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Optimization of saffron drying parameters to achieve a higher drying rate and improved product quality

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ABSTRACT

The optimization of the saffron drying process parameters significantly affects the quality of the final product. In this article, the saffron essential oil is extracted and characterized, and fresh and dried saffron quality is compared. The impact of parameters such as drying temperature, light source during the drying process, and the use of vacuum in the drying process on the rate of drying and color intensity of dried saffron is investigated. The results obtained from GC-MS, FT-IR, and UV-Vis tests indicate that over 60% of the concentration of the essential oil derived from saffron consists of 14 main compounds, among which safranal and picrocrocin are the most significant. Throughout the drying process, the quality of the dried saffron does not change significantly, and a drying temperature of 120°C and the absence of direct light improve the quality of the dried saffron. Additionally, a temperature of 70°C is the optimal temperature for drying saffron under 0.3 atmospheric vacuum conditions.

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1. Introduction

Saffron, also known as red gold, consists of the dried red stigmas of the *Crocus sativus* plant, and it belongs to the iris family. It is one of the most valuable agricultural and medicinal products in the export of Iran, as well as being a precious spice worldwide [1, 2].

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Saffron may have different industrial applications in the production of different products like traditional medicine, edible additives for coloring and flavoring food, and dyeing textiles [3, 4]. The famous active compounds in saffron include crocin (the agent responsible for saffron's reddish-yellow color), picrocrocin (the compound that imparts the bitter taste to saffron), and safranal (the predominant volatile compound in saffron) [5, 6]. In addition, saffron possesses medical applications such as sedative, anti-tumor, antidepressant, and mood-enhancing properties [7, 8].

Among various agricultural products, saffron is one of the valuable crops that globally holds a unique position. Iran, with the production of over 200 tons per year, and accounts for approximately 90% of the cultivated area and 93.7% of global production, is the largest producer of saffron in terms of both quality and quantity in the world [9-11].

After saffron cultivation, product processing is the most important topic of interest in the marketing process because the processed saffron can be exported to target markets in various countries. It can be said that saffron processing is one of the main parts of the revenue-generating process in this field. Drying is the most crucial step required for saffron processing. Proper drying of saffron not only prevents spoilage and deterioration but can also enhance product quality and increase consumer satisfaction [12].

The saffron drying process has been studied by various researchers [12]. For instance, Atefi and his coworkers [13] investigated the effects of freeze-drying, electric oven drying, and shade drying on the chemical and physical properties of saffron. According to their results, the freeze-dried and shade-dried samples exhibited the highest and lowest color intensity, respectively. Furthermore, these researchers demonstrated that the freeze-dried sample contained the least amount of safranal, while the sample dried in the electric oven had the highest amount of safranal.

Hemati Kakhki dried saffron by using four different methods, including two traditional Iranian and Spanish methods, and two industrial methods of vacuum drying and cabinet drying (using hot air). They showed that drying saffron with a cabinet dryer at atmospheric pressure better preserved the main characteristics of the product, such as color, flavor, and aroma. The Spanish method, vacuum dryer, and traditional Iranian method ranked next in terms of preserving these attributes, respectively [14].

To achieve a product with desirable physical and chemical properties and a higher shelf life, this study aimed to optimize the saffron drying process and provide a suitable alternative to traditional saffron drying methods. Additionally, the effects of various parameters such as oven

temperature, drying time, light intensity, and drying method on the drying rate and quality of the final product were examined.

2. Materials and Methods

2.1. The Used Materials

The saffron flowers used in this study were collected from the fields surrounding Nishabur County (Miangolgeh, Zammeh village, Iran). Figure 1 shows the various parts of the saffron flower. The saffron stigmas were separated manually, and 0.5 grams of the separated stigmas were used for each experiment. To prevent potential errors, the samples for each series of experiments were prepared together and stored in a sealed container in a refrigerator at 4°C. Additionally, the experiments were conducted consecutively. For the collection and extraction of the active compounds present in the samples, ethanol with 99% purity from Merck (Germany) was used in the experiments.

