicinal purposes and textile dyeing [10].

*P. Amygdalus* L. The tree was also used as a natural colourant in the manufacture of textile dye [11] and for some therapeutic purposes [15]. Almond shells contain a variety of phenolic compounds, phenolic acids, and some other constituents of the colouring components found in almond shell extracts [12].

L-Ceratonia, Siligaqua tree commonly known as the carob tree. It is an evergreen tree belonging to the Leguminosae (*Fabaceae*) family, that grows in many parts of the world [13]. The L-Ceratonia, Siligaqua tree was also used in the manufacture of dyes and some medicinal purposes [14]. According to high-performance liquid chromatography (HPLC), the tree contains a wide variety of compounds, including flavan-3-ol groups and their galloylesters, gallic acid, (+)-catechin, epicatechingallate, and quercetin glycoside, as well as condensed tannins (proanthocyanidins), hydrolyzable tannins (gallotannins and ellagitannins) and others [14,15].

L. Roth Reichardia tingitana plant is a common wild plant belonging to the *Asteraceae* family of the genus Reichardia, [16], and it has pharmacological activities [17]. Based on a preliminary phytochemical analysis, the L. Roth Reichardia tingitana plant contains phenolics, tannins, flavonoids, coumarins, volatile oils, glycosides, flavonoids, locations, esters, high stress levels, and/or triterpenes [17,18].

The aim of the study was to extract eco-friendly natural dyes and we investigated the possibility of dyeing fabric using natural dyes extracts and assessment of the colour fastness properties of the dyed fabrics.

## MATERIALS AND METHODS

### Collection of plants materials

The dyeing materials of Ceratonia siliqua L. (carob pods and bark), P.Amygdalus L. (almond shell), Oleaeuropaea L., guava Pisdium, Malva parviflora (leaves) and Reichardia tingitanaL.(flower), were collected from Aljelate region, Libya (Figure 1), and natural mordant materials Punicagranatum (Pomegranate rind) and Lemon Peel were collected and dried in shade, Ferrous Chloride as a metallic mordant.

### Textile materials

Silk, nylon and cotton fabrics were chosen for this study. Fiber samples were cut into 4 × 2 cm. The silk, nylon and cotton were purchased from local shops in Tripoli and Zawia cities, Libya.

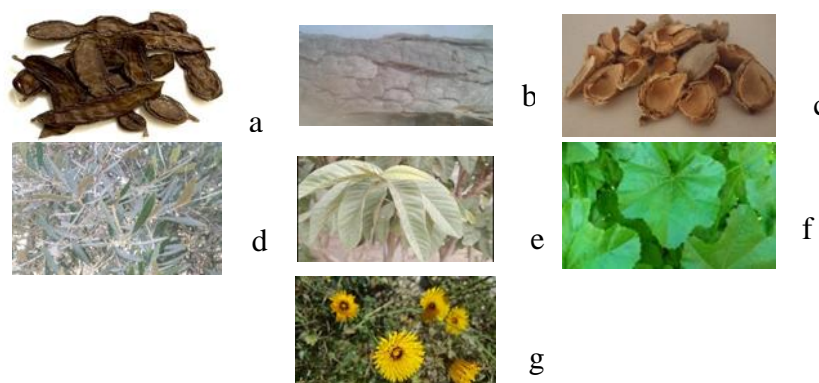


Figure 1. Plants used in study: a) carob pods. b) carob bark. c) almond shell. d) olive leaves. e) guava leaves. f) malvus leaves. g) *R. tingitana* L. Flower.

### Preparation of dyeing material

The shade dried (pods, bark, shell, flower) of plants materials. were washed with distilled water and dried. The material was finely powdered with the help of a grinding machine.

### Extraction of dye

Boiling water was used to extract the dyes using a bath liquor ratio of 25 grams per 500 milliliters. For one hour, the extraction procedure was completed.

### Conventional dyeing

Samples of fabric were boiled in dye extract for one hour. After cooling, they were air dried and cleaned with cold water [19].

### Dyeing after treatment with a mordant

Silk, nylon and cotton samples were boiled with mordant in distilled water (3g/l) for 20 minutes. Then, the mordanted fabrics and the dyeing both solutions were putted together in the dyeing beaker for 60 minutes. After dyeing, the unfixed

dyestuff was removed by rinsing with cold water, and then air dried [19]. The same procedure was followed for other mordant dyeing.

