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Saffron in Natural Dyeing

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Abstract

Nowadays, the scientific studies relating to natural dye sources in worldwide are quite extensive. In this context, natural dyeing refers to natural dyes in the fiber or to the dye source content. In this work, the saffron dye plant has been investigated. This plant is named Crocus sativus L. (Iridaceae) in Latin. The dye source in the plant is its stigma. Saffron is one of the most expensive dyes in the world. The colorants present in the plant are crocin and crocetin. These colorants can be used in natural dyeing. They are also investigated in terms of their dyeing properties.
Keywords: Saffron; Natural Fibres; Colour; Dyes; Crocin; Crocetin.

Introduction

Natural dyeing is as old as textile history. In this context, colours have always been interesting from past to present. Among dye plants, saffron is an ancient dye plant [1]. Saffron (*Crocus sativus* L.) is an inconsiderable and perennial plant growing up to 30 cm [2].

In the colouring of textiles, natural dye sources (including dyes) with mordants (especially natural mordants) play an important role. Saffron of these dye plants was used in the dyeing of silk. This plant contains crocin and crocetin carotenoids [3]. Their chemical formulae are $C_{44}H_{64}O_{24}$ (Crocin) and $C_{20}H_{24}O_4$ (Crocetin) [4].

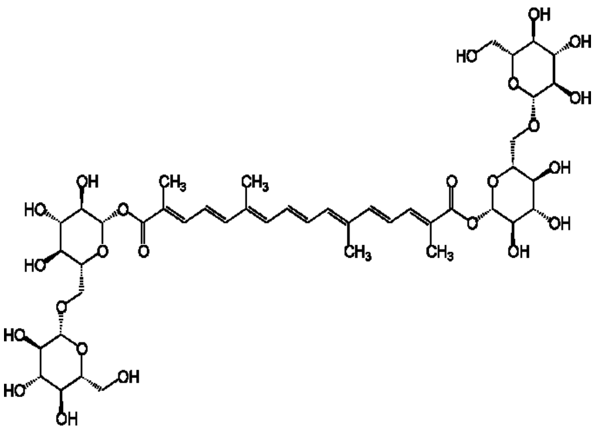

Fig. 1. Structure of crocin compound [5].

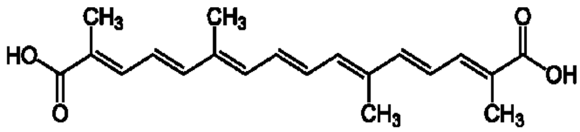

Fig. 2. Structure of crocetin compound [6].

Saffron dye plant

This plant was first dye source in terms of history recognized by humans [7]. It can be used as a dye source to colour natural and synthetic fibres. In its stigma, it has a yellow colour caused by crocin dye [8]. Saffron has been used as a textile dye plant including natural dyes for a very long time. In this sense, this plant was used with an iron mordant to obtain a golden yellow hue on paper in Medieval Times [9]. It has been evaluated to use as a dye source for a very long time. An aqueous extract of this dye source has been used to colour wool and cotton [10]. It was considerable dye source in ancient Greece. At the same time, this dye source was used in the colouring the wedding clothes in Rome. In dyeing wool and silk with using saffron plant, the material is first treated with an alum mordant, then until the obtaining the desired colour, it is immersed in the dye mixture [11]. Saffron plant belongs to the Iridaceae family. The plant was famous to the ancient Greeks and Romans [12]. Its stigmas have good dyeing characteristics. This plant is a direct yellow dye source. The present source is suitable for colouring wool and silk fibres [13].