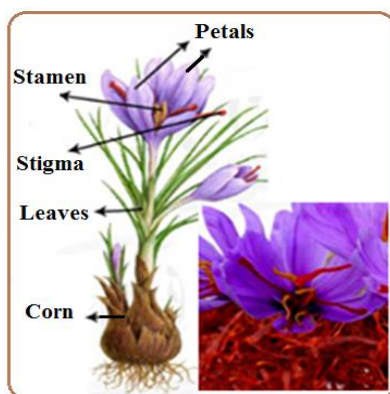


Fig. 1. Different parts of saffron flowers.

2.2. Experimental Method

In this study, various methods for the drying of saffron were employed, and the effect of the change of each parameter on the characteristics of the dried product was examined with constant values of other parameters. An electric oven (AV 35, Hakim Azma Taghiz, $200 \pm 2^\circ\text{C}$) with a capacity of 40 liters and a power of 1500 watts was used to investigate the effects of temperature and drying time. For each experiment, 0.5 grams of fresh saffron stigmas were precisely weighed to 0.001 grams and placed in an oven at a specified temperature. The samples were removed from the oven at specified time intervals and weighed, and this process continued until the weight of the samples stabilized.

In all of the experiments, the air temperature and the temperature of the holder plate (for drying samples) were measured by a digital thermometer and they were held constant at about 25°C .

To investigate the effect of light intensity on the characteristics of the final product, five saffron samples were placed in five different locations. 1) In a dark place (cabinet) with airflow. 2) In the shade of a room. 3) In direct sunlight. 4) In front of a 250-watt mercury vapor lamp (at a distance of 50 cm). 5) In front of an infrared lamp (60 watts, inside a desk lamp and at a distance of 15 cm). These samples were also weighed at various time intervals until they reached a stable weight.

To investigate the effect of vacuum on the drying rate of the product, a vacuum oven (TAT-2033, Hakim Azma Taghiz, 5 L, $0.1 \pm 5\%$ bar) was used. In this section, different samples were placed in the vacuum oven at a pressure of 0.3 bar and at various temperatures. At desired time intervals, the samples were removed from the oven and weighed. This process continued until the weight of the samples stabilized. The samples obtained from each experiment were stored in two forms: dried powder, and collected in ethanol, in sealed containers. Finally, they were kept in the refrigerator away from light until the subsequent experiments.

To reduce experimental error, all experiments were repeated four times, and the average results are reported in the figures. The moisture of samples was estimated by using the following equation:

$$\text{Moisture} = \frac{\text{Weight of sample} - \text{Weight of dried sample}}{\text{Weight of sample}}$$

2.3. Characterization Methods

For characterization of the produced essential oil, gas chromatography-mass spectrometry (GC-MS), Fourier-transform infrared spectroscopy (FTIR), and ultraviolet-visible (UV-Vis) spectroscopy methods were employed.

The GC-MS experiments were conducted using an Agilent GC-MS system (7890A)-MSD(5975C). This device has a DB5-MS capillary column (30-meter length and an inner diameter of 0.25 mm). The oven temperature of the device started at 40°C for an initial time of 5 minutes. It was increased to 270°C at a rate of 10 degrees Celsius per minute, and finally held at this temperature for 10 minutes. Helium was used as the carrier gas at a flow rate of 1 mL/min, with a split ratio of 1 to 20. The injector temperature was set to 220°C, and the used detector was a 5975C VL MSD detector at a constant temperature of 300°C.

The functional groups of the compounds present in fresh and dried saffron were analyzed by a Fourier-transform infrared spectrometer (RXI, Perkin Elmer). The corresponding spectra were examined in the 400 to 1400 cm^{-1} wavelength range.

The UV-Vis spectrophotometer (Lambda, Perkin Elmer), equipped with a Peltier Temperature Programmer and LCB-Lab Teach water bath, was used to analyze the color intensity of the saffron samples during the various experiments.