Silk, nylon and cotton samples were boiled with mordant in distilled water (3g/l) for 20 minutes. Then, the mordanted fabrics and the dyeing both solutions were put together in the dyeing beaker for 60 minutes. After dyeing, the unfixed dyestuff was removed by rinsing with cold water, and then air dried [19]. The same procedure was followed for other mordant dyeing.

### ***Fastness properties***

The dyed material was tested for wash fastness and rubbing fastness. The colour is usually rated by the loss of depth of colour in original sample [20].

### ***Hand washing***

Dyed and undyed fabric samples in contact were agitated in a soap solution (Ariel, Egypt) (0.5 g/l) for 20 minutes at 40°C. After rinsing and drying, the change in color of the dyed samples and the bleeding to the white fabric (cotton) was determined visually. The changes were graded with marks 1 - 5 (1 = poor, 5 = excellent) [19].

### ***Colour fastness to rubbing***

The rub fastness of the dyed fabrics was carried out by rubbing the fabrics manually and checking for fading of colour [9].

### ***UV/ Visible absorption measurement***

UV-Visible spectroscopy was used to characterize the dyes in the crude extract. Spectral analysis was performed on the crude extraction solution (25g/500ml), which was made by dissolving it in distilled water for 60 minutes.

## **RESULTS AND DISCUSSIONS**






















### ***Colour shades of dyed fabrics***

The whole extraction and dyeing process is environmentally safe. The obtained results showed the dyeing potential of all natural plant sources as a dyeing source. Knowledge, documentation, and proper evaluation of the dye industry as well as dyeing techniques are needed to increase the use of natural dyes.

Dyes are molecules which absorb and reflect light at specific wavelengths to give human eyes the sense of colour. Dyeing in textiles is a process in which colour is transferred to a finished textile or textile material (like fibers) to add permanent and long-lasting colour [21]. An essential component in giving the fabric colour is the use of mordants. The fabric samples had different shades due to the type of mordant used. Improved colour intensity outcomes rely on the type of mordant utilized. [22]. The colour intensity of the non-mordanted fabrics was lower than that of the mordanted fabrics. Without a mordant (control), the silk, cotton, and nylon fabrics produced brownish shades Table (1).

Additionally, the results show that a wide range of colours can be achieved by using natural and mineral mordants to dye fabric samples containing plant extract. that is displayed in table (2), the variety of colours attained. In general, depending on the kind of plant, natural mordants produced colours ranging from dull to dark, whereas synthetic mordants like ferrous chloride produced darker hues in varying shades.

**Table 1. Results of colours dyed fabrics without mordants with plants extracts.**
































































Plants used	Part used	Fabrics		
		Nylon	Silk	Cotton
Carob	Bark			
Carob	Pods			
Almond	Shell			
R. tingitana L.	Flower			
Guava	Leaf			
Olive	Leaf			
Malvus	Leaf			

When iron mordant is used, the colour and shade are clearly altered in comparison to dyes without a mordant; however, the colour is not altered as much when lemon peel mordant is used. These outcomes were caused by the fixing agent's

chemical composition being influenced by the samples' stability characteristics. Lemon peels yield the lightest shades; this could be because one of the components of lemons, citric acid, has the ability to bleach [23].

In general, iron mordant dyeing results in a darker colour than other techniques. Although darker colours have been seen on cotton in carob bark, almond shells, guava, and olive leaves, all samples seem to have more colour on silk and nylon than cotton. Different molecular groups, like flavonoids and tannins, are present in these plants. Darker samples on silk were obtained with species that contained tannin (carob, almond, *R. tingitana* L) [15], flavonoids in guava, olive, and malvus [7], and carob [15]. Darker colours are obviously better for dyeing because they can be lightened to create lighter shades if needed. Conversely, the dyed samples displayed the brightest shades when made of silk and nylon. The fruits and vegetables with the brightest colours were olives, guava, malvus (leaves), and carob (bark). pointing out that in the majority of plants, silk and nylon have the highest saturation levels.

