

Melatonin for improving grapes quality of “Halawani” cultivar

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DOI: <https://doi.org/10.36077/kjas/2026/v18i3.15381>

Received date : 8/3/2024

Accepted date: 13/5/2024

Abstract

Grapevines are among the most commonly grown fruits on Earth and are also the most consumed fruits in the world. The nutritional and economic values of grapes are commendable, as are the consumption of the fruit itself or its derived products, such as juices, wines and raisins. Melatonin, a hormone typically associated with the control of sleep in humans, also has a significant role in the process of fruit ripening. The current study was intended to investigate the effects of the foliar application with melatonin on enhancing the grapes quality. The commercial melatonin was foliarly applied in three concentrations (0, 3 and 6 mg/L) at three different phases: upon flowering, at pre-veraison phase and at veraison phase. Fruits were gathered and assessed based on their physical and chemical properties. The findings showed that melatonin application had a significant effect on the grape chemical properties as measured by grape flesh and seeds contents of TSS, total acidity, total sugar, anthocyanin, antioxidants, phenols, flavonoids and resveratrol. Melatonin at 6 mg L⁻¹ was the most effective and resulted in significant increases of fruit and seed contents of total sugar (9.25 and 9.59%), anthocyanin (16.16 and 16.99%), phenols (6.37 and 6.31%), flavonoids (16.64 and 16.15%), and resveratrol (10.7 and 9.99%). Further research is necessary to fully comprehend the intricate relationship between melatonin and bioactive compounds, this would lead to the development of strategies to increase the amount of melatonin in grapes.

Keywords: Grape, melatonin, fruit quality, anthocyanin.

Introduction

Yield regulation and homogeneity of grapes maturity are important for commercial qualities and productivity. The period from flowering to maturity is the production determinants, which can be affected by environmental conditions, physiological

factors and agricultural practices within the vineyards (21). The yield quality and its acceptance by the consumer depend on the integration of fruit ripening, berry size, berry color and taste which are determining the marketing value especially in colored



grape varieties (23). It is well known that agricultural practices such as fertilization and treatment with chemicals and especially growth regulator hormones (GRH) for this purpose have the main influence (9).

Melatonin is a GRH which has multiple benefits and a broad spectrum of actions that are beneficial to plant growth and tolerance to both biotic and abiotic stresses (31).

The commercial GRH stimulants are increasingly used to obtain a uniform fruit color for some table grape varieties. Color fading or deficiency in colored or red table grape varieties is a main problem that grape vines growers may face. This problem is a combined to Halawani grape grown widely in Iraq that shows fluctuation in color deficiency. Approaching to solve this problem in uneven skin coloring in different table grape varieties, applying some growth regulators such as abscisic acid and melatonin has been used to improve fruit coloring and quality (20).

Melatonin has been recently reported to have a role in anthocyanins biosynthesis, which is the pigments responsible for the color in grape skins. Anthocyanins are flavonoids, water-soluble pigments that are responsible for variant fruit colors ranging from red to blue. In addition to their aesthetic role,

anthocyanins are potent antioxidants and are thought to have several health benefits (33). They are predominantly found in the skin of grapes, especially in dark-skinned varieties.

The melatonin levels in different parts of grapes vary as the grape's growth and maturity progressed. A research by Vitalini et al. (36) demonstrated two different patterns: the highest concentration of melatonin in the skin was observed during the pre-veraison period, this then decreased by 47% during the season of veraison. While the transition from the pre-veraison phase to the veraison phase increased the melatonin content of seeds by 68%, the content of fruit flesh increased by 95%. Additionally, the amount of melatonin in the seeds was significantly greater than that of the skin and grape flesh during the veraison.

Melatonin, a powerful antioxidant itself, has been found in significant amounts in grape skins (16). The role of melatonin in the biosynthesis of anthocyanins in grape fruits is multi-faceted, as it not only enhances the aesthetic appeal of grapes but also boosts their nutritional profile, making them an excellent addition to a healthy diet (36). In grapes, phenolic compounds also play a critical role in the fruit's quality and health-promoting properties, largely due to their



antioxidant activity, so the recent studies have indicated an interconnected role between melatonin and phenolic compounds in grapes, contributing significantly to the fruit's antioxidant activity (40). Preliminary studies suggest that melatonin could enhance the secondary metabolites, in grape including phenolics, flavanols and volatiles (25).