After preparing and diluting the saffron solution in ethanol, the absorption process was conducted at a wavelength of 257 nanometers for the identification of picrocrocin, 330 nanometers for safranal, and 440 nanometers for crocin [13, 15-17]. The changes in the concentration of these three main compounds were examined by comparing the corresponding peaks.

3. Results

3.1. Identification of the Main Chemical Components

Identification of the main chemical components presented in the essential oil extracted from saffron (using ethanol as a solvent) was performed by GC-MS analysis and the results are presented in Figure 2. The most important components with the highest concentrations are listed in Table 1. As shown in this table, approximately 65% of the concentration of the essential oil extracted from saffron is comprised of 14 main chemicals.

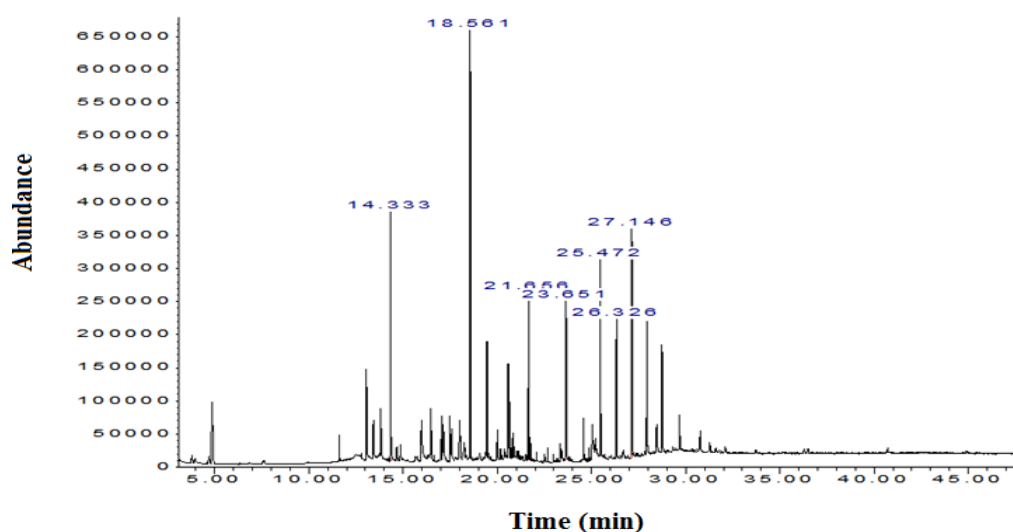


Fig. 2. Chromatogram of saffron extract by GC-MS.

Table 1. Phytocompounds extracted from saffron extract (GC-MS analysis).

Number	Name of components	A%	Chemical Formula	Retention Time (min)
1	Propane,2,2-Diethoxy	4.267	C ₇ H ₁₆ O ₂	4.856
2	2-Cyclohexen-1-one,3,5,5-trimethyl-\$Isophoron	3.434	C ₉ H ₁₄ O	13.05
3	Safranal	7.361	C ₁₀ H ₁₄ O	14.328
4	4-Hydroxy-3,5,5-trimethylcyclohex-2-enone	2.868	C ₉ H ₁₃ O	15.97
5	2-Isopropylidene-3-methylhexa-3,5-dienal	2.014	C ₁₀ H ₁₄ O	17.478
6	2,6-di(t-butyl)-4-hedroxy-4-methyl-2,5-cyclohexadien-1-one	2.492	C ₁₅ H ₂₄ O ₂	17.999
7	Picrocrocin	10.626	C ₁₆ H ₂₆ O ₇	18.559
8	Hexadecane	3.153	C ₁₆ H ₃₄	19.45
9	Heptadecane	2.416	C ₁₇ H ₃₆	20.576
10	Octadecane	3.934	C ₁₈ H ₃₈	21.651
11	9-Octadecenoicacid(Z)-\$Oleicacid\$Redoil	3.034	C ₁₈ H ₃₄ O ₂	25.036
12	Docosane	4.640	C ₂₂ H ₄₆	25.468
13	Tricosane	3.601	C ₂₃ H ₄₈	26.321
14	Crocetin	11.172	C ₂₀ H ₂₄ O ₄	28.738

