Journal of Food Science and Technology (Iran)

Scientific Research

ww.fsct.modares.ir

Effect of Sulphur dioxide replacement with Nano zinc oxide on physicochemical and sensory properties of raisin

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ARTICLE INFO

Article History:

Received:2024/10/30

Accepted:2024/12/11

Keywords:

Coating,

Sulfur Dioxide,

Raisin Nano Zinc Oxide.

DOI: 10.22034/FSCT.22.161.56.

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ABSTRACT

The aim of this study was to investigate the effect of coating grapes by spraying with different concentrations of zinc oxide nanoparticles (0.5 and 1 g/L) both before and after harvest compared to postharvest fumigation of grapes with sulfur dioxide (2.5, 3.5, and 4.5 g sulfur per kg of grapes) on the physicochemical and sensory properties of raisins. The grapes were sun-dried and the resulting physicochemical properties of raisins, such as moisture content, pH, water activity (aw), acidity, free sulfur dioxide, total sulfur dioxide and sensory properties, were evaluated using a factorial design based on a completely randomized statistical model. The results showed that coating with zinc oxide at a concentration of 1 g/L before and after harvest and fumigation with sulfur dioxide after harvest affected the moisture and aw levels of the raisins, with moisture content increasing and aw decreasing compared to the control ($p<0.05$). No significant differnces was observed in pH of all the raisins coated with zinc oxide as compared to control (3.7) except for the sample coated with 0.5 g/L nano zinc oxide before harvest (3.45). By increasing the sulfur concentrations up to 3.5 and 4.5 g/kg grape, the pH of all the raisins compared to control (3.7), increased (3.8), and decreased (3.45), respectively ($p<0.05$). Acidity of the all the sulfur-treated samples increased ($p<0.05$), compared to the control (0.817 %). The concentration of free and total sulfur dioxide was significantly higher in the sulfur-treated samples compared to other treatments and the control ($p<0.05$). Raisins from grapes coated with 0.5 g/L zinc oxide prior to harvest and raisins treated with 2.5 g/kg sulfur dioxide received the highest overall sensory acceptability ratings from panelists ($p<0.05$).

[DOI: 10.22034/FSCT.22.161.56]

1-Introduction

grape (*Wine vine*) is one of the most important garden products in the world, which is often consumed due to its good taste and multipurpose feature [1,2]. Consumption of grape products such as raisins, juice and grape juice is always widely accepted in the world [1]. Raisin is the dried product of the ripe fruit of various varieties of grapes with and without seeds and is one of the most nutritious dried fruits in the world. The global production of raisins in 2022 is estimated at 1.4 million tons. About 76% of the world's raisins are produced in Turkey (395 tons), America (313 tons), China (170 tons), Iran (125 tons) and South Africa (71 tons). [3]. From a nutritional point of view, raisins are rich in vitamins (group B vitamins) and minerals needed by the body, including iron, potassium and calcium. It is also a good source of fiber and antioxidants [4]. Depending on the type of variety, method and drying conditions, raisins are often known as sunny, sour and sulphurous raisins. Raisins are sometimes named based on the type of consumption, such as pilaf raisins, green raisins, coarse raisins or raisins, which are often common in the market of Iran [5]. Fumigation with sulfur gas is a traditional and common method to combat microbial spoilage of fruits and especially grapes before drying with the aim of increasing shelf life, preserving color and reducing microbial contamination in storage. The necessity of frequent use of sulfur gas during storage, on the one hand, and the presence of residual sulfur in the product (maximum allowed 10 ppm), which, in addition to the negative impact on consumer health, seriously harms the marketability, color and quality of its aroma and flavor, on the other hand, has reduced the acceptance of this method. Adding sulfur dioxide to grapes before drying is used to preserve color, control browning, and have an antimicrobial effect. Consumption of raisins Sulfur is less popular due to the dangers that sulfur consumption creates for the consumer [6].. Edible coatings reduce the waste of this product by maintaining the quality and increasing the storage life of fresh fruits, especially grapes. Therefore, the use of edible coatings, especially coatings with antimicrobial properties, with the aim of controlling or reducing losses and increasing the life of grapes after harvesting, has attracted a lot of attention. These types of covers corruption Food products particle for direct object with formation one layer half Trava in around It and as a result decrease Permeability to oxygen, dioxide Carbon and They reduce humidity [7 and 6]. Zinc oxide is one of the metal oxides that is used in various industries, including the food industry [8]. with use from technology nano and with decrease Dimensions Particles in limit nanometer power activity superficial Material and ratio the reaction giving they with circumference around to guide increase Sections active superficial increase He finds. Zinc oxide nanoparticles to title one installation Ayman (GRAS) by office food and medicine America recognized and application it in industries food and medicine confirmation done is [6،8]. safety in use and Also feature Antimicrobial oxideRoy from one Sleep and increase power against microbe it with decrease Dimensions in limit nanometer and therefore use from ConcentrationI seeY a lot less, from Sue other Nanoparticles oxide Roy particle for direct object to title optionA appropriate direction cover give the fruits and to especially grape Raised does [6]. Until now, there have been extensive researches regarding the use of fruit coating technology, especially various grape varieties with different formulations, including nanoparticles. Xydroi on grape black [6]، essential oil Carvacrol on grape، Combinations lipid on Features raisins [9]، wax Carnauba and Monoglycerol Stearate on Features raisins [10] coverHi edible on base starch and gelatin on Features grape red [11]، Combinations Alginate with Vanillin on Features grape [12] and solution aloe Vera before from Collection of figures grape [13] done. Also use Acetic acid has been considered as a substitute for sulfur in the production of raisins [14]. This research will examine the production of healthy raisins free of unsafe additives for the health of the consumer. Keeping the green color by reducing enzymatic browning reactions in raisins like sulfur gas is one of the goals of coating raisins with zinc oxide nanoparticles. The main goal of this research is to evaluate the use of zinc oxide nanoparticles compared to fumigation with sulfur gas on the physicochemical, sensory and marketability characteristics of raisins produced in Iran.