**Table 2. Results of colours dyed fabrics in the presence of mordants with plants extracts.**

Plants used	Part used	Fabrics			
		Mordants	Nylon	Silk	Cotton
Carob	Bark	Pomegranate rind			
		Lemon Peel			
		Ferrous Chloride			
Carob	Pods	Pomegranate rind			
		Lemon Peel			
		Ferrous Chloride			
Almond	Shell	Pomegranate rind			
		Lemon Peel			
		Ferrous Chloride			
<i>R. tingitana</i> L.	Flower	Pomegranate rind			
		Lemon Peel			
		Ferrous Chloride			
Guava	Leaf	Pomegranate rind			
		Lemon Peel			
		Ferrous Chloride			
Olive	Leaf	Pomegranate rind			
		Lemon Peel			
		Ferrous Chloride			
Malvus	Leaf	Pomegranate rind			
		Lemon Peel			
		Ferrous Chloride			

**Colour fastness of dyed fabrics**

**Results of the colour fastness properties of dyed fabrics samples (without mordant)**

**Silk**

Washing: According to Table (3) results regarding the dyed silk sample's colour fastness, the colour change can range from very good when using *R. tingitana* L. flowers and olive leaves to excellent when using carob (bark, pods). The

combination of guava leaves and almond shells works well with malvus leaves, but it does not cause any noticeable stains on cotton.

**Dry and wet rubbing:** The results of the colour fastness of dyed samples typically range from very good to excellent for dry rubbing and from moderate to very good for wet rubbing, as shown in table (3).

#### **Cotton**

**Washing:** Table 3 shows that the cotton samples' colour fastness ranged from very good to excellent for the most of plants, and no staining was noted. **Wet and Dry Rubbing:** The degree of colour fastness ranged from very good to excellent. Table (3).

#### **Nylon**

**Washing:** The degree of colour fastness ranged from very good to excellent with the all samples, and no staining was noticeable. Table (3). **Wet and Dry Rubbing:** According to Table (3), the level of colour fastness varied from very good to excellent when dry rubbing and from moderate to very good when wet rubbing.

### **Results of colour fastness for dyed samples in the presence of mordants**

#### **Silk**

**Washing:** The results of the colour fastness of washing for the dyed silk samples, which are displayed in tables (4,5,6), demonstrate that all of the plant samples had colour changes ranging from very good to excellent. Lemon peels and ferrous chloride mordants do not show any staining on the cotton. Dry and wet rubbing when using pomegranate rind and lemon peel mordants, the colour fastness results ranged from very good to excellent for both dry and wet rubbing Table (4, 5) and varied in strength from moderate to weak with carob pods, almond shells, and *R. tingitana* L. flowers for wet rubbing Table (6).

#### **Cotton**

**Washing:** The colour fastness of cotton samples to change in colour was from very good to excellent with the most samples in the presence of pomegranate peel mordant, Table (4). The degrees of colour fastness of the dyed samples for the change in colour in the presence of ferrous chloride mordant were from very good to excellent with all samples Table (6). While the degree colour fastness of the all samples in the presence of lemon peel mordant ranged from very good to excellent. and poor in mavlus leaves and *R. tingitana* L. flowers Table (5). This is caused by a weak dye-fibre bond formation between the natural dye-mordant and the fiber, as well as a colour shift brought on by the dye-metallic mordant-fibre complex breaking during washing and ionizing the natural dyes. Because most natural dyes contain hydroxyl groups that ionize in alkaline or acidic conditions, many fabrics dyed with natural dyes under acidic conditions change colour when washed with alkaline detergents or soap [24].

**Dry and wet rubbing:** The colour fastness of the dyed samples shows between very good to excellent for dry rubbing, and from moderate to very good for wet rubbing of the pomegranate rind and lemon peel mordants Table (4 ,5). The ferrous chloride mordant performed exceptionally well on dry rubbing, but in wet rubbing it produced moderate to weak results in all samples (Table 6). This could be because wet rubbing breaks dye mineral complexes into simple particles [25].

#### **Nylon**

**Washing:** Tables (4,6) show that the dyed samples had excellent colour fastness in the current ferrous chloride and pomegranate rind mordants, and no staining showed up on the cotton. While most plants showed excellent colour fastness when lemon peels were added as a mordant Table (5). The dye's washing stability is dependent on two factors: the rate of dye diffusion and the state of the dyes inside the fibers. As a result, its molecular size increased and it showed good washing fastness. Furthermore, a complex composition containing a mordant produces a faster colour due to its dye-insoluble effect. Furthermore, it is possible to explain why the dyed sample's good washing stability without the need for a mordant was caused by the colouring agent's affinity for hydrogen bonds and van der Waals forces [26]. **Dry and wet rubbing:** When using pomegranate rind and lemon peels as mordants for dry and wet rubbing Table (4,5), the degree of colour fastness varied from very good to excellent; however, when using carob bark, guava leaves, and *R. tingitana* L. flowers in the presence of ferrous chloride as a mordant, it was weak. This is attributed to a difference in the extent to which the low aqueous soluble ferrous-tannate complexes were able to diffuse within the dyed fiber. It was expected that the big molecular size complex that developed in the dyeing bath would have very poor diffusional behavior and would primarily deposit on the dyed fiber's periphery, resulting in a low rubbing fastness. Table (6) [27].

