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Efficacy of Green-Synthesized Calcium Nanoparticles from *Sideritis pisidica* on Postharvest Quality of 'Ağın Beyazı' Grape Berries

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ABSTRACT

The research aimed first the synthesize and characterize calcium-based nanoparticles (SPNPs) obtained from *Sideritis pisidica* leaf extract by green synthesis method, and then the post-harvest use of these nanoparticles for the preservation of 'Ağın Beyazı' grape berries. *S. pisidica* is a medicinal and aromatic plant that grows especially in southwestern Türkiye and possesses antioxidant and antimicrobial phytochemicals, qualifying it for green nanoparticle synthesis. The synthesized SPNPs were characterized by UV-visible and FTIR spectroscopy. The grapes were treated with SPNPs (25, 50, 100 mg L^{-1}) and stored at 4 °C for 20 days. Physicochemical parameters, including firmness, respiration, total soluble solids, acidity, pH, and color, were measured during storage. SPNPs did not significantly affect firmness, pH, acidity, or total soluble solids content but did affect respiration and color retention. The 100 mg L^{-1} SPNP treatment led to higher respiration rates in the later storage phase, possibly due to increased metabolism from the thick or dense SNPS coating. Meanwhile, the treatment also led to a suppression of further yellow color development (b*) at a later stage of storage, thereby indicating protection against pigment degradation or accumulation. Overall, SPNPs had a limited impact on the structural and chemical composition of grape berries, but their significant effect on color retention suggested their potential as an environmentally friendly postharvest application.

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Sideritis pisidica Kullanılarak Yeşil Sentezlenmiş Kalsiyum Nanopartiküllerinin 'Ağın Beyazı' Üzümlerinin Hasat Sonrası Kalitesi Üzerine Etkinliği

ÖZET

Araştırma, önce *Sideritis pisidica* yaprağı ekstresinden yeşil sentez yöntemiyle elde edilecek kalsiyum bazlı nanoparçacıkların (SPNP'ler) sentezini ve karakterizasyonunu, daha sonra bu nanoparçacıkların 'Ağın Beyazı' üzüm meylerinin muhafazası için hasat sonrası kullanımını amaçlamıştır. *S. pisidica*, özellikle Türkiye'nin güneybatısında yetişen, antioksidan ve antimikrobiyal fitokimyasallara sahip, yeşil nanopartikül sentezi için uygun tıbbi ve aromatik bir bitki özelliği taşımaktadır. Sentezlenen SPNP'ler, UV-görünür ve FTIR spektroskopisi kullanılarak karakterize edilmiştir. Üzüm taneleri 25, 50 ve 100 mg L^{-1} konsantrasyonlarındaki SPNP çözeltileri ile muamele edilerek 4 °C'de 20 gün süre ile muhafaza edilmiştir. Muhafaza esnasında meyve sertliği, solunum hızı, toplam çözünür kuru madde, titre edilebilir asitlik, pH ve renk gibi fizikokimyasal özellikleri izlenmiştir. SPNP uygulamaları sertlik, pH, asitlik ve toplam çözünür kuru madde üzerinde belirgin bir etki göstermemiştir. Ancak 100 mg L^{-1} nanopartikül uygulaması, muhtemelen kalın veya yoğun SNPS kaplamasından kaynaklanan artan metabolizma olayları nedeniyle, muhafazanın sonlarına doğu solunum oranlarına bir yükselişe neden olmuştur. Bu arada, aynı uygulama, muhafazanın sonlarında doğru daha fazla sarı renk gelişiminin (b*)

Bahçe Bitkileri

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Meyve renk ölçümü

baskılanmasına yol açarak renk maddelerinin bozulmasına veya birikimine karşı koruma sağlayabilmiştir. Genel olarak, SPNP'ler üzüm tanelerinin yapısal ve kimyasal bileşimi üzerinde sınırlı etkide bulunmuş, ancak renk koruma üzerindeki belirgin etkileri bunların bir çevre dostu hasat sonrası uygulaması olarak değerlendirilebilme potansiyelini ortaya koymuştur.