2- Materials and methods

2-1- Materials

This research was carried out in one of the vineyards of Malair city on a number of white Bidane grape trees (*Vinifera L*) was done. Zinc oxide nanoparticles from Nano Pishgaman Company.Mashhad, Iran(and sulfur from the Chemical Industry Development Research Institute)IRDCI, Iran) was purchased. All chemicals and reagents from Charlo company (Shadow Company, Spain) was prepared.

2-2- Methods

Aqueous suspension of zinc oxide nanoparticles in concentrations of 0.5 and 1 g per liter was prepared in sterile distilled water and prepared for 45 minutes at 25 degrees Celsius and 25 kHz frequency in order to uniformly disperse nanoparticles in the solution in an ultrasonic bath.TI-H-5, Elma GmbH, Germany) was placed. Spraying nanozinc oxide solution on grapes was done during three growth stages including flowering stage, immature stage and physiological ripening stage of grapes (20 days before harvest) [15]. In another method, grape bunches of the same size and weight were immersed in a suspension containing zinc oxide nanoparticles at concentrations of 0.5 and 1 g per liter in sterile distilled water for 5 minutes and then rinsed. Gassing with sulfur dioxide in concentrations of 2.5, 3.5, 4.5 grams per kilogram of fresh grapes was done by placing the freshly harvested bunches in the fumigation chamber with sulfur gas at ambient temperature, which carried out the gas transfer operation within 30 minutes using the propeller at the bottom of the chamber. Freshly harvested grape samples without coating and without gasification were used as control samples. All the grape samples were dried in the sun and after equalizing the humidity and separating the tail, stem and waste, they were packed in polyethylene bags [14, 6]. The different coded treatments are shown in Table 1.

Table 1. The description and code of different samples.

Treatment Number	Description	Code of raisin samples
1	Untreated raisin	R
2	Raisins by spraying ZnO NPs (0.5 L/g) on the bunch on the tree	R1
3	Raisins by spraying ZnO NPs (1 L/g) on the bunch on the tree	R2
4	Raisins by spraying ZnO NPs (0.5 L/g) after harvesting	R3
5	Raisins by spraying ZnO NPs (0.5 L/g) after harvesting	R4
6	Raisins carbonated with SO_2 (2.5g/kg)	R5
7	Raisins carbonated with SO_2 (3.5g/kg)	R6
8	Raisins carbonated with SO_2 (4.5g/kg)	R7

3-2- Tests

1-3-2- Investigating the distribution and dispersion of produced zinc oxide nanoparticles

The size and distribution method of zinc oxide nanoparticle suspension prepared for distribution on the surface of raisin samples using a model scanning electron microscope MIRA3Company product TESCAN The Czech Republic was measured at a voltage of 30 kV [6].

2-3-2- percentage of humidity

About 5 grams of the samples were placed in a porcelain crucible in an oven under vacuum at a temperature of 70 degrees Celsius and a pressure of 150 millibars for 8 hours. After complete drying, the samples were weighed again and the difference in the weight of the

[DOI: 10.22034/FSCT.22.161.56]

samples compared to the initial weight was expressed as percentage of moisture [16].

