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OPTIMIZATION OF DRYING TEMPERATURE OF SAFFRON PETALS AND USING THEM AS A COLORANT IN CELLULOSIC FIBERS WITH ALUMINIUM SULPHATE AS MORDANT

Abstract

Recently the awareness of the demand on eco-friendly dyes in textile applications is increased since the natural dyes can reveal better biodegradability and generally have a higher compatibility with the environment. As natural dye, saffron petals are used in this study for dying cellulosic fabrics. Crocus sativus (saffron) has cyanic color flowers with major colorant of anthocyanin. Attractive color and functional properties of anthocyanins make them a good substitute for synthetic pigments in the textile industry. These natural soluble water colorants are rather unstable and influenced by final processing treatment. The drying process is critical to the stability of saffron petals anthocyanins. The saffron flower waste was dried and grounded into small and fine particles. Anthocyanin was extracted and the measurement of her absorbance was done. The results showed that the highest amount of anthocyanin was obtained when saffron petals were treated by moderated temperatures. The natural dye was extracted by aqueous method at boiling conditions. The extracts were then applied on cotton fabric with and without the use of aluminium sulphate as mordant. The color of cotton fabrics dyed without mordant and with mordant was estimated and was observed that the intensity of color varies. It was pointed out that the color was more intensive when the dyeing process was done in presence of mordant. To determinate the amount of the color absorbed by fibres, was carried out the measurement of

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the absorbance from the solutions taken from each dyeing process. These preliminary results are helpful information for the use of saffron flowers as a natural dye in textile industry.

Key words: saffron petal, anthocyanin, color stability, drying treatment, cotton fabrics, mordant, dyeing.

1. Introduction

Chemical processing is irrevocable for textile coloration. During textile processing huge amount of various essential dyes and auxiliary chemicals are habitually consumed. So, in keeping with volume and composition, effluent of textile plants are most polluting amongst all industrial sectors. This huge amount of toxic and hazardous wastewater is finally discharge into the rivers, canals and water streams resulting in adverse effects on flora and agricultural land and thus presumed as one of the major sources for environmental pollution.

The specialists around the world are attempting to build up a cleaner innovation and ecologically supporting methods of cotton coloring for conforming to continuously requesting ecological regulation and to save water, energy and time. Sustainable textile coloration is possible either by using green ingredients or zero discharge of wastewater.

In this regards natural dyes are eco-friendly, safe, cheap, need no special care, uncommon and soothing shades, wonderful and rich in tones, act as health cure, have no disposal problems, non-carcinogenic, non-allergic, non-toxic, easily biodegradable, require simple dye house to apply on matrix and mild reactions conditions are involved in their extraction and application. There are various potential sources such as plants, insects/animals, microbes, minerals and renewable bio resource products.[1] Among a lot of sources, historically, saffron was particularly important as a dye plant.

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Saffron is a perennial plant with the botanical name of Crocus Sativus Linn which belongs to Iridaceae family. This plant is cultivated mainly in warm countries like Iran, Morocco, Egypt, Israel, Spain, Italy, Greece. [4,5] And recently is also successfully cultivated in Albania for the production of flowers. A spice made from the dried stigma of the flowers is considered as the world's most expensive spice. The spice has culinary and medicinal properties. The red stigma of the flower is harvested in the morning when the flowers are open. The collected stigma is then dried and converted into powder. Approximately 1 kg of flower is required to produce 12 g of dried saffron spice. After harvesting the stigma from saffron flower, the petal part of the flower which is violet in colour is thrown as a waste. [7]

Saffron (Crocus sativus) is produced largely in Iran, with more than 90% of total annual saffron production in the world. [5,6] It has cyanic color flowers with major colorant of anthocyanins. [6]

Anthocyanins are a group of naturally occurring phenolic compounds, which play an important role in the color quality of many flowers, fruits, vegetables and related products derived from them. Recently, anthocyanins were reported to have important biological activity, presenting antioxidant, antimutagenic, anticancer and antiobesity properties, as well as reducing the risk of coronary heart disease. Besides their biological activities, the bright colour of anthocyanins (orange, red, purple, blue), ensures a high potential of being used as natural dyes as an alternative to synthetic colorants. The colour of anthocyanins depends essentially on the chemical structure of different forms in which they can be found, these structures are strongly related to the pH value of the solution. For example, the flavilium cation I (red colour) is the predominant species at pH = 1, while at pH values between 2 and 4, the quinoidal blue base II predominates. However, due to their high reactivity, anthocyanins easily convert to colorless or undesirable brown degradation compounds. Among many factors that can influence anthocyanin stability, the most significant is temperature. So in this point of view, in this study is observed their stability in function of heating temperature. [2,3,6] Anthocyanins of petal extract can be used as a natural resource of colorants in textile industry.