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INTRODUCTION

Application of nanoparticles to store and maintain fruit is a revolution in postharvest technology, offering fresh strategies to increase shelf life, reduce spoilage, and maintain the quality of fruit. Due to their high surface area and bioactive properties, nanoparticles synthesized from silver (Ag), zinc oxide (ZnO), and titanium dioxide (TiO_2) possess strong antimicrobial activity that inhibits the growth of spoilage and pathogenic microorganisms on the fruit surface. These particles can damage microbial membranes, generate reactive oxygen species, or impair microbial metabolism, thereby preventing postharvest spoilage (Othman et al., 2014; Baran et al., 2022; Karakaş et al., 2024). In addition to their antimicrobial action, nanoparticles have also been incorporated into edible coatings and biodegradable packaging materials to develop nanocomposite systems that can act as physical and biochemical barriers. These findings reduce respiration rates, water evaporation, and microbial deterioration, enhancing the shelf life of tender fruits such as strawberries, bananas, and mangoes (Rai et al., 2012). Chitosan-based films containing silver nanoparticles, for example, have been shown to greatly extend the shelf life of fresh fruits by inhibiting the growth of common spoilage microorganisms (Emamifar et al., 2010)." Additionally, the use of ZnO nanoparticles in food packaging has demonstrated antimicrobial activity, UV shielding, and oxygen scavenging abilities, which help preserve fruit quality (Ashfaq et al., 2022). Despite their potential applications, the use of nanoparticles in the food industry raises safety concerns, regulatory needs, and environmental exposure issues. This has led to increased interest in green synthesis techniques using plant extracts or biopolymers, offering more environmentally friendly alternatives with reduced toxicity risks. With advancing research and technology, nanoparticle-based postharvest preservation technology is poised to be a leading contributor to sustainable postharvest management that aligns with modern food safety and environmental requirements.

Application of nanoparticles or food coatings to grape berries has been the most viable method in recent years to attain postharvest improvement in quality, shelf-life extension, and prevention of spoilage. ZnO, Cu, or chitosan nanoparticles have antimicrobial and antioxidant activities against fungal infection and oxidative stress on grapes (López et al., 2017; Hashim et al., 2019; Abou El-Nasr et al., 2021). In the same vein, nanomaterial-reinforced or natural compound-reinforced food coatings also have the ability to form semi-permeable films that make it easy to regulate respiration, evaporation of moisture, and microbial growth (Dhall, 2013). Nano-coatings have also been found to not only preserve the physicochemical characteristics of grapes, including firmness, color, and sugar content, but also decrease the use of synthetic preservatives, which is compatible with sustainable agriculture practices (Sonker et al., 2016). Despite these benefits, further studies are needed to understand the long-term safety, consumer acceptability, and environmental friendliness of nanoparticle use in viticulture.

Calcium nanoparticles, synthesized from precursors such as calcium chloride ($CaCl_2$) or calcium carbonate ($CaCO_3$), are reported to possess vast potential in postharvest technology by extending the shelf life and quality of fresh produce such as fruits and vegetables. The nanoparticles may be employed in edible coatings or packaging materials to prevent microbial spoilage, restrain moisture loss, and decelerate senescence in fruits and vegetables (Karnwal et al., 2025). For instance, nano-coatings containing calcium nanoparticles reinforce the firmness of the cell wall by cross-linking with pectin to guarantee freshness in fruits like strawberries and apples (Li et al., 2023; da Silva Bruni et al., 2024). Calcium oxide (CaO) nanoparticles also exhibit intense antifungal activity, representing a chemical fungicide-free solution for postharvest disease control (Li et al., 2023). While such benefits are present, regulatory clearance, consumer acceptability, and large-scale production protocols need to be addressed in order to make it feasible for extensive use in the food sector.

Sideritis pisidica Boiss. & Heldr., a south-western Turkish local endemic species in the family *Lamiaceae*, mainly grows on calcareous mountains and rocky soil. It is used in traditional medicine and tea for its pharmaceutical properties, such as anti-inflammatory, antimicrobial, and antioxidant effects (Piozzi et al., 2006). *Sideritis* species contain bioactive compounds like flavonoids, phenolic acids, and diterpenoids that contribute to their pharmacological activities (Fraga et al., 2012). Phytochemical studies of *S. pisidica* revealed have identified its essential oil composition and phenolic content, which have potential applications in food preservation, pharmaceuticals, and cosmeceuticals (Deveci et al., 2019). Given the growing demand for plant antimicrobials and natural products, *S. pisidica* holds promise for further research and cultivation. However, its ecological specialization and limited geographical distribution highlight the need for sustainable harvesting and conservation.

This study aimed to assess the effectiveness of calcium-based nanoparticles synthesized from *Sideritis pisidica* Boiss. & Heldr. leaf extract in preserving the postharvest quality of cold-stored 'Akın Beyazı' grapes.