3-3-2- Water activity (a_{In})

The water activity of the ground raisin sample, which indicates the ratio of the water vapor pressure of the food to the saturated vapor pressure of pure water at the same temperature, using a water activity measuring device (Novasina, Switzerland) was measured after calibration at a temperature of 22 degrees Celsius [17 and 18].

4-3-2- Measurement (pH)

value (pH) samples using the device pH digital meter (Metrohm, Switzerland) was measured. 10 grams of the ground sample was brought to a volume of 100 ml with 90 ml of distilled water and mixed completely, and after 5 minutes, the electrode of the device was placed in it and after the display number was fixed, pH It was read [19].

5-3-2- Measurement of acidity

Acidity of the samples based on the potentiometric method with the help of (pH m) and during neutralization with 0.1 M soda solution in the presence of phenolphthalein reagent until reaching (pH) equivalent to 1/8 of the measurement and its value was calculated as a percentage based on grams of tartaric acid per 100 grams of sample [16].

6-3-2- Measurement of free and total sulfur oxide

Free sulfur dioxide, which is the amount of sulfur dioxide that is free and unbound on the surface of raisins, and total sulfur dioxide, which is the sum of free and total sulfur dioxide in raisins, were measured by the method of Mehraban Seng Atash et al. (2014) using neutralization using sodium thiosulfate [4].

4-2- Sensory test

Raisin samples after coding with the help of 10 men and women in the age range of 25 to 40 years old who had received the necessary training regarding the characteristics of color, aroma, smell, taste, texture and overall acceptance using a pleasurable test (Hedonic Scale) 5 points were evaluated. In this evaluation, the number 5 was very good, the number 4 was good, the number 3 was average, the number 2 was weak, and the number 1 was very weak. [20].

5-2- Statistical analysis

The data obtained from the experiment based on the factorial method and in the form of a completely random statistical design with 3 repetitions and variance analysis of the resulting data with the help of software (SPSS, 26) and the comparison of means was done with Duncan's multi-range test at the 95% probability level.

3- Results and discussion

1-3- Scanning electron microscope image

The scanning electron microscope image of the suspension containing zinc oxide nanoparticles with a concentration of 1 gram per liter is shown in Figure 1. In this image, nanoparticles are observed in a uniform form with a spherical appearance and no pores. These results were consistent with the observations of Suresh et al. (2018) regarding the production of zinc oxide nanoparticles suspension free of clumps and interconnectedness in dimensions less than 100 nm. [21]. Also, Timosuk et al.'s report (2020) showed that the sample of zinc oxide nanoparticles is homogeneous in shape and size in the range of 20 to 60 nm [22]. Al-Badiri et al. (2018) also reported that zinc oxide nanoparticles are formed in a very uniform way and the particle size is less than 100 nm [23].

[DOI: 10.22034/FSCT.22.161.56]

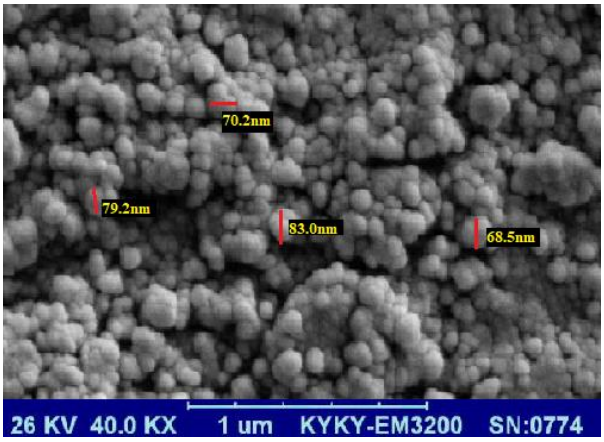

Fig. 1. SEM image of Nano zinc oxide suspension (1 g/L)