Saffron dye plant can be used to colour wool, silk and cotton materials without a mordant. As a

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mordant, tin and alum can be also applied to the materials. For unmordanted wool, an orange-yellow colour can be obtained [14]. This dye plant can be used in the dyeings of wool, silk and cotton. In the dyeings of saffron, an alum mordant gives an orange-yellow colour. This colour is known as the saffron yellow [15]. Saffron dye can be applied to material as a direct dye. In this case, mordants are not used. In the dyed material, the colour is among yellow and reddish [16]. Saffron was used to provide a yellow colour present in manuscripts and fibres. The dried flower stigmas (about 200 stigmas) can provide 1 g of dye [17]. This plant was a notable plant during the Crusades in Europe and used extensively in a short time as a dye plant. It has been cultivated in Anatolia for 3500 years [18]. Saffron dye plant has been also used in the colouring of the clothing relating to Hindu and Buddisth monks in the several regions of the world such as India, Tibet and China [19]. The dried saffron contains 10 % of crocin dye. Saffron dyes are soluble in water. Therefore, it can be said that they consist of environmentally friendly compounds [20]. Saffron is the one of main sources in terms of the yellowish colourful of textile fabrics [21]. The Romans used the saffron dye plant to obtain a pale tint in the past [22]. The Greeks and Romans utilized the saffron dye in the colouring of their clothes and in water used for the bath [23]. Saffron is peculiar to Iran and India. In the dyeing with saffron, the mordants used can improve the colour strength and fastness values [24]. Saffron presents an ancient yellow dye. The dye is obtained from the stigmas which are extracted with water [25]. Saffron has been a yellow dye source especially for the dyeing of wool since antiquity [26]. It is stable to natural light, moulds and pH. This plant produces a powerful dyeing characteristic [27].

In the Ottoman era, saffron was premier in the field of natural dyeing. Furthermore, it was also utilized to produce ink [28]. The name of saffron dye plant is derived from the Arabic ("za-faran"). It means yellow in the Arabic language [29]. Saffron has been cultivated in Anatolia for 3500 years. Phrygians wore clothes dyed with saffron yellow. The clothes of Persian kings were also dyed with saffron [30]. Saffron dye has been used as a substance that gives colour to beauty products as lip rouge [31].

Saffron dye plant has been used to create the yellow coloured regions in paintings, fabrics, Oriental carpets and Buddhist monks. It can be said that there are some features related to this plant such as dye characteristic, highly soluble in water. At the same time, it plays an important role in the decreasing of oxidation relating cellulose [32].

Saffron can be used in natural dyeing with alum ($KAl(SO_4)_2.12H_2O$) (yellow colour), iron ($FeSO_4.7H_2O$) (yellowish-brownish) and tin ($SnCl_2.2H_2O$) (light yellow) mordants. Apart from these, the direct dyeing with using saffron plant is also achieved (yellow colour). In the decoration of manuscripts which related to the thirteenth century and the middle ages, saffron was used for yellow and orange colours [33].

Saffron was also used to obtain a green pigment. This pigment is named as "verde azurro". In the 15th century Florence, saffron was mixed with indigo to form this pigment. The saffron was also used to obtain a green pigment with verdigris (copper sulphate) and vinegar [34]. The dyes present in saffron are of the known old dyes. Saffron dye was detected on linen sample obtaining from Egypt in the past (*ca.* 1050 B.C.) [35]. Saffron is grown in Iran, Azerbaijan, India, Pakistan, China, New Zealand, Greece, Italy, France, Spain, Morocco, Egypt and Israel in the world [36]. Other carotenoids in saffron are β-carotene, lycopene and zeaxanthin. An aqueous saffron extract quickly dyes wool, silk and vegetable fibres orange or yellow according to the amount of colorant used in terms of the colour's intensity [37]. Saffron (*Crocus sativus*) which includes crocin dye as a main component is in usage from Ancient Greece and Rome [38].