MATERIAL and METHOD

Nanoparticle synthesis

Sideritis pisidica Boiss. & Heldr. leaves and green shoots were ground into a powder. The powder was then mixed with methanol (1:10 ration) and vortexed. The mixture was shaken at a temperature of 115 °C for 7 h. Afterword, the mixture was stored at 4 °C for 16 h before filtration using filter paper. The synthesis of nanoparticles was initiated by adding 50 ml of methanol, 400 ml of CaCl2 (44.75%) and 20 ml of NaOH (8%) solutions to 1 g of the filtrate. The mixture was then stirred for 24 h. The nanoparticle extract from the mixture was obtained by evaporating the methanol in an evaporator.

Plant material and nano particle coating

The 'Ağın Beyazı' grape variety, which is indigenous to Elazığ province in Türkiye, was selected as the plant material for the present study. The fruit samples were collected randomly to ensure uniformity, and were then subjected to a rigorous inspection to ascertain their disease-free status. Following this, the fruit samples were transported to the laboratory, where the treatments were administered. A series of solutions were prepared from the nanoparticle samples obtained, with concentrations of 25, 50 and 100 mg L^{-1}, respectively. The grapes were then divided into four batches. The first batch was immersed in distilled water as a control, while the subsequent batches were immersed in solutions containing varying concentrations of nanoparticles (25, 50, or 100 mg L) for a duration of 2 min, respectively. The coated grapes (50 g) were placed into 100-ml PET clamshells and stored at 4°C for a period of 20 d. The measurements and analyses listed below were performed during or at the end of this period.

Characterization of the synthesized nanoparticles

UV-visible and Fourier transform infrared spectroscopy (FTIR) techniques were used for characterization. A UV–visible spectrophotometer was used as the internal standard to obtain the optical properties of the nanoparticles over a wavelength range of 200–800 nm. FTIR spectroscopy was used to determine the different functional groups present in the synthesized nanoparticles.

Firmness, color assessment, and respiration rate of berries

Fruit firmness was assessed using a texture analyzer (TA-XT Plus) with a 2 mm diameter and 76 mm long probe. The values obtained were expressed in Newtons (N). The color of the fruit's skin was measured with a hand-held reflectometer, and L*, a*, and b* color coordinates were recorded. L* indicates brightness or darkness, a* indicates the red to green axis, and b* indicates the yellowness to blueness axis. The respiration rate was estimated by incubating 50 g of the fruit sample in clamshells for 24 h at 4 °C and expressed in units of ml kg^{-1} h^{-1}.

Physical and chemical properties of berry juice

The fruit juice was pressed and filtered through cheesecloth for physico-chemical analysis. Quantitation of total soluble solids was done by a refractometer, and estimation of total titratable acidity was done by NaOH (0.1 N) titration (pH end-point 8.2) and then calculated as a percentage tartaric acid. The pH was measured with a digital pH meter.

Statistical analysis

Experiment was conducted using a completely randomized design in five replicates per treatment. One-way analysis of variance (ANOVA) was performed for all the data using PC SAS software version 9.1 to determine the effect of SPNP concentrations on the measured parameters. In those situations where the differences were established to be significant ($p < 0.05$), post-hoc analysis was used with Duncan's multiple range test (DMRT) to distinguish means. DMRT was chosen because it can compare more than one treatment mean while maintaining a good balance between protection against Type I error and sensitivity. DMRT is particularly useful in agriculture and biological science when treatment effects are small and where identification of homogeneous groups of treatment is crucial to interpretation.

RESULTS and DISCUSSION

Characterization of nanoparticles

UV-visible absorption spectra for *Sideritis pisidica* Boiss. & Heldr. extract, $CaCl_2$ precursor, and as-synthesized nanoparticles are represented in Figure 1. The ultraviolet region of the spectrum exhibited intense and broad absorption by the plant extract, particularly between 250–350 nm, which is typically attributed to π–π* transitions in phenolic compounds and aromatic rings of flavonoids, phytochemicals extensively documented in the *Sideritis* genus (Karakaya et al., 2017). On the other hand, the $CaCl_2$ solution had minimal or no absorption as would be expected for an inorganic salt that has no conjugated chromophores. The absorption spectrum of the resulting nanoparticles, however, showed a significant shift and broadening of the absorption range. Specifically, the nanoparticle solution showed enhanced intensity of absorption and a red-shifted shoulder into the visible region (400–600 nm) that confirms the production of the nanoparticles. This transformation is invariably accompanied by electronic structure changes, particle size reduction, and surface modification, changes typical of plant-mediated synthesis of nanomaterials (Iravani, 2011). The observations confirm that *S. pisidica* phytoconstituents in the extract not only supported the reduction of calcium ions but also capped nanoparticles through capping processes. Spectral results hence confirm efficient biosynthesis of calcium-based nanoparticles in a green synthesis process.