2-3- Humidity and water activity (a_{In}) Raisin samples

The amount of moisture in raisins is an important indicator in the ability to store this product. Reducing moisture in raisins has a significant effect on increasing its shelf life [24 and 25]. The moisture content of the studied raisin samples was in the range of 12.53 to 15.63 percent, which corresponded to the maximum moisture content of raisins (16 percent) according to the Iranian National Standard No. 17. [26]. Comparison of the average percentage of humidity and well Sample raisinsThe examined in figure (2) is shown. Compared to the control sample, the highest amount of moisture was measured in the treatments coated with zinc oxide nanoparticles and the lowest in the treatments gassed with sulfur dioxide. The results showed that the fumigation method with sulfur gas had the greatest effect in reducing the moisture content of raisin samples so that The highest percentage of moisture in the treatment samples R4 and the lowest in the treatment samples R7 It was measured (05/0p<). According to the results of Figure 2, spraying zinc oxide nanoparticles on the samples before or after harvesting did not have a significant effect on the moisture content of the produced raisin samples. On the other hand, increasing concentration Sulfur dioxide gas caused a significant decrease in the moisture content of production samples (05/0p<). Coverage in general It limits the moisture loss process of raisins. The higher percentage of moisture in treatments coated with zinc oxide nanoparticles can be Creating a thin layer of zinc oxide nanoparticles It is related that it prevents the rapid decrease of the moisture content of the product compared to the control sample [25]. Moisture reduction of raisin samples Gassed with sulfur dioxide can also be attributed to accelerating the removal of moisture from the product due to the creation of small cracks on the surface of the skin of aerated grapes. [24]. Ghasemzadeh et al. (2008) also stated that raisins coated with raisins with a coating composed of starch, pectin, and gum had more moisture compared to samples without coating [25]. amount (well) raisin samples depending on the type of treatment in the range It varied from 0.41 to 0.58 (Figure 2). Most foods with water activity less than 0.6 have good microbial stability [16]. usually the amount of water activity (well) raisins in samples with moisture 13 to 18 percent, in range It varies from 0.50 to 0.65, depending on the type of grape variety, up to 0.8 has also been reported in some types of raisins [24]. According to the results of Figure 2, coating grapes with zinc oxide nanoparticles and gassing with sulfur dioxide, respectively, a_{In} Raisins increased and decreased production. These results were consistent with the results of changes in the moisture content of the produced raisins. The most and the least a_{In} respectively in the raisins treatment R4 and R7 It was measured. The measurement of water activity as a key factor with the aim of predicting the stability and microbial and biochemical safety of raisins has been investigated and emphasized by Ake et al. (2018). [27].

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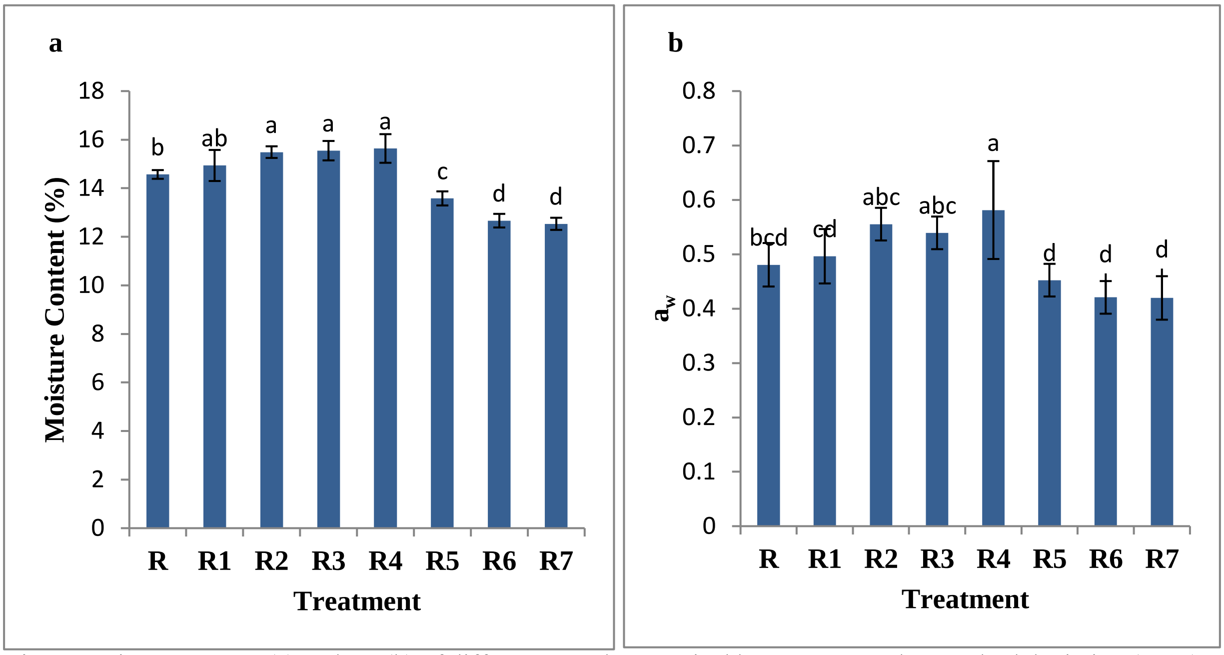

Fig. 2. Moisture content (a) and aw (b) of different samples. Vertical bars represent the standard deviation (n = 3). Different letters are significantly different (p <0.05).