The family Iridaceae belonging to the saffron (*Crocus sativus*) has 1758 species. The main application areas related to the saffron dye are food, textile and medicinal industry. Natural dyes obtaining from the saffron tree can be also applied on the cotton fabric by means of ultrasonic-assisted extraction by using different mordanting techniques such as pre-mordanting and post-mordanting. In the dyeings obtained by using the pre-mordanting technique, the colourfastness observed is among good and very good. The fastness was appreciable in post-mordanting. Cotton and wool fabric dyed with the dyes present in the saffron extracted with pre-enzymatic treatment produced good wash and light fastness. For the pre-treatment of fabrics, the chitosan instead of the mordant can be also used. In this case, the chitosan was more effective. When the microwave technique for the extraction of the dyes of saffron is applied on cotton fabric, can be obtained the improved colour strength and extraction yield and antimicrobial activity on the fabric [39].

Saffron dye was also used to colour Persian carpets and rugs [40]. The dye which is obtained from saffron has a powdered form. This dye can be used to variegate wool, cotton and silk. The major component present in the dye is crocin. The aqueous extract related to the saffron has a glittery yellow colour. The glossier yellow colour is obtained from α-crocin. This compound is more soluble in water compared to the carotenoids. The saffron dye shows low fastness to washing in terms of wool and cotton. This situation can be resolved with the mordanting agents [41].

The homeland of the saffron plant is Anatolia. It has been cultivated in Anatolia for 3000 years. This plant has been used not only as a dye plant but also in medicine and cosmetics. Homer, Hippocrates and Plinus and many ancient writers mentioned saffron in their writings. Saffron was also used in dyeing textiles in ancient cities such as Sidon and Tyre [33].

Saffron plant was prevalently utilised in the Ottoman Empire for the production of the yellowish colours in the rugs [42].

Saffron plant which has a yellow colour was a very symbolic plant for Buddhists. This colour was a colour selected by Buddha. Traditionally, the robes coloured with this plant showed a sign sample [43].

Generally, 1 g saffron can be used in order to dye 200 g wool a pale yellow colour. Whereas, 4 g saffron gives a deep yellow colour [14].

Today, saffron cultivation is carried out only in Davutobası, Yörük, Aşağıgüney, Geren, Yazıköy and Değirmencik villages of Safranbolu in Turkey [44].

One stigma of saffron weighs approximately 2 mg [45].

Saffron dyes

Crocin is the most powerful component present in saffron dye plant. This dye has the colouring effect. At the same time, the dye can be easily dissolved in water. But it is somewhat carotenoid in nature [46].

Crocin is a glycoside which has a yellow-orange colour. This compound is easily soluble in hot water, slightly soluble in absolute alcohol, glycerine, and propylene glycol, insoluble in herbal oils. Crocetin has also sparingly soluble in water and mainly organic solvents, whereas soluble in pyridine and similar organic bases and dilute sodium hydroxide [47].

Crocin is an unusual carotenoid compound. This compound has responsibility related to saffron's colour (it has a shade yellow tinged with orange) [48].

Crocin is a carotenoid compound. This is a rare compound in nature. The compound is soluble in water without difficulty [49].

Crocin compound is a peerless hydrophilic characterized carotenoid. This carotenoid compound exists in the saffron dye plant. The presented compound is easily dissolved to give a reddish colour in water. The acid hydrolysis results in the production of crocetin and glucose. This situation occurs in a humid locality. Whereas crocetin and gentiobiose are created by means of alcoholic ammonia hydrolysis relating to crocin. The crocin can be dissolved in a strong sulphuric acid. This solution has a deep blue colour [50].

Saffron dye plant contains crocin compound. This compound dissolves in water and ethanol. The dissolution causes the extraction of crocin from the saffron plant [51].

The dyes in saffron (crocin and crocetin) can be determined by UV-Visible spectrophotometry. Crocin has two maximum absorption wavelengths in methanol (about 464 and 433 nm). Whereas, crocetin has three maximum absorption wavelengths in pyridine (about 464, 436 and 411 nm) [52].

The colour properties of carotenoids arise from the conjugated double bonds in their structures. Carotenoids must contain at least seven conjugated double bonds for the formation of a particular colour. As the number of conjugated bonds increases, the colour intensity also increases [53].