The growth of grape clusters is complex and involves three distinct phases. During the first phase, organic acids and some other compounds are stored in the vacuoles. The second phase (veraison) is marked by sugar accumulation, and rapid coloration due to anthocyanin accumulation in red grape varieties (21). In the third phase (ripening), the berries tend to have high glucose and

Materials and Methods

The experiment was carried out in a private orchard in Babylon governorate during the growing seasons 2022 and 2003 to study the effect of foliar spraying of melatonin on some secondary metabolites in grapes produced by the 10-year-old Halawani variety grown in parallel lines with dimensions (4 × 2) m. Twenty-seven grapevine were selected for the study, winter pruned (mid-January) for the two years of the study. Each vine had 84 eyes distributed

fructose and low content of organic acids, They are softer with better distinguished tastes (44). The color of the grape skin is one of essential quality requirements, as full color increases the market value of table grapes. The color of grapes is also an indicator of the quality of the fruits and the cluster's content of anthocyanins and phenolic compounds. Accordingly, this research was conducted to investigate the possibility of improving the quality and color of table grapes "Halwani" variety by foliar spraying with melatonin and to study the effect of the latter in stimulating the production of some secondary metabolic compounds of medical importance in the fruits.

on 7 canes, 12 eyes each (4). Organic fertilizer was added at a rate of 7 kg vine⁻¹ in mid-December and NPK (18: 18: 18) was applied by 500 g vine⁻¹ at the beginning of March (14). The experiment was laid down in a randomized complete design (RCBD), for two years where the grapevines were sprayed three times with melatonin hormone at 0, 3 and 6 mg L⁻¹ (3). The results were analyzed according to the analysis of variance and the averages were compared



using the Duncan's multiple range test ($p \leq 0.05$).

The vines were sprayed three times with 30 days intervals. The 1st spray was at the stage of flower loosening on April 15, 2022 and April 20, 2023 (7). The second spray was at the end of berry set, while the third application was at the stage of fruit ripening. At the end of the season, the grapes contents of biochemicals compounds were assessed including:

Total soluble solids (TSS): Estimated by the hand refractometer (2).

Total acidity (TA): It was estimated according to Ranganna (29).

Total sugars: The total sugars of grape juices were estimated according to the Lane and Eynon method described in A.O.A. C (1).

Total sugars / Total acidity (TS / TA)

Determination of melatonin in fruits flesh and seeds: Melatonin was estimated in grapes flesh and seeds. A 5 g sample of fruit flesh and seeds were separately mixed with a mixture of 50 mL of acetonitrile and water (1:1, V/V). The mixture was placed for 15 minutes in an ultrasonic bath, then 15 mL of the sample solution was transferred to a 100 mL volumetric flask and dissolved with a mixture of equal volumes of acetonitrile and

water. Melatonin was determined using high-performance liquid chromatography using C-18 separation column with acetonitrile/deionized distilled water/phosphoric acid (70:25:5 volume) at a flow rate of 1 mL/min, at 35 °C shaft temperature with injection volume of 100 μ L (34).

Anthocyanin pigment in fruit and seed content of anthocyanin was measured according to the method described in Ranganna (29).

Total flavonoid in fruit and seed: The total amount of flavonoids in the raw extract was determined according to Baba and Malik (5) based on of aluminum chloride titration in the presence of rutin (mg g^{-1} dry weight). The absorption of the model was recorded at a wavelength of 400 nm.

Total phenols: Total phenols were estimated according to the method proposed by Laouini and Ouahrani (22) using a gallic acid-based reagent and a Folin-Ciocalteu reagent. The total extraction was 100 μ L of alcoholic extract with 500 μ L of Voln reagent added to 1.5 mL of (20% sodium carbonate) making a final volume of 10 mL. After two hours of interaction, the absorption value was recorded at a wavelength of 765 nm. The total amount of



phenols was expressed as a percentage of the calibration graph of gallic acid (mg g^{-1} dry weight).

Resveratrol: It was estimated according Capruci et al. (8).

Extraction and purification method: A 100 g unpeeled fruits was used to estimate resveratrol by Adding it to 50 mL of ethanol 96% left in dark for 48 hours, then centrifuged at 4000 rpm for 20 minutes followed by filtration through 0.45 μm

filters. The samples obtained were kept in the dark until the extract underwent chromatographic analysis.

Determination: HPLC chromatographic separation was performed using a defined C18 column at room temperature 20°C. The reading was carried out using ultraviolet spectrometry at a wavelength of 280 nm for the cis-resveratrol shape and a wavelength of 307 nm for the trans-resveratrol shape.