**Table 3. Colour fastness properties of fabrics dyed with aqueous extracts of plants samples in the absence of mordant.**

Plants	Silk Fabric						Nylon Fabrics						Cotton Fabrics					
	Colour fastness degree						Colour fastness degree						Colour fastness degree					
	washing		Rubbing				Washing		Rubbing				Washing		Rubbing			
	CC	CS	Dry		Wet		CC	CS	Dry		Wet		CC	CS	Dry		Wet	
CC			CS	CC	CS	CC			CS	CC	CS	CC			CS	CC	CS	
<b>Carob Bark</b>	5	5	5	4	4	4	5	5	4	5	5	4	5	5	5	4	3	3
<b>Carob Pods</b>	5	5	4	4	4	4	5	5	5	5	5	4	5	5	4	4	4	3
<b>Almond Shell</b>	5	5	5	4	4	4	5	5	5	5	5	4	4	5	4	4	4	4
<b>R. tingitana. Flowers</b>	4	5	5	4	3	5	5	5	5	5	5	4	1	5	4	4	1	2
<b>Guava leaves</b>	5	5	5	4	5	4	5	5	5	5	5	4	4	5	4	4	4	4
<b>Olive leaves</b>	4	5	5	5	4	4	5	5	5	5	5	5	4	5	5	5	4	4
<b>Malvus leaves</b>	3	5	5	4	4	4	4	5	5	5	4	4	2	5	4	4	4	4

**Table 4. Colour fastness properties of fabrics dyed with aqueous extract of plant samples in the presence of pomegranate rind m**

Plants	Silk Fabric						Nylon Fabrics						Cotton Fabrics					
	Colour fastness degree						Colour fastness degree						Colour fastness degree					
	washing		Rubbing				Washing		Rubbing				Washing		Rubbing			
	CC	CS	Dry		Wet		CC	CS	Dry		Wet		CC	CS	Dry		Wet	
CC			CS	CC	CS	CC			CS	CC	CS	CC			CS	CC	CS	
<b>Carob Bark</b>	4	5	5	4	5	4	5	5	5	4	5	3	4	5	4	4	4	3
<b>Carob Pods</b>	5	5	5	5	5	4	5	5	5	5	5	4	5	5	4	4	3	4
<b>Almond Shell</b>	5	5	5	4	5	4	5	5	5	5	5	4	4	5	4	4	4	4
<b>R. tingitana. flowers</b>	5	5	5	5	5	4	5	5	5	4	5	4	5	5	5	4	3	4
<b>Guava leaves</b>	2	5	4	5	5	4	2	5	5	5	5	4	2	5	4	4	4	3
<b>Olive leaves</b>	5	5	5	5	5	4	5	5	5	4	4	4	5	5	4	5	4	3
<b>Malvus leaves</b>	5	5	5	5	5	4	5	5	5	5	5	4	5	5	4	5	4	4

**Table 5. Colour fastness properties of fabrics dyed with aqueous extracts of plant samples in the presence of lemon peel mordant.**

Plants	Silk Fabric						Nylon Fabrics						Cotton Fabrics					
	Colour fastness degree						Colour fastness degree						Colour fastness degree					
	washing		Rubbing				Washing		Rubbing				Washing		Rubbing			
	CC	CS	Dry		Wet		CC	CS	Dry		Wet		CC	CS	Dry		Wet	
CC			CS	CC	CS	CC			CS	CC	CS	CC			CS	CC	CS	
<b>Carob Bark</b>	5	5	4	4	4	4	5	5	5	4	4	4	5	5	4	4	4	4
<b>Carob Pods</b>	5	5	4	4	4	4	5	5	5	4	5	4	4	5	4	4	4	4
<b>Almond Shell</b>	5	5	4	4	5	4	5	5	5	4	5	4	5	5	4	4	4	4
<b>R. tingitana. Flowers</b>	5	5	5	5	4	4	2	5	5	5	4	4	1	5	4	5	3	3
<b>Guava leaves</b>	2	5	4	4	4	3	5	5	5	5	4	4	5	5	4	4	4	3
<b>Olive leaves</b>	4	5	5	5	4	4	5	5	4	4	5	4	4	5	4	4	4	4
<b>Malvus leaves</b>	5	5	4	4	4	4	4	5	5	5	5	4	1	5	5	4	4	4