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Dyes are substantive or adjective. Substantive dyes are absorbed and fixed by chemical bonds within the fibers without further chemical treatment. However, most natural dyes are adjective dyes and need the use of mordants to help their absorption and fixing on fibers. Metal salts act as chemical bonds between the dye molecules and the functional groups of the fibers, and generally change the color produced by the dye. [8] Mordants play very important role in imparting color to the fabric. The mordants used in combinationin different ratios gave varying shades. Better colour strength results are dependent on the metal salt used.

Functional groups such as amino and carboxylic acid on the fiber can occupy the unoccupied sites on interaction with the fiber. Thus, a ternary complex is formed by the metal salt on which one site is with the fiber and the other site is with the dye. The chromatophore of the dye makes it resistant to photochemical attack, but the auxochrome may alter the fastness. The resistance of a dye or pigment to chemical or photochemical attack is an inherent property of the dye chromophore.

In present study, an attempt has been made to utilize the petal part of the saffron flower which is considered as a waste to extract dye for application on cotton textile. The utilization of the saffron flowers waste will add the value to the saffron growers.

According to the importance of anthocyanins, as well as their instability and sensitivity, the present study aimed also to investigate the effects of drying processes on the stability of anthocyanins of saffron petal.

2. Materials and methods

2.1. Materials

The saffron flowers after harvesting their stigma were collected from Dumrea area, Elbasan, Albania in November 2016. This process is done by hand at room temperature.

Cotton was from department of Textile and Fashion and aluminium sulphate was used as mordant.

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2.2. Dehydration process

The dehydration of samples (30 g of fresh petals) was carried out in thermostat. For drying process were used different temperatures (40, 60, 80, 100 and 120 ^{o}C) in different time, respectively (120, 90, 40, 30 and 20 minutes) using memmert thermostat-BD 240 08-49972. All the experiments were repeated three times and the tray load was the same in all experiments.

2.3. Extraction of anthocyanin

Saffron petals were grinded in small particles. For extraction of anthocyanin from saffron petals 0.1 g of dried saffron petals were mixed with 250 ml of hydrochloric acid solution of 0.1N in a dark colored bottle. After five minutes in 25 ^{o}C ± 1, the samples were filtered through a filter paper (Whatman no. 2) using a funnel. For determination of anthocyanin, were carried out with a 1 cm pathway cell on a UV-1200 spectrophotometer the measurements of absorbance of the filtered solution at 516 nm.

2.4. Dye extraction

The dyeing matter was prepared by taking 20 g saffron and 800 ml tap water. When the flask started to boil, we left it for 60 minutes in this condition. Then the solution was cooled down in room temperature and was filtered to obtain the dyestuff. Water was added until 800 ml.

2.5. Dyeing process

The dyeing process was performed in a dye bath with a bath module 1:75 by exhaustion method in aqueous conditions. The fabric samples were boiled for 60 minutes with the dye and the dye and the mordant together in dyeing bath.

2.6. Color measurement

Color measurement and dye absorption of cotton were evaluated by measuring absorbance with UV-1200 spectrophotometer. It was measured the absorbance of the solution after the

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dyeing process remained in the dyeing bath. In other words we measured the dilution of dyeing bath as an indirect indicator of the amount of the dye absorbed from the fibre. If the value of absorbance is lower this means that the amount of coloring solution taken from the fibre is higher.

3. Results and discussion

4.

4.1. Effect of temperature in anthocyanin stability

Heat treatment is one of the most important factors that influence anthocyanin stability as they are not stable at high temperatures. Saffron petals were heated in thermostat, drying at five different temperatures (40 °C, 60 °C, 80 °C, 100 °C and 120 °C respectively) in order to investigate the degradation temperature of anthocyanin. From Figure 1, it was observed that the stability of saffron petals anthocyanins gradually accessed with increase of the heating temperature and with a maximum peak at 60 °C and had decrement of heating time until 120 °C. The results suggested that saffron petals anthocyanins tended to degrade at high temperatures. Anthocyanin degradation rate increases as temperature rises during processing and storage. According to the results (Table 1), saffron petals treated in 60 ºC contained the highest amount of anthocyanin after drying according to the temperature instability of anthocyanins. According to literature all the measurements for anthocyanin extract must be done around 518 nm, but for our measurements all the measurements of absorbance were done at 516 nm. That's because our anthocyanin extract has a broad peak with maximum peak at this number of wavelength, as it is shown in the graph below.