Carotenoids consist of isoprenoid units. The general structure of carotenoids is as eight five-carbon isoprenoid units. After the centre of the molecule, the order of arrangement is reversed. There is a symmetrical appearance. Carotenoids give maximum absorption in the visible region at a wavelength of 400-500 nm [54].

Carotenoid colorants are the compounds on the basis of a polyisoprenoid. The plants and animals can contain yellow, orange and red colorants. Carotenes are the general name of chemical components generating of only carbon and hydrogen atoms. Xantophyll compounds also include oxygen atoms. Owing to the deficiency in polar and solubilizing groups, the present colorants mostly show low solubility in water. As a reverse situation, aqueous saffron extract can be utilized to colour wool, silk and cellulose fibres [55].

Carotenoid compounds are characteristic non-cyclic dyes [56]. Crocetin dye is an aglycone molecule. This aglycone belongs to crocin and exists in saffron as a natural dye. Crocetin (MW: 328.4 g/mol) because of its structure is a sense carotenoid to thermal, light and pH usages [57].

Crocetin is a hydrophobic and fat-soluble molecule. This molecule is a conjugated polyene dicarboxylic acid [58].

Crocin dye has a valuable place among the glycoside carotenoids. Crocetin in crystalline form has a melting point (285 °C). In this plant, there are the red needles, whilst this in solution media is a yellow colour [59].

The carotenoids in saffron were extracted by several solvents for example chloroform, acetone, ether, hexane and methanol. The methanol of these solvents provides a dark red color. This extract contains the maximum yield of crocin compound [60].

Crocetin compound is a red colored amorphous substance. This substance can be easily soluble in water and alcohol. In basic solutions, it is also soluble. And, it gives an orange-red colour. At the same time, the substance is easily soluble about to give an extensive blue colour in concentrated sulphuric acid. In the reaction of crocetin and lead acetate, a deep brown precipitate is obtained [61].

Acid dyes are categorized in terms of direct dyes. These dyes are used in the colouring of fabrics as nylon and wool. The saffron dye has an acidic characteristic property. The pH values have acidic range which it has 4.5-5.5. With using tannic acid, the fastness can be improved after dyeing [62].

The crocin dye (in saffron) is rather powerful compound. One part of this compound dissolves in 150.000 parts water. This solution has a bright yellow colour [63].

Due to its high solubility, the crocin has a broader application in terms of a dye by comparison other carotenoids. It was initially discovered by Solomon and Carrar in crystal form [46].

Saffron is a yellow dye which is obtained from the stigma. This stigma contains crocin compound. The crocin is soluble in water. It is the gentioboside of crocetin [64].

Saffron dye molecules may cause the coloration of fabrics. In these molecules, colour is owing to the existence of long conjugated double bond [65].

The carotenoids can be synthesized by means of only plants, fungi and prokaryotes. These compounds are also named as "tetraterpenoids" [66].

Crocetin is an aglycone of crocin. This aglycone is occurs naturally in the saffron dye plant [57].

Acid dyes are carried out of an acidic environment. The dye compounds can have either sulphonic or carboxylic group(s). These groups can create an electrovalent bond by means of amino groups related to wool and silk [67].

Some selected studies

In a study reported by Basker and Negbi in **1983**, the use of saffron (as a dye) was mentioned. Besides, in this study it was said that saffron was a valuable dye source in ancient Greece. In addition, saffron was utilised to dye the wedding robe in Rome [11].

In a study published by Tsatsaroni and Eleftheriadis in **1994**, cotton and wool fabrics were dyed by means of the aqueous extract of saffron. The natural dyeings were obtained with and without the mordants of metal salts. Their fastness and colour values were examined [3].

In a study published by Liakopoulou-Kyriakides et al. in **1998**, cotton and wool fibres were dyed with saffron colorants. In the dyeings, the enzymatic effect was evaluated. On the other hand, the natural dyed cotton and wool samples were also studied in terms of wash and light fastness [60].