**Table 6. Colour fastness properties of fabrics dyed with aqueous extract of plant samples in the presence of ferrous chloride mordant.**

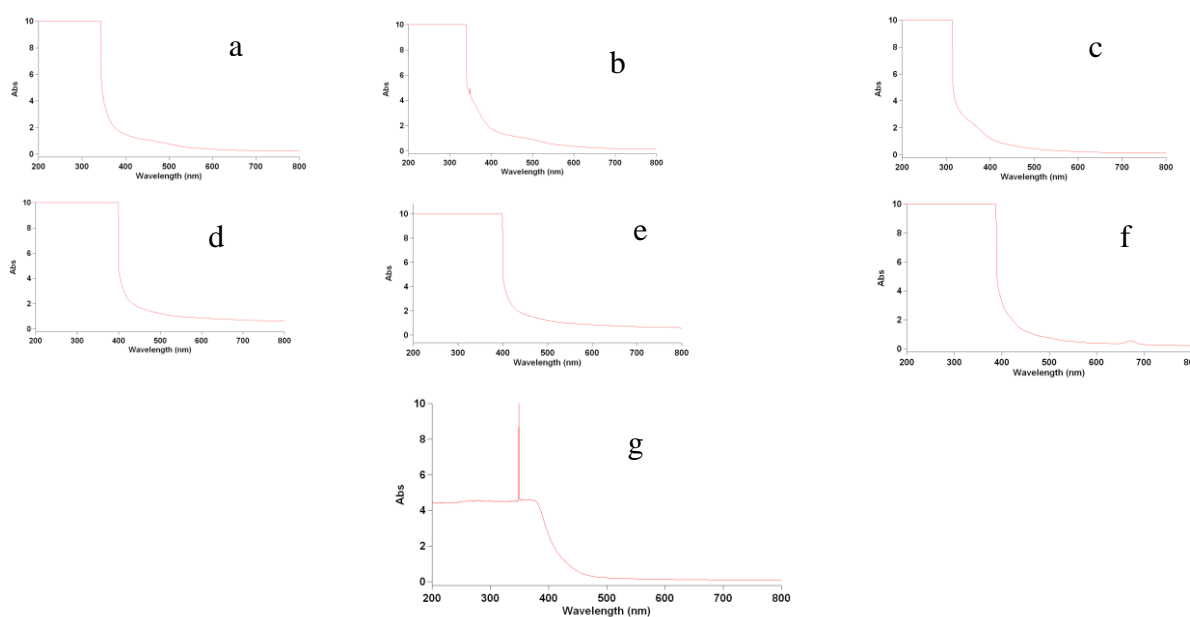
Plants	Silk Fabric						Nylon Fabrics						Cotton Fabrics					
	Colour fastness degree						Colour fastness degree						Colour fastness degree					
	washing		Rubbing				Washing		Rubbing				Washing		Rubbing			
	CC	CS	Dry		Wet		CC	CS	Dry		Wet		CC	CS	Dry		Wet	
CC			CS	CC	CS	CC			CS	CC	CS	CC			CS	CC	CS	
<b>Carob Bark</b>	5	5	5	4	3	4	5	5	5	4	2	4	5	5	4	4	2	3
<b>Carob Pods</b>	5	5	5	4	2	2	5	5	5	4	3	4	5	5	4	4	2	2
<b>Almond Shell</b>	5	5	5	4	2	3	5	5	5	5	4	4	5	5	4	4	3	4
<b>R. tingitana. flowers</b>	5	5	5	5	1	4	5	5	5	4	1	3	5	5	4	4	1	3
<b>Guava leaves</b>	4	5	5	4	4	3	5	5	5	4	2	4	5	5	4	4	3	3
<b>Olive leaves</b>	5	5	5	5	3	4	5	5	5	4	5	4	4	5	4	4	2	3
<b>Malvus leaves</b>	5	5	4	4	4	4	5	5	5	4	4	4	4	5	4	4	4	4

CC: Change of Colour, CS: Staining of colour.