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Table 1. Effect of drying process on stability of saffron petals anthocyanins

Sample no.	Drying temperature (ºC)	Drying time (min)	Absorbance value of anthocyanin
1	40	120	0.077
2	60	90	0.244
3	80	40	0.194
4	100	30	0.185
5	120	20	0.157

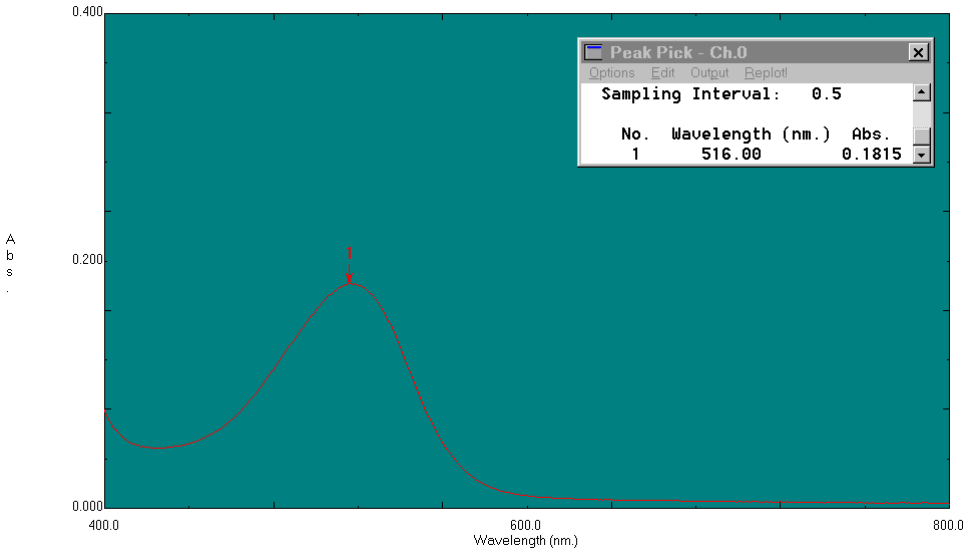

Figure 1. Absorbance curve of anthocyanin extract. λ_{max}= 516 nm.

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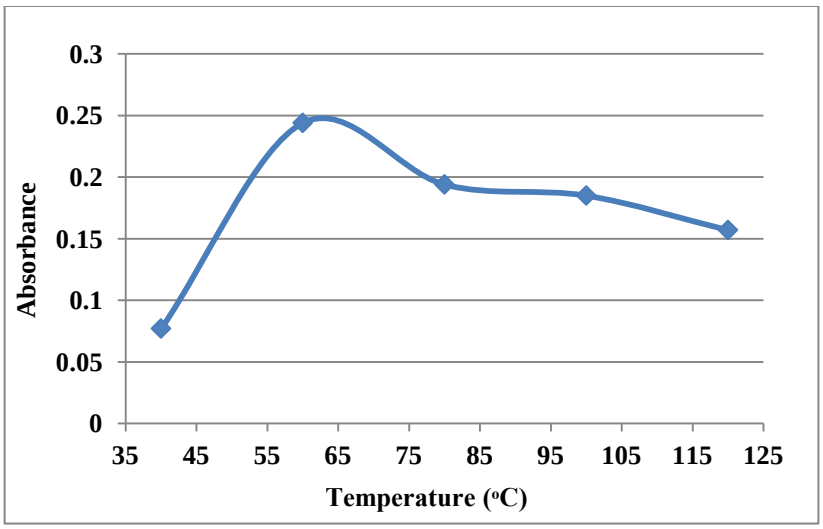

Figure 2. Effect of heating on stability of saffron petals anthocyanins.

4.2. Colour

Cotton samples colored with saffron coloring solution with and without mordant are presented in figure 3.

As it can be seen visually the white cotton fabric sample is colored into two new yellow colors that differ from each other by shadow. The light yellow is for dyeing with coloring solution of saffron and the dark yellow is for the dyeing with coloring solution of saffron but in presence of aluminium sulphate as mordant. The presence of aluminium sulphate as mordant makes the binding of the dye more stronger with the fibre and also gives resistance to the color as it can be seen in the figure below. According to literature, this is because of aluminium ions of the mordant that acts as electron acceptors for electron donors to form coordination bonds with the dye molecule, making them insoluble in water and prolong in this way also the resistance of the color.