3.2. FTIR Analysis of the Fresh and Dried Saffron

As shown in Figure 3, to investigate the effects of the drying process on the main chemical bonds presented in saffron, FT-IR tests were conducted on saffron particles before and after the drying process. The results of this figure indicate that three main changes are observed after the drying of saffron. These changes include a reduction in the height of the peaks observed in the ranges of 3400, 1060, and 870 cm⁻¹. This decrease in peak heights occurs while other presented bands have not changed significantly due to the drying process. Based on the results reported in various references [18-22].

The peak observed in the wavelength range of approximately 3400 cm⁻¹ corresponds to the O-H groups present in water, alcohols, and phenols, and the reduction in the size of this band indicates a decrease in the concentration of these compounds in saffron due to the drying process. Additionally, the peak observed in the range of 1060 cm⁻¹ indicates the presence of C-O and O-H groups in alcohols and phenols, which have also experienced a reduction in the concentration of these chemicals.

The peaks observed in the range of 870 cm⁻¹ are associated with the C-H groups present in aromatics, which also indicate a reduction in the concentration of aromatics due to the drying process.

The peaks observed in the regions of 2850 to 3000 cm^{-1} correspond to the stretching vibrations of C-H groups present in aldehyde compounds of saffron, such as safranal. Additionally, the presence of bands in the regions of 1220 to 1390 cm^{-1} is attributed to the stretching vibrations of ester groups ($\text{O}=\text{C}-\text{O}$) found in substances like dimethylcrocin, as well as alcoholic groups in crocins and picrocins, with no significant changes observed in these peaks.

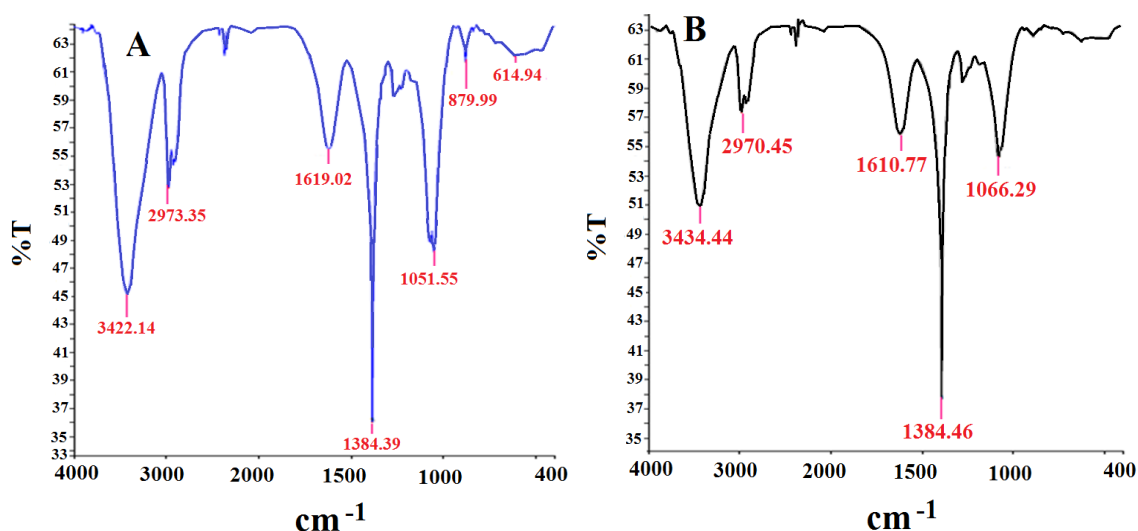


Fig. 3. A) FT-IR spectrum of fresh saffron extract, B) FT-IR spectrum of dried saffron extract at 120°C.