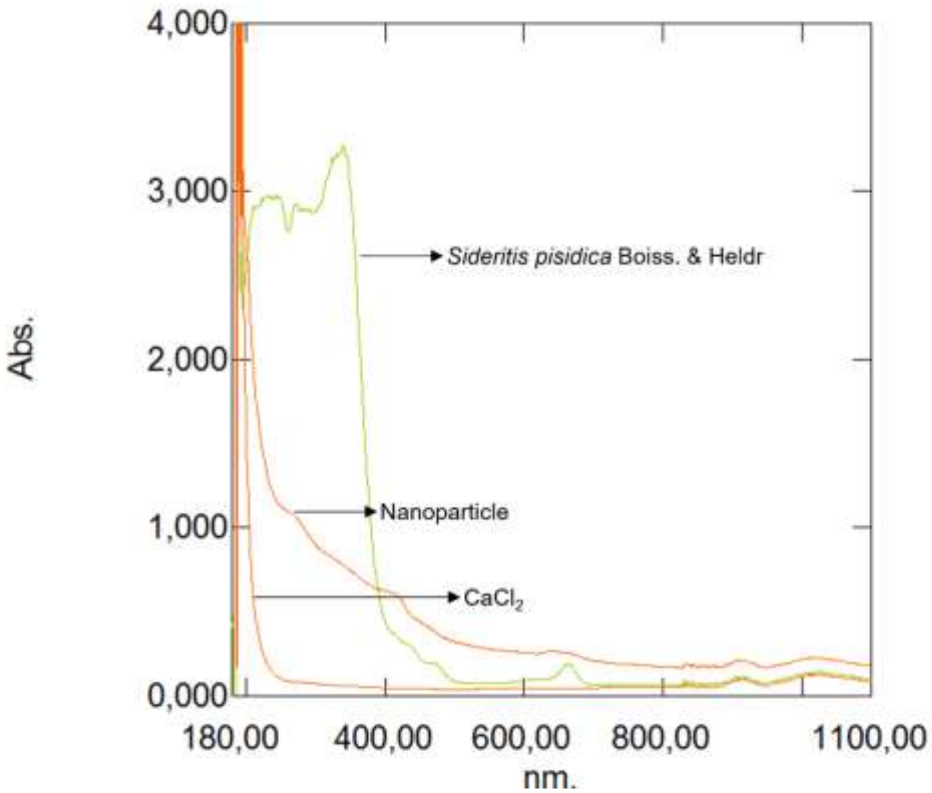

Figure 1. UV-visible spectrum of nanoparticles
Şekil 1. Nanopartiküllerin UV-görünür spektrumu

The FTIR spectrum provided in this example clearly shows the efficient green synthesis of *Sideritis pisidica* Boiss. & Heldr. derived calcium-based nanoparticles from the extract and $CaCl_2$ precursor (Figure 2). The pure $CaCl_2$ had nearly negligible characteristic peaks due to its plain ionic character. The blue line of *S. pisidica* extract showed broad bands at ca. 3300 cm^{-1}, which are assignable to phenolic groups O–H stretching vibrations, and peaks at ca. 1600 cm^{-1} and 1400 cm^{-1} corresponding to C=C aromatic and C–H bending vibrations, respectively (Weyer et al., 2002). In the red region of synthesized nanoparticles, peak shift and widening of the O–H band, and the emergence of new bands at 1000–1100 cm^{-1} (stretching of C–O bond) and at 870 cm^{-1} (Ca–O bond) showed interaction among phytochemicals and Ca^{2+} ions, indicating nanoparticle development (Jadhav et al., 2022; Gupta et al., 2025). These were signs of effective capping and stabilization of calcium-based nanoparticles through bioactive ingredients in *S. pisidica*, in agreement with findings from other plant-mediated syntheses.