2-3- The effect of different treatments on the amount pH and acidity of raisin samples

Among the factors affecting the rate pH And the acidity of raisins during storage is the amount and composition of sugars, organic acids, intensity of respiration, metabolic reactions and its moisture content [28 and 29]. pH Raisin samples It ranged from 3.45 to 3.8 (Figure 3). Ali Askari et al. (2012) also pH Local and imported Moroccan raisins in the range of 3/5 up to 4 announced [24]. The highest amount pH Belonging to the treatment R3 And the least pH belonging to the treatments R1 and R7 was The results showed that coating grapes with zinc oxide nanoparticles in high concentrations (1 gram per liter) before and after harvesting pH It reduced the production of raisins compared to the control sample (3.73). pH Raisins from grapes treated with Sulfur dioxide gas to concentration 3.5 g/kg of grapes compared to the control sample There was no significant difference, but with the increase of sulfur gas concentration up to 4.5 g/kg of grapes, there was a decreasing trend (0.05p<). The results of comparing the averages Sample acidityRaisins (Figure 3) showed that coating grapes before and after harvest using zinc oxide nanoparticles did not significantly change the acidity of produced raisins compared to the control sample (0.817%), while gassing the grape samples after harvest using different concentrations of sulfur dioxide significantly increased the acidity of produced raisins compared to the control (0.05%).p<). The effect of low and high concentrations of zinc oxide nanoparticles as well as the spraying stage on grapes had no significant effect on the acidity of the produced raisins. Using different concentrations of sulfur dioxide gas did not have a significant effect on the acidity of produced raisins. The highest acidity in raisin samples gassed with sulfur dioxide at a concentration of 4.5 grams per kilogram of grapes and the lowest acidity It was measured in raisin samples coated with zinc oxide nanoparticles at a concentration of 0.5 g/liter in the pre-harvest stage.

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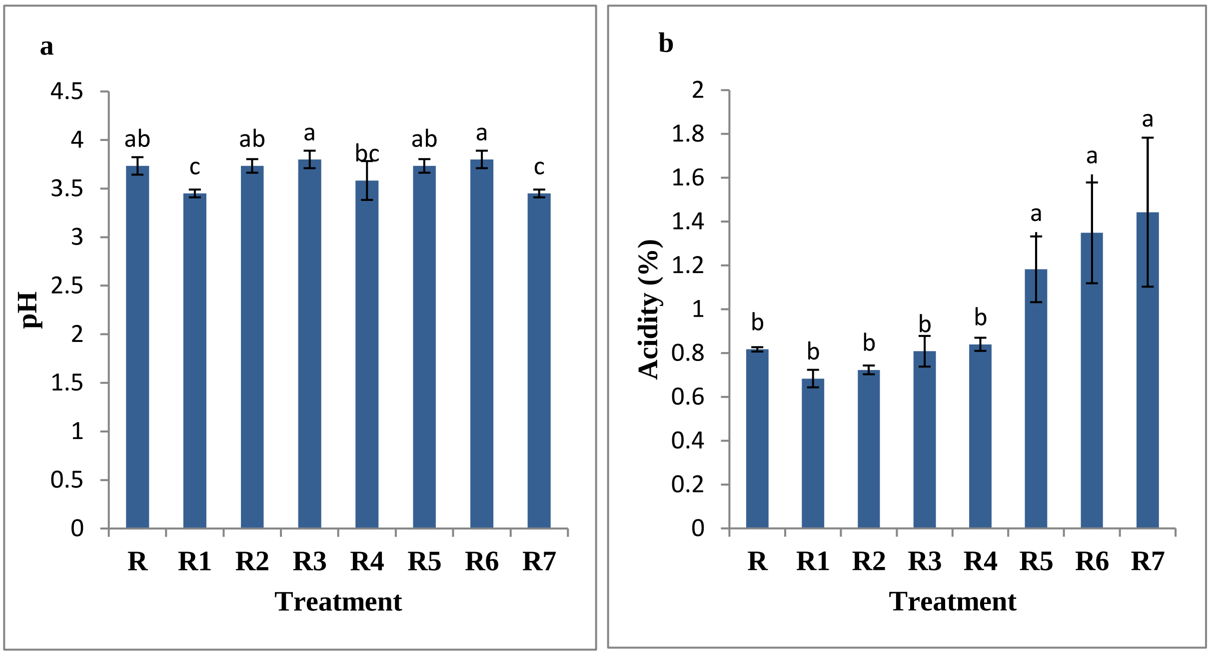