In a study reported by McNab et al. in **2004**, it appeared that the reference to saffron usage dated back to Egyptian times. It was mentioned that saffron was quite popular in Persia in Classical times. But weld (*Reseda luteola* L.) replaced the saffron dye plant. Because weld presented improved fastness properties and was a cheaper dye source [68].

In a study published by Kamel et al. in **2009**, cotton fabrics were dyed with the aqueous saffron extract. For the dyeing of cotton fabrics, two methods (traditional and ultrasonic methods) were used. The colour strength obtained by the ultrasonic method was the better compared to the traditional method [69].

In a study published by Jarosz et al. in **2009**, saffron dye ingredients were detected by HPLC. This chromatographic detection technique produced an important role for the identification dyes present in saffron. The presented technique permitted the separation and determination of *trans*- and *cis*-isomers of crocins [9].

In a study published by Varella et al. in **2009**, the dye characterisation including saffron dye plant was realised. Its main colorants were found to be crocetin, crocin, carotenes, lycopenes, and zeaxanthin. Light and washing fastness of wool and cotton dyed directly with saffron were investigated. Mordants used before dyeing wool and cotton samples were $FeCl_3$, $SnCl_2$, $K_2Cr_2O_7$, $ZnCl_2$, $CuSO_4$, and $KAl(SO_4)_2$. The values obtained with $CuSO_4$ used in dyeing wool samples with saffron dye plant were higher in terms of light and washing fastness than the values obtained with other mordants. K/S values were determined for wool (1.7724) and cotton (1.0568) samples directly dyed with saffron. Colorimetric data of wool and cotton samples mordanted with different mordants were presented [70].

In a study reported by Abdel-Kareem in **2012**, the natural dye sources used in different historical periods of Egypt were investigated. In this sense, the saffron dye plant was also mentioned in the study. The obtained colours depending on mordants using in terms of the four different references in the part of saffron dye of the study were exhibited [71].

In a study reported by Mortazavi et al. in **2012**, saffron petals were used to dye wool fibres. For the dyeing of fibres, different dyeings were performed by means of diverse mordants. Iron (II) sulfate and sodium dichromate mordants were used to improve the fastnesses (wash and light). These mordants were the best mordants among the mordants used in the study for this fastness [72].

In a study reported by Raja et al. in **2012**, saffron was used to dye pashmina shawl. The aqueous extract was obtained for this dyeing. In the dyeings with saffron extract, three different mordants were used. At the same time, the dyeings were also realised as without mordant. pH was conducted at 4-5 or 7-8. The light and washing fastness values were easily obtained. The L^* values were increased for the all dyed fabrics with washing with detergent. The maximum L^* value (78.77) was obtained for the dyeing at pH 7-8 (mordanted with aluminium sulphate, then washed). The maximum K/S value (1.986) was achieved with using stannous chloride mordant at pH 4-5 [73].

In a study published by Bathaie et al. in **2014**, the saffron colorants were investigated and discussed. At the same time, the saffron was worked as a dye. In the colouring of textiles, the saffron was also mentioned [10].

In a study reported by Vishkulli et al. in **2016**, the aqueous saffron extract was obtained to dye cotton fabrics. For mordantings, two mordants ($Fe_2(SO_4)_3$ and $Al_2(SO_4)_3$) were used. The fabrics were dyed with colouring saffron extracts of 2.5 % and 5 %. The dyeings were achieved without and with mordant. Absorbance and transmittance values related to the dyed fabrics were evaluated [74].

In a study published by Hylli et al. in **2016**, it could be said that cotton and wool yarns were dyed by means of saffron dye plant. Mordantings were performed via two types of mordants. These mordants were iron (II) sulfate ($Fe_2(SO_4)_3$) and copper (II) chloride ($CuCl_2$). The saffron extract for dyeing was obtained by water. As a dye source, the petals of saffron were used to colour the yarns. The dye plant was utilized at 2.5 % and 5 % rates. Three types mordanting procedures were applied. In addition, the values of absorbance and transmittance were examined for the saffron-dyed yarns. On the other hand, the colour fastness to washing and perspiration were investigated. The saffron dye plant cultivated in Albania was acceptable to dye the wool and cotton yarns [75].