3.3. Effects of Temperature on the Drying Rate of Saffron

Figure 4 shows the drying time of saffron at five different temperatures of 40, 60, 80, 100, and 120°C in an atmospheric oven. As indicated in this figure, at 40°C, the complete drying of saffron stigmas approximately takes 80 minutes, whereas, at 60°C, only 30 minutes are required for complete drying, with saffron reaching over 95% of its final moisture after just 20 minutes. At higher temperatures up to 120°C, less than 10 minutes are needed for complete drying. Although the results of this section indicate that increasing the temperature significantly accelerates the drying process, the effect of temperature on the quality of the product must also be considered.

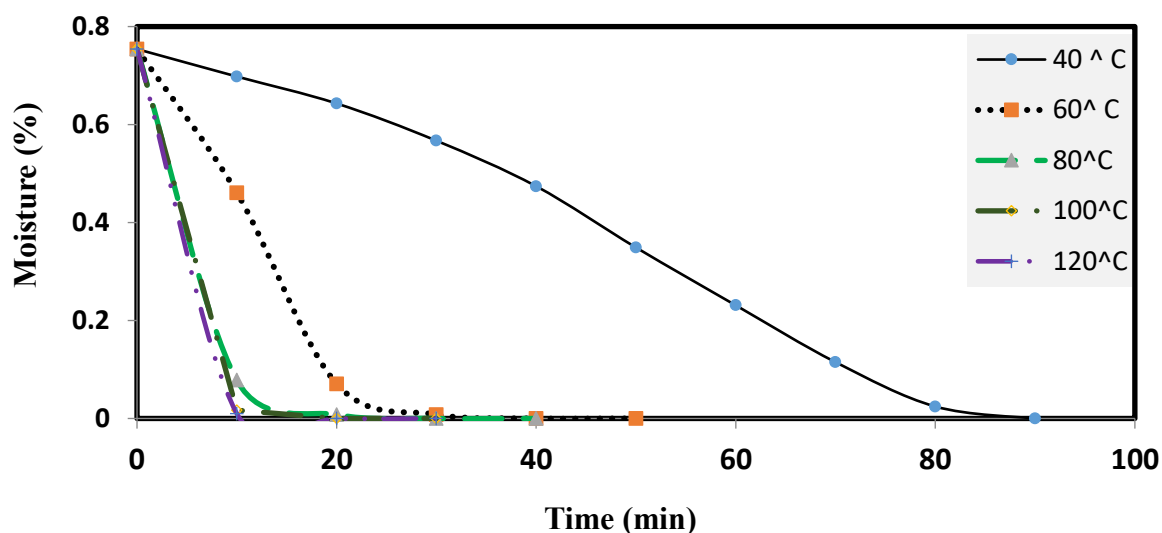


Fig. 4. The effect of temperature on the drying speed of saffron at atmospheric pressure and inside the oven (dark conditions).

Figure 5 illustrates the effect of drying temperature on the drying rate of saffron in a vacuum oven. Due to the rapid drying speed under these conditions, a temperature of 30°C was chosen as the starting temperature for the study, along with temperature intervals of 10°C.

As expected, the drying rate of saffron in a vacuum oven is significantly higher than at atmospheric pressure, which is due to the delicate and thin structure of the saffron stigmas. Figure 5 shows that at a temperature of 30°C, it takes only 45 minutes for the saffron to dry completely. In contrast, at 40°C, only 30 minutes are required for complete drying. It is also evident that the drying time decreases with increasing temperature.