Fig. 3. pH (a) and acidity (b) of different samples. Vertical bars represent the standard deviation (n = 3). Different letters are significantly different (p <0.05)

3-3- Free sulfur dioxide and whole raisin samples

The amount of sulfur dioxide absorbed in raisin seeds is referred to as residual sulfur dioxide [24]. This amount includes the amount of free and total sulfur dioxide, in which free sulfur dioxide is equal to the amount of sulfur dioxide that is free and unbound on the surface of raisins. The amount of total sulfur dioxide refers to the sum of free and bound sulfur dioxide [4]. The comparison results of the average free and total sulfur dioxide belonging to the samples obtained from different treatments are presented in Table 2. Free and total sulfur dioxide was not detected in control raisin samples and samples coated with zinc nanooxide. The results of Table 2 showed the increasing trend of free and total sulfur dioxide in the samples of raisins treated with sulfur dioxide at the same time as the concentration of sulfur dioxide gas increased (0.05).p<). The amount of free sulfur dioxide for raisins gassed with 2.5, 3.5 and 4.5 grams per kilogram of sulfur dioxide was 67, 141 and 243 milligrams per kilogram of raisins, respectively, and the amount of total sulfur dioxide was 352, 643 and 918 milligrams per kilogram of raisins. According to the results of other studies, increasing the contact time of raisins or grapes with sulfur dioxide gas increases the amount of residual sulfur dioxide gas in the product, which was consistent with the results of this study [30]. Also, the amount of sulfur absorption in raisins produced during the drying stage of smoked grapes with sulfur gas has a direct dependence on the gassing time and the concentration of sulfur used [31]. The results of this research are consistent with the results of Lidakis et al. (2003) on raisins [31] and Song et al. (2000) on green bananas [32].

Table 2. The free SO_2 and total SO_2 of different samples.

Treatment	R	R1	R2	R3	R4	R5	R6	R7
Free SO_2	ND	ND	ND	ND	ND	$67±9^{c}$	$141±8^{b}$	$243±40^{a}$
Total SO_2	ND	ND	ND	ND	ND	$352±111^{c}$	$643±71^{b}$	$918±67^{a}$

Different letters are significantly different ($p < 0.05$)

4-3- Sensory evaluation of raisin samples

The effect of coating grapes with zinc oxide nanoparticles and fumigation with di gas Sulfur oxide significantly changed the sensory characteristics of the produced raisins (0.05p<). The average sensory scores of color, smell, taste, appearance and overall acceptance of raisins produced by adding zinc oxide nanoparticles and sulfur dioxide are shown in Figure 4. Raisins produced by fumigation with sulfur dioxide gas compared to the samples coated with nano-zinc oxide and the control sample received more color scores from the evaluators. The highest sensory color score was detected in the raisin samples gassed with 2.5 grams of sulfur dioxide per kilogram of grapes and the lowest color score was detected in the raisin samples coated with zinc nanooxide (1 gram per liter) in the pre-harvest stage. Raisins produced by fumigation with sulfur dioxide gas had a lower odor score compared to the samples coated with zinc nanooxide and the control sample. The lowest odor score was detected in the raisin samples gassed with 4.5 grams of sulfur dioxide per kilogram of grapes, and the highest odor score was detected in the raisin samples coated with zinc nanooxide (0.5 grams per liter) in the pre-harvest stage (0.05).p<). Regarding the sensory points of taste, texture and overall acceptance of raisins produced with both methods of fumigation with sulfur gas (2.5 grams of sulfur dioxide per kilogram of grapes) and coating with nanoparticles of zinc oxide (0.5 grams per liter) in lower concentrations compared to the control sample and other samples, they received the highest score from the evaluators (0.05).p<). These results show the adverse effect of using high concentrations of sulfur gas and zinc oxide nanoparticles in the production of raisins, which has an adverse effect on the sensory acceptance of the final product by the consumer. Iqbal et al. (2020) reported that increasing the duration of gassing grapes with sulfur gas during drying reduces the sensory characteristics of the produced raisins in terms of texture, color, aroma and taste [30].