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Figure 3. Color hue of cotton samples

To have not only qualitative results but also quantitative ones, were done the measurements of absorbance of the colouring solution that remained in the coloring bath after the dyeing process. The results obtained are shown below in table 2 and are presented in the figure 3 as a comparable graphic. All the measurements of absorbance were done at 330 nm wavelength. The blank probe didn't shown any absorption peak in this wavelength, while the colouring solutions showed high peak at this value, (as shown in figure 5). So by this way the blank probe didn't interfere the absorbance values of our samples during the measurements since it's absorbance was zero at 330 nm. From the table and the graph below it, it is seen that the absorbance value tends to decrease from A to C and the lower value is obtained in C case, where the dyeing is done in the presence of aluminium sulphate as mordant. This means that the higher is the amout of the coloring solution taken from the fibre, which corresponded also with the photo presented in figure 3.

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Table 2. Absorbance values

Sample	A	B	C
Absorbance	1.578	1.573	0.878

A- Coloring solution

B- Coloring solution without $Al_2(SO_4)_3$

C- Coloring solution with $Al_2(SO_4)_3$

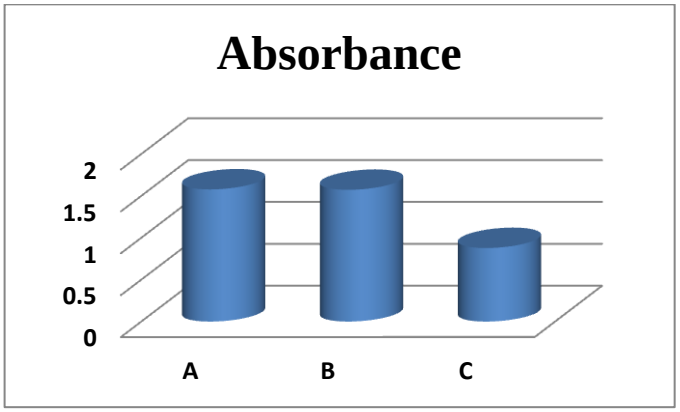

Figure 4. Absorbance values for each sample

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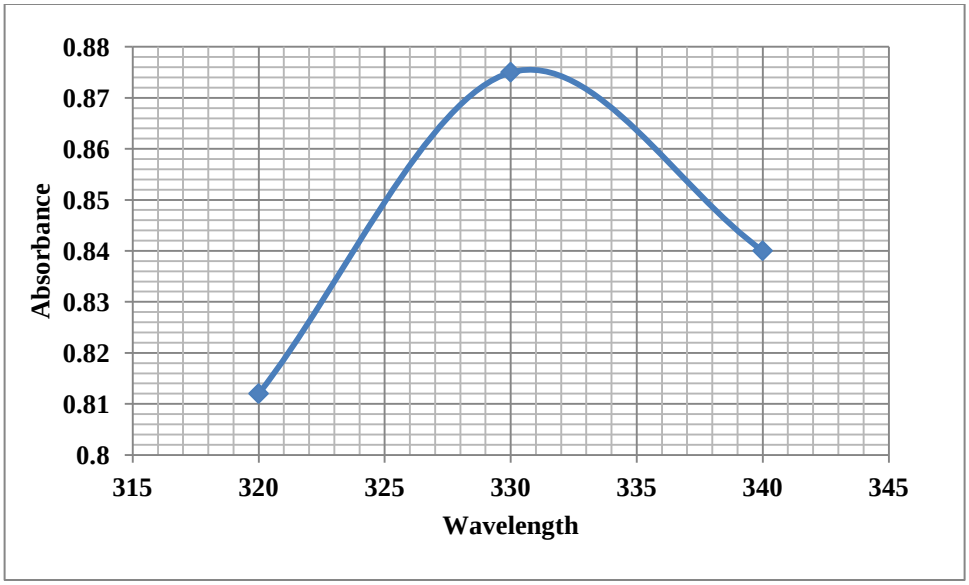

Figure 5. Absorption curve of saffron solution. λ_{max}= 330 nm.

5. Conclusions

The trend to use natural dyeing is growing because they are biodegradable and friendly with the environment. Fresh saffron petals are used in this study to determine the most favourable way how to dry them and the dried saffron petals were used as a dye for cotton fabric samples. The results showed that the highest amount of anthocyanin was obtained when the saffron petals were treated in 60 ºC. This is also true according to the temperature instability of anthocyanins reported in literature. According to the results the stability of saffron petals anthocyanins gradually accessed with increase of the heating temperature with a maximum peak at 60 ºC and had decrement with heating time until 120 °C. The dyeing results showed that saffron petal can give different shades according to the way of colouring. Here is valuable to mention also the effect of the mordant used in this study, in the hues of color. So overall, saffron can be a good natural dyestuff for cotton dyeing, but need more experiments and study for further details to use them for dyeing different fabrics.

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