Results and Discussion

Spraying with melatonin showed a significant effect on most of grapes quality characteristics, as melatonin at 6 mg L^{-1} resulted in the highest fruit content of TSS which increased from 11.90 for the 1st season to 15.72% in the second one (Table 1) Untreated control vines contained significantly the highest percentage of total

acidity (0.57 and 0.56%) in grapes juice compared to the other treatments. The melatonin at 6 mg L^{-1} increased fruit content of total sugars and total sugar /total acidity ratio with significant difference from the lower concentration and the control, respectively (Table1).

Table 1. Effect of spraying melatonin at different levels on grapes content of TSS, total sugars, total acidity and total sugars/total acidity ratio

Melatonin (mg L^{-1})	TSS (%)		Total sugars (%)		Total acidity (%)		Total sugars / Total acidity	
	2022	2023	2022	2023	2022	2023	2022	2023
0	17.14 c	16.15c	14.58 c	14.59 c	0.57 a	0.56 a	25.30 c	25.79 c
3	18.15 b	17.85 b	14.94b	15.04b	0.53 b	0.52 b	28.14 b	28.88 b



6	19.18 a	18.69 a	15.93 a	15.99 a	0.50 c	0.49 c	31.69 a	32.51 a
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Values are means of 3 replications, means within a column followed by different letter are significantly different according to Duncan's multiple range test ($P \leq 0.05$)

The results also showed that fruit content of anthocyanin increased significantly up to 16.61 and 16.99% in grapes from vines sprayed with melatonin at 6 mg L⁻¹ compared to lower rate in those treated with 3 mg L⁻¹ and the non-sprayed control (Table 2). A significant increase in melatonin content in fruit and seeds were also recorded in grapes from plants sprayed with melatonin at 6 mg L⁻¹ for the two growing seasons followed by the treatments of 3 mg L⁻¹ and the control respectively.

Findings (Table 3) also indicated significant effect of melatonin on the fruit and

seed contents of total phenols. The highest rate was in the treatment of 6 mg L⁻¹ recording an increase of 6.37 and 6.31% in fruit and 4.78 and 2.60% in the seeds for the two years respectively, with significant difference from the two other treatments. Similarly, flavonoids were also increased in fruit flesh (16.64 and 16.15%) and seeds (7.16 and 6.63%) significantly by spraying melatonin at 6 mg L⁻¹ compared to lower rates recorded in the control and when using melatonin at 3mg L⁻¹.

Table 2. Effect of spraying melatonin at different levels on 'Halawani' grapes (fruit and seeds) content of anthocyanin and melatonin for two successive seasons

Melatonin (mg L ⁻¹)	Anthocyanin in fruit (mg 100g ⁻¹ FW)		Melatonin in flesh fruit (mg Kg ⁻¹ FW)		Melatonin in seeds (mg Kg ⁻¹ FW)	
	2022	2023	2022	2023	2022	2023
0	222.1 c	222.4 c	35.46 c	35.72 c	8.39 c	8.37 c
3	230.7 b	229.9 b	37.76b	37.66b	9.07 b	9.02 b
6	259.0 a	260.2 a	46.52 a	46.40 a	10.67 a	10.66 a

Values are means of 3 replications, means within a column followed by different letter are significantly different according to Duncan's multiple range test ($P \leq 0.05$)

Table 3. Effect of spraying melatonin at different levels on ‘Halawani’ grapes (fruit and seeds) content of total phenols and flavonoids for two successive seasons

Melatonin (mg L ⁻¹)	Total phenols (mg gallic 100g ⁻¹ FW)				Flavonoids (mg rutin 100g ⁻¹ FW)			
	Fruit flesh		Seeds		Fruit flesh		Seeds	
	2022	2023	2022	2023	2022	2023	2022	2023
0	544.6 c	544.9 c	128.1 c	128.1 c	139.4 c	139.3 c	85.87 c	86.22 c
3	555.7 b	556.3b	129.7 b	129.6 b	144.5 b	144.6 b	87.30 b	87.33 b
6	579.3 a	579.3a	134.1 a	134.0 a	162.6 a	161.8 a	92.02 a	91.94 a

Values are means of 3 replications, means within a column followed by different letter are significantly different according to Duncan’s multiple range test ($P \leq 0.05$)

As shown in Table 4, the fruits flesh and seeds of the vines sprayed with melatonin at 6 mg L⁻¹ outperformed and achieved the highest content rate of resveratrol with an increase of 10.07 and 9.99% in fruit and

60.44 and 62.61% in seeds for the two years respectively, while the control treatment recorded the lowest rates with significant differences from those treatment with 6 and 3 mg L⁻¹ melatonin respectively