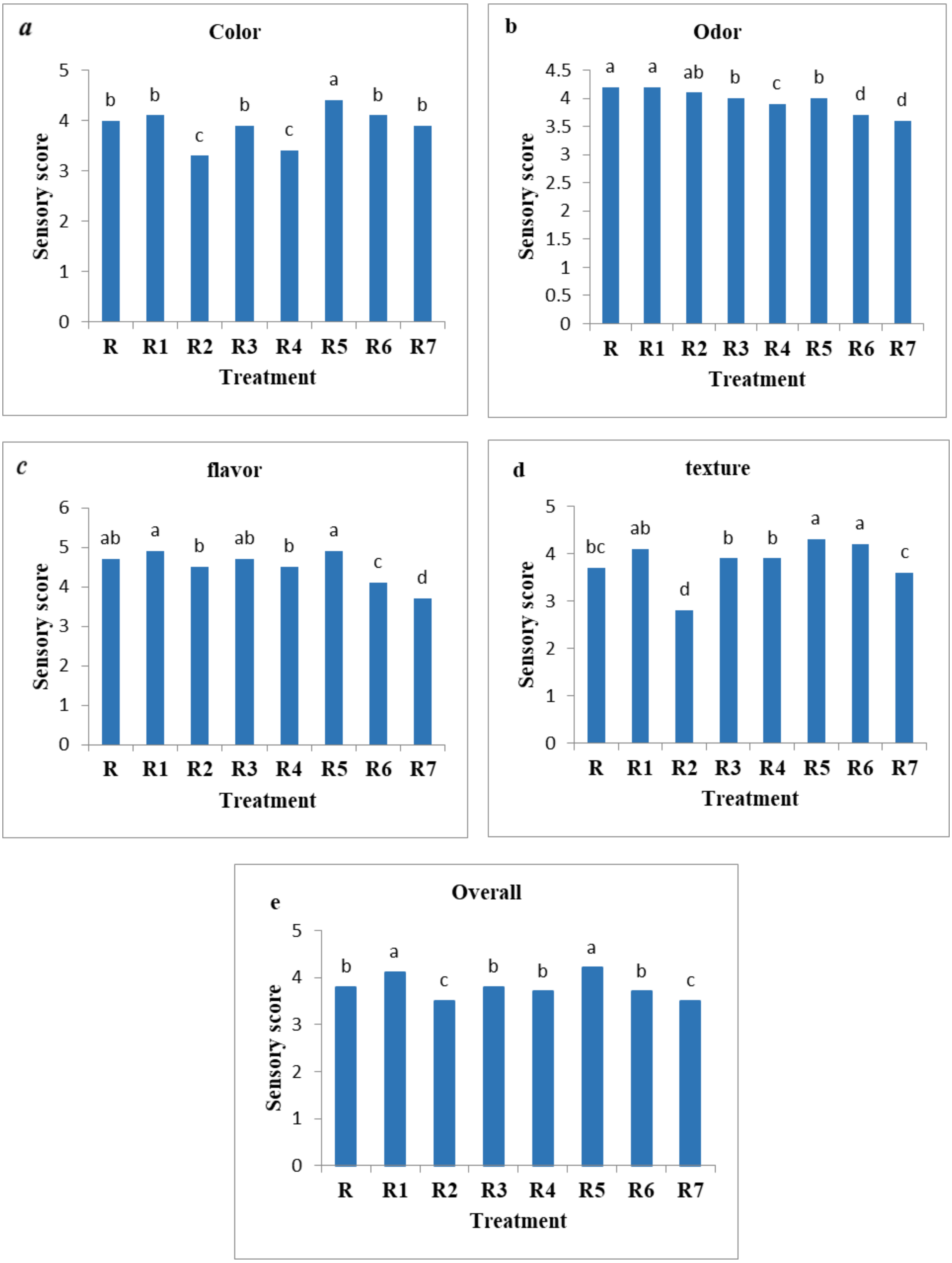

Fig. 4. Sensory score of raisin samples: Color (a), Odor (b), flavor (c), texture (d), Overall (e)

4- General conclusion

The results of this research showed that The amount of humidity and a_{In} Produced raisins By increasing the concentration of zinc oxide