### UV-Visible spectroscopic study

The UV-Vis spectra of the crude plants extract dyes in an aqueous solution is shown in figure (2). The characteristic spectrum of almond shell extract is characterized by colour ranging from light to dark browns. These colours are chemically related mainly to polymers of phenolic compounds and tannins polymerization. Generally, phenolic compounds include many great organic substances that have the common characteristic of possessing an aromatic ring with one or more substituent hydroxyl groups and a functional side chain [28]. The UV-Vis spectrum of almond shell extract show one absorption band at 342 nm region may be attributed to the presence of flavonoids related in the extract solution [28].

Carob pods colourant shows two absorption maxima at 338 nm and 348 nm. These results could be explained that extract is rich in polyphenols and flavonoids, flavones and flavanones in the sample composition [29] while carob bark shows one absorption band at 314 nm demonstrated the presence of flavonoids and tannins [28] as well as *R. tingitana* L. flower colourant shows one major peak at 369 nm that is explained the presence of phenolics, tannins, flavonoids in extract [17]. The aqueous extract of olive leaves indicate two absorption peaks: the first at a wavelength of 387 nm and the second peak at a wavelength of 673 nm. This explain that the extract could be due to the presence of the phenolic hydroxyl groups in their structure such as oleuropein, hydroxytyrosol, and luteolin- 7-O-glucoside acid and flavonoids [30]. As for guava leaves show one peak at wavelength of 399 nm, and this is attributed to the leaves containing poly phenols such as flavonoids, tannins, essential oils and chlorophyll [8,9]. Malvus leaf extract showed one absorption peak at wavelength 348 nm, the presence of phenolic compounds in the MS colourants provide a pale yellow colour which explains the presence of flavones and flavonols and other active compounds in the leaves [10].



**Figure 2.** Visible spectrums of the aqueous extracts: a) almond shell. b) carob pods. c) carob bark. d) *R. tingitana* L. flower. e) guava leaves. f) olive leaves. g) malvus leaves.

### CONCLUSION

It was found from this study that isolated dyes from the parts of plants (shell of almond, bark and pods of carob, flower of *reichardia tingitana* l. and leaves of olives , guava and malvus) can be successfully used for dyeing of cotton ,silk and nylon to obtain a wide range of dark and light colours by using ecofriendly natural mordants with regards to colour fastness ,test samples exhibited excellent to washing and fairly good to good fastness to rubbing and theses data also helpful for textile industries. These results give us different data to evaluate of fastness properties and the use of these dyes will greatly contribute to achieving a safe, ecofriendly and green environment.

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## استخلاص وتطبيق الأصباغ الطبيعية الصديقة للبيئة المستخرجة من النباتات المحلية الليبية على المنسوجات

إيمان السائح<sup>1</sup>، نجم الدين اللالي<sup>2</sup>، أمجاد السائح<sup>1</sup>، وعد الطروق<sup>1</sup>، نور الهدى عطية<sup>1</sup>

<sup>1</sup>قسم الكيمياء، كلية العلوم العجيلات، جامعة الزاوية، ليبيا

<sup>2</sup>كلية الصيدلة، جامعة الزاوية، ليبيا

### المستخلص

**الأهداف.** أجريت في الدراسة الحالية تجارب لاستخدام مستخلص معزول من أجزاء من النباتات (قشرة اللوز، لحاء وقرون الخروب، زهرة نكد طنجي، وأوراق الزيتون، الجوافة، والخبيزة مع الماء كصبغة طبيعية. طرق الدراسة. تم تقييم القدرة الصبغية للمستخلصات عن طريق الصباغة على الأقمشة القطنية والحريرية والنايلون تحت ظروف الصباغة العادية باستخدام مثبتات اصطناعية وطبيعية مختارة. كان المثبت الاصطناعي هو كلوريد الحديدوز بينما كانت المثبتات الطبيعية هي قشر الليمون وقشر الرمان واختبار ثبات لونها لخصائص الغسيل والفرك. النتائج. تم اختيار النتائج التي وجدت لكل مثبت أعطت ظلال فاتحة وداكنة. كما تم تقييم ثبات الغسيل والفرك للعينات المصبوغة، مما أعطى درجات ثبات متوسطة إلى ممتازة. وهذا التقييم مفيد أيضاً للصناعات النسيجية. الخاتمة. توصلت هذه الدراسة إلى استنتاج مفاده أن الصبغة المستخرجة من النباتات يمكن استخدامها كصبغة نسيج مع مثبتات طبيعية مختارة.

**الكلمات الدالة.** المثبتات، الأصباغ الطبيعية، النسيج، اختبار ثبات اللون، النبات.