Table 4. Effect of spraying melatonin at different levels on ‘Halawani’ grapes (fruit and seeds) content of resveratrol for two successive seasons

Melatonin (mg L ⁻¹)	Resveratrol in fruit flesh (mg Kg ⁻¹ FW)		Resveratrol in seeds (mg Kg ⁻¹ FW)	
	2022	2023	2022	2023
0	10.92 c	10.91 c	3.16 c	3.13 c
3	11.13 b	11.06 b	3.82 b	3.88 b
6	12.02 a	12.11 a	5.07 a	5.09 a

Values are means of 3 replications, means within a column followed by different letter are significantly different according to Duncan’s multiple range test ($P \leq 0.05$)

The grapes maturity stage begins with a stage known as veraison, which is a critical

stage in the development of berry in which chemical changes determine the quality of



the berries. These changes included sugars accumulation and color change and decrease of acidity and other changes (28).

The interact between sugars and acids gives flavor and taste to grapes, as the accumulation of sugars associated with the decrease of organic acids in the berry is important in the physiology of grape fruits ripening (30).

It was observed that sugars increase in grape berry with a decrease of organic acids. This is in line with former findings in apple (35), peach (Zhou *et al.*,43),sweet cherry (11) and plum (12). It seems that the exogenous addition of melatonin has increased grapes contents of sugars, total soluble solids and decreased total acidity. This may be due to the increase in sugars produced by photosynthesis, as melatonin can enhance the effectiveness of photosynthesis by increasing the amount of chlorophyll in the leaves (13). Additionally, it may be linked with the regulation of genes involved in sugar metabolism, within melatonin application which affecting the sweetness of the fruit. During pear fruit ripening, it was found that melatonin increased the content of soluble sugars, especially sucrose and sorbitol, which was associated with down-regulation of sucrose invertase gene expression and up-regulation of sucrose phosphate synthase expression (24).

The concentrations of melatonin in different parts of grapes increase as the grape's growth and maturity continue. A study by Vitalini *et al.* (36) showed two different patterns: the highest concentration of melatonin in the skin was during the pre-veraison period, this then decreased by 47% during the transition

from pre-veraison to veraison, while the concentration of melatonin in the seeds was significantly higher than that in the skin and flesh. In this study, the amount of melatonin in the meat of fruit increased, this suggests that melatonin has a significant impact on the health and quality of grape fruit.

Many secondary compounds have been identified in the fruits and seeds of grapes, such as anthocyanins, phenolic compounds and their derivatives, as all grape fruits parts (fruit skin - fruit pulp and seeds) contain a large amount of phenolic compounds and their derivatives (42). Flavonoids are the largest part of these compounds in red and black varieties. These include anthocyanins, flavanols, and non-flavonoid phenolic compounds such as phenolic acids, stilbenes and resveratrol (27and 19). Melatonin also showed a positive effect on total phenol, total flavonoids and resveratrol in both of flesh and seeds. These secondary metabolites are believed to contribute to the fruit's color, flavor, aroma, and health benefits (26 and 25). Studies have shown that melatonin can enhance the production of certain secondary metabolites, significantly increase the content of phenolic compounds, flavonoids, and anthocyanins in some plants, like blackberry (10), Sweet cherry (39) and Red pear (32). The application of melatonin has been found to increase the anthocyanin content in grape skins, thereby enhancing their color and antioxidant properties (40). On the other hand, melatonin has been observed to stimulate the production of resveratrol. This phenolic compound is found in grape skins and known for its health benefits, including anti-inflammatory



and anti-cancer properties (40). This increase in resveratrol content not only enhances the nutritional value of grapes but also potentially increases their market value (15). In the context of grape fruits, melatonin contributes to phenol production in several ways, as melatonin appears to play a crucial role in maximizing the antioxidant potential

Conclusion

In conclusion, the use of melatonin in grape cultivation offers promising prospects for enhancing the quantity and quality of secondary metabolites, thereby improving the overall value of the fruit. In summary, melatonin appears to play a critical role in the antioxidant system of grapes, possibly enhancing the activity of phenolic compounds. This interaction could have significant implications for the quality of grapes and grape-derived products.

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of fruits. This may be due to melatonin direct action on free radicals and its ability to stimulate antioxidant enzymes and gene responses., it enhances the fruit's ability to withstand oxidative stress, potentially leading to better quality and nutritional content (41and 6).

However, further research is needed to develop practical and efficient application methods of melatonin, which could revolutionize viticulture sectors, and more research is needed to fully understand the mechanisms behind this interaction and its full impact on grape and wine quality.

Conflict of interest

The authors declare no conflict of interest.



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