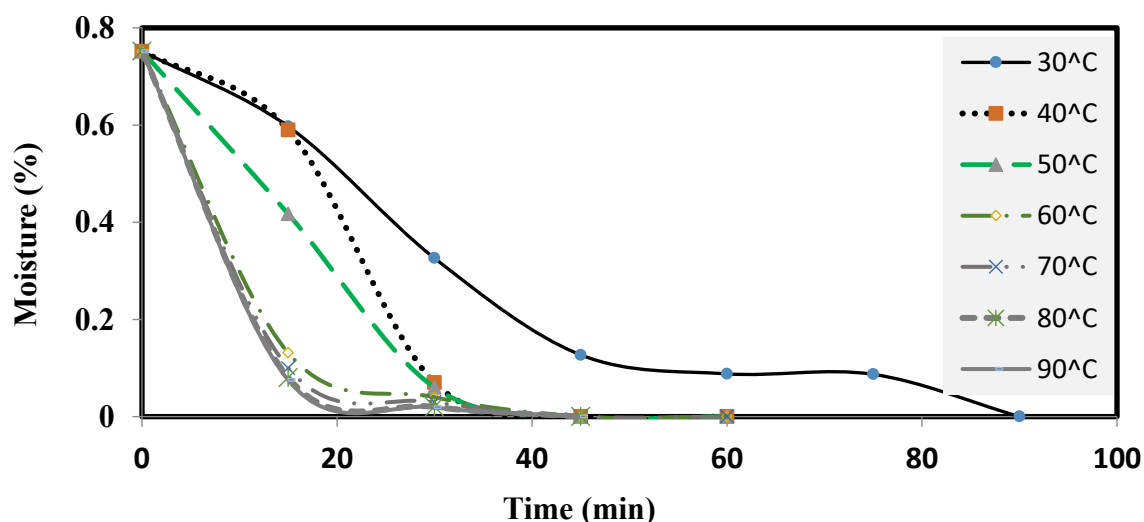


Fig. 5. The effect of temperature on the drying speed of saffron in a vacuum oven at a pressure of 0.3 bar.

3.4. Effects of the Light Source on the Drying Rate of Saffron

Figure 6 illustrates the effect of light intensity on the drying rate of saffron. According to the results shown in this figure, light has a significant impact on the drying speed of saffron. Drying saffron in a dark cupboard or the shade outdoors (at 25°C and approximately 15% relative humidity) requires over 350 minutes, whereas under sunlight, mercury lamp light, and infrared lamp light, the drying times are 90, 40, and 15 minutes, respectively. As the light intensity increases, the thermal energy supplied to the saffron also significantly increases, leading to an enhanced drying rate. As observed, the drying speed increases with the transition from shade to direct sunlight and it increases more by using the light emitted by the mercury vapor lamp. Additionally, the use of infrared lamps accelerates the drying process due to the radiant power of infrared rays that cause heating.

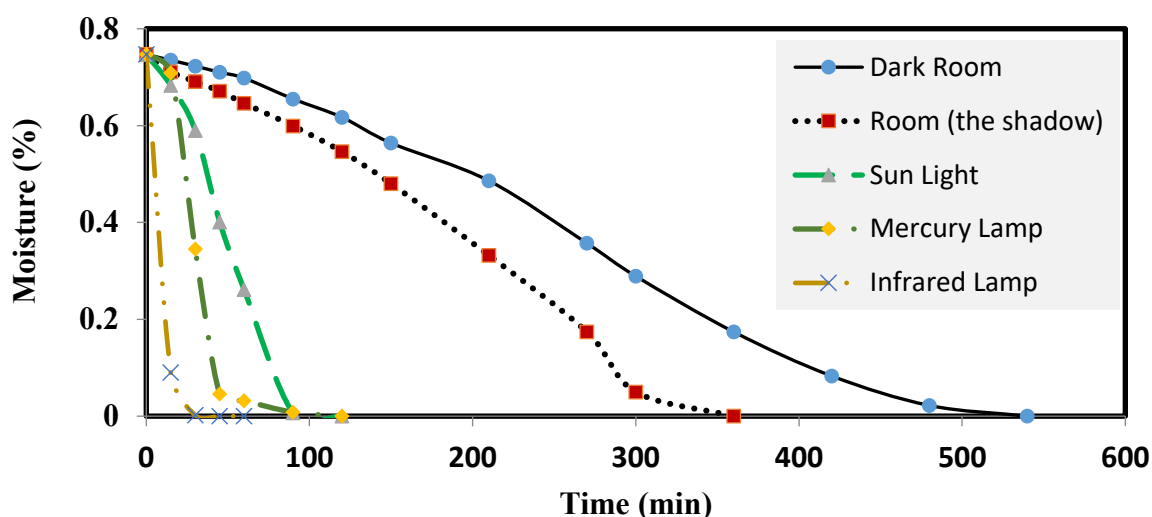


Fig. 6. The effect of a light source on the drying speed of saffron in atmospheric conditions.