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nanoparticle coating on the raisin product before or after harvesting fresh grapes, and with increasing concentration Sulfur dioxide during the drying stage of fresh grapes decreases amount pH Raisin production also decreased with increasing concentration of zinc oxide nanoparticles sprinkled on the tree, increasing and increasing concentration of zinc oxide nanoparticles sprinkled on grape fruit after harvest. Also the amount pH Raisins with increasing concentration Sulfur dioxide It increased to the level of 3.5 (grams per kilogram of fresh grapes). Acidity of production raisins By increasing the concentration of zinc oxide nanoparticles sprayed on the tree and after harvesting and concentration Sulfur dioxide increased Increasing the concentration of sulfur gas had a direct effect on the amount of sulfur in the final product. Raisins produced with both fumigation methods with sulfur gas (2.5 grams per kilogram of grapes) and coating with zinc oxide nanoparticles (0.5 grams per liter) in lower concentrations compared to the control sample and other samples, received the highest score from the evaluators in terms of overall sensory quality (0.05).p<). In general, the use of zinc oxide nanoparticles with a concentration of 0.5 grams per liter to coat raisins is introduced as a safe additive. The results of this research emphasized the necessity of using new technologies such as nano coatings such as nano zinc oxide in the processing of raisins and the production of healthy products free of unsafe additives, especially sulfur. This importance not only plays a decisive role in the health of the consumer, but by reducing the waste of this product and increasing its added value, it will also improve the export of this valuable product.

5-Resources

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[DOI: 10.22034/FSCT.22.161.56]

مجله علوم و صنایع غذایی ایران

سایت مجله: **www.fsct.modares.ac.ir**

مقاله علمی_پژوهشی

اثر جایگزینی دی‌اکسید گوگرد با نانو اکسیدروی بر ویژگی‌های فیزیکوشیمیایی و حسی کشمش تولیدی

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اطلاعات مقاله

تاریخ های مقاله :

تاریخ دریافت: ۱۴۰۳/۸/۹

تاریخ پذیرش: ۱۴۰۳/۹/۲۱

کلمات کلیدی:

پوشش دهی،

دی‌اکسید گوگرد،

کشمش،

نانو اکسیدروی.

DOI: 10.22034/FSCT.22.161.56.

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چکیده

هدف از این مطالعه بررسی تأثیر پوشش‌دهی انگور با افشاندن غلظت‌های متفاوت نانوذرات اکسیدروی (۰/۵ و ۱ گرم در لیتر) در دو مرحله قبل و پس از برداشت در مقایسه با روش گازدهی انگور تازه برداشت شده با دی‌اکسید گوگرد (۲/۵، ۳/۵ و ۴/۵ گرم گوگرد در هر کیلوگرم انگور) بر ویژگی‌های فیزیکوشیمیایی و حسی کشمش تولیدی بود. انگورها به روش آفتابی خشک شدند و ویژگی‌های فیزیکوشیمیایی درصد رطوبت، مقدار pH، مقدار aw، درصد اسیدیته، دی‌اکسید گوگرد آزاد، دی‌اکسید گوگرد کل و ویژگی‌های حسی کشمش تولیدی پس از یکنواخت شدن رطوبت با روش فاکتوریل و بر اساس طرح آماری کاملا تصادفی ارزیابی گردید. پوشش دهی انگور قبل و پس از برداشت با نانوذرات اکسیدروی در غلظت ۱ گرم در لیتر و گازدهی با دی اکسید گوگرد پس از برداشت، به ترتیب میزان رطوبت و aw کشمش تولیدی را در مقایسه با نمونه شاهد افزایش و کاهش داد ($p<0.05$). pH نمونه‌های پوشش داده شده با نانوذرات اکسیدروی قبل از برداشت به جز در غلظت ۰/۵ گرم در لیتر (۳/۴۵)، در مقایسه با شاهد (۳/۷) تفاوت معنی‌داری نداشت. با افزایش غلظت دی‌اکسید گوگرد تا مقدار ۳/۵ و ۴/۵ گرم گوگرد در هر کیلوگرم انگور، pH نمونه‌های کشمش در مقایسه با شاهد (۳/۷) به ترتیب افزایش (۳/۸) و کاهش (۳/۴۵) یافت ($p<0.05$). اسیدیته نمونه‌های گازدهی شده با گاز گوگرد در مقایسه با نمونه شاهد (۰/۸۱۷ درصد) افزایش معنی‌داری داشت ($p<0.05$). غلظت دی‌اکسید گوگرد آزاد و دی‌اکسید گوگرد کل در نمونه‌های گازدهی شده در مقایسه با سایر نمونه‌ها و نمونه شاهد به شکل معنی داری بیشتر بود($p<0.05$). کشمش حاصل از انگورهای پوشش داده شده با نانواکسیدروی در غلظت ۰/۵ گرم در لیتر در مرحله قبل از برداشت و کشمش حاصل از انگورهای گازدهی شده با گوگرد با غلظت ۲/۵ گرم در هر کیلوگرم انگور بیشترین امتیاز حسی پذیرش کلی را از ارزیاب‌ها دریافت نمودند($p<0.05$).