In a study reported by Çınar and Önder in **2019**, a review about the cultural heritage of Anatolia : Crocus sativus L. (saffron) was realised. In this work, the chemical content of saffron was also mentioned [58].

In a study reported by Ali et al. in **2019**, wool fibres were pre-treated with chitosan and nano chitosan and then dyed with saffron. For this procedure, the microwave heating method was used. The treated wool fibres have a higher colour strength compared to the untreated fibres. At the pH of 5, nano chitosan treated wool fibres have the highest K/S value compared to the others. The fastness properties were also investigated. In addition, the development in properties of wool fibres was also evaluated [76].

In a study published by Xu et al. in **2021**, the history of saffron in China was investigated. This study briefly mentioned saffron dye [32].

In a study reported by Lachguer et al. in **2021**, the coloration of wool fabrics was obtained by means of an eco-friendly dyeing with saffron dye plant. The functional groups related to the saffron extract were investigated by the FTIR spectroscopy. The dyeings were obtained without and with the mordant. The colour analyses of the natural dyed wool fabrics were also performed. In addition, the effect of dye concentration, pH, temperature and dyeing time was also investigated. This investigation was realised on the colour strength. In the dyeings, colours from brown to green were obtained [77].

In a study reported by Wenger in **2022**, the history of saffron was investigated. In this review, the saffron usage as a dye source (paintings, textiles, carpets, etc.) was also mentioned [78].

In a study published by García in **2022**, a part related to saffron dye plant was mentioned [16].

In a study reported by Sadeghi-Kiakhani et al. in **2023**, saffron and weld dye plants were used to colour nylon fabrics. Therefore, the colorants were obtained from the plants. For the mordanting of nylon fabrics, different metal salts were used. The use copper salt as a mordant for the pre-mordanting enhanced the colour strength and fastness properties. The extraction from the plants was acquired by means of various solvents. For saffron, the important successful solvent used in the study was acetone. The parameters effecting the dyeing properties were also investigated. An extraction method was utilised and the some parameters (temperature, the solvent type, etc.) were varied and optimized [2].

Conclusions

In this study, the colouring property of the saffron dye plant and the dyes (crocin and crocetin) present in saffron are given. Extracting dye from saffron is an environmentally friendly process. For the extraction process, water is the solvent most commonly preferred. It must be said that saffron has the ability to dye fabric much more than its own weight. It can also be said that saffron has historically been a more valuable plant in natural dyeing compared to other dye sources due to its economic value. If we consider the use of lake pigments produced from the saffron plant, for example in paintings, there is no doubt that they will add more value to the relevant work of art than those obtained from other dye sources.

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Conflict of Interest

There is no conflict of interest in the publication of this article.

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الزعفران في الصباغة الطبيعية

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المستخلص

في الوقت الحاضر، الدراسات العلمية المتعلقة بمصادر الصبغة الطبيعية في جميع أنحاء العالم واسعة النطاق للغاية. وفي هذا السياق، يشير الصباغة الطبيعية إلى الأصباغ الطبيعية في الألياف أو إلى محتوى مصدر الصبغة. في هذا العمل، تم التحقيق في نبات صبغة الزعفران. يُطلق على هذا النبات اسم *Crocus sativus L. (Iridaceae)* باللاتينية. مصدر الصبغة في النبات هو وصمة عاره. الزعفران هو أحد أغلى الأصباغ في العالم. المواد الملونة الموجودة في النبات هي الكروسين والكروسيتين. يمكن استخدام هذه المواد الملونة في الصباغة الطبيعية. كما يتم التحقيق فيها من حيث خصائص الصباغة الخاصة بها.

الكلمات المفتاحية: الزعفران؛ الألياف الطبيعية؛ اللون؛ الأصباغ؛ الكروسين؛ الكروسيتين.