3.5. Effects of Drying Temperature on the Color Intensity of Dried Saffron

To investigate the effect of drying temperature on the color intensity of dried saffron, the samples extracted in ethanol were analyzed using a UV-VIS experiment. The results are presented in Figure 7. Based on the reported results, as the drying temperature increases from 40 to 120°C, the concentration of coloring compounds in saffron essential oil increases. This indicates that higher temperatures enhance the extractability of the compounds present.

This phenomenon may be due to the increased degradation of the vascular tissues within saffron, as the destruction of these tissues intensifies with rising drying temperatures, allowing the essential oil particles to exit more easily and quickly from the plant vessels into the solvent. Moreover, by increasing the drying temperature, the drying time decreases sharply and it may cause a decrease in the evaporation of volatile compounds.

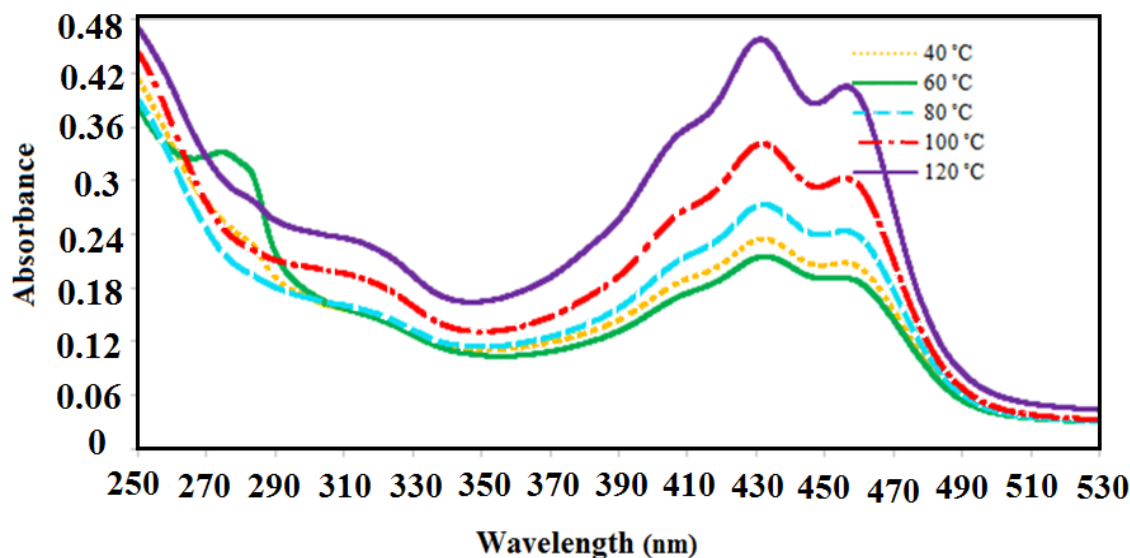


Fig. 7. The effect of oven temperature on the color intensity of saffron essential oil.

Raina et al. [23] demonstrated that increasing the temperature of the electric oven from 20 to 40°C resulted in an increase in crocin concentration; however, further increases in temperature led to a decrease in crocin levels.

Akhondi and his coworkers [24] reported a decrease in crocin concentration at temperatures above 65°C and an increase in safranal concentration at higher temperatures with shorter drying times by using an electric oven.

According to the results of Delshad et al. [25], increasing the temperature up to about 50°C resulted in a slight increase in the concentration of crocin, while temperatures above that led to a decrease in crocin levels. Furthermore, increasing the temperature to 55 degrees Celsius improved the level of picrocrocin. Additionally, prolonged drying times at the highest temperature level (70 degrees Celsius) led to a decrease in picrocrocin, and extended drying times at elevated temperatures in the electric oven resulted in a reduction in safranal levels.

3.6. Effects of Light Source on the Color Intensity of Dried Saffron

Light is composed of electromagnetic waves that can penetrate various surfaces up to several millimeters and ultimately be converted into heat. The effect of light on color intensity and concentration of coloring compounds in dried saffron is shown in Figure 8. As observed in this figure, the highest quality of dried saffron corresponds to products dried in darkness and shade. When using high-intensity light sources such as sunlight or mercury vapor lamps, the high intensity of the emitted light degrades the active compounds in saffron, resulting in a significant reduction in the quality of the obtained essential oil.

The results of this figure demonstrate that to preserve the quality of saffron, it should be dried in darkness or shade and away from direct sunlight.

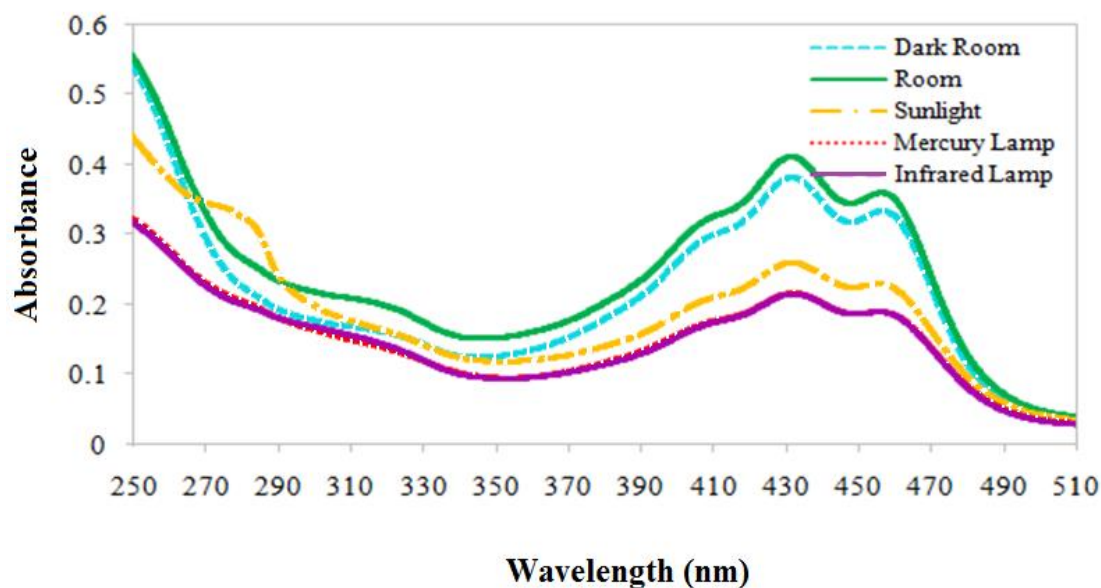


Fig. 8. The effect of light source on the color intensity of dried saffron.

4. Conclusion

Appropriate processing of saffron is needed to produce better saffron with higher added value. The drying process of saffron is considered one of the most important aspects of saffron processing, which is examined in this article. The drying temperature is one of the main studied parameters which was evaluated within the temperature range of 40 to 120°C.

The results of the experiments indicate that with increasing drying temperature, the drying rate of saffron has significantly increased, and therefore, the drying time decreased sharply. Additionally, the rise in drying temperature has improved the quality of the product, resulting in an increased concentration of coloring agents. Conversely, an increase in light intensity during the drying process has significantly reduced the concentration of the main coloring compounds present in saffron essence.

By examining the saffron drying process under vacuum conditions, it was found that although, as previously expected, increasing the temperature and vacuum drying significantly enhance the rate of drying, various factors such as temperature and drying rate have different effects on the quality of the dried product. Overall, the best product quality under vacuum conditions was achieved at a temperature of 70°C.

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Conflicts of Interest The authors declare no competing interests.

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