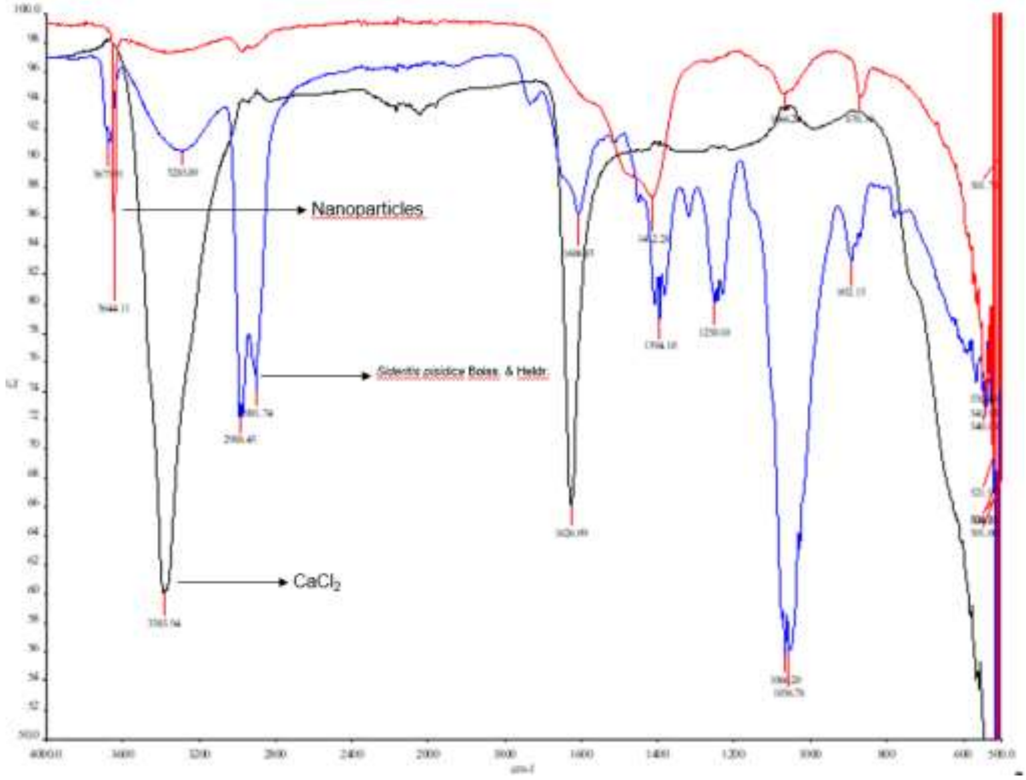

Figure 2. FTIR spectrum of nanoparticles. Black lines represent $CaCl_2$, the blue line represents *Sideritis pisidica* Boiss. & Heldr., and the red lines represent the nanoparticles.

Şekil 2. Nanopartiküllerin FTIR spektrumu. Siyah çizgiler $CaCl_2$, mavi çizgi Sideritis pisidica Boiss. & Heldr. ve kırmızı çizgiler nanopartikülleri temsil etmektedir.

Firmness

Firmness is one of the important fruit quality traits in grape berries, which significantly determines shelf life and consumer acceptance upon harvesting. The initial firmness at the beginning of the experiment was 2.02 ± 0.07 N (Figure 3). The firmness values slightly decreased through day 5 and ranged from 1.86 ± 0.08 N (control) to 1.95 ± 0.09 N (100 mg L^{-1} SPNP), but no statistically significant differences were observed. The same pattern was observed through day 10, where the firmness values were also not statistically different among treatments (ranging from 1.77 ± 0.11 N to 1.88 ± 0.11 N). On day 15, similar to previous days, no treatment showed statistically significant differences, and the firmness values ranged from 1.71 ± 0.06 N to 1.80 ± 0.15 N. Until the end of the storage period (day 20), the vague decrement in firmness continued, and the values of all groups ranged from 1.69 ± 0.11 N (control) to 1.73 ± 0.12 N (100 mg L^{-1} SPNP), with no significant differences among treatments.

These results are partially consistent with previous studies, wherein nanoparticle coatings, especially those based on chitosan and silver nanoparticles, were reported to be ineffective in retaining firmness in various fruits during storage by minimizing oxidative stress, water loss, and microbial spoilage (Salem et al., 2019; Gemail et al., 2023). Other studies, however, still went on to prove that nanoparticles are able to function towards preventing postharvest softening through mechanisms such as inhibiting ethylene, stabilization, and antimicrobial function of the cell wall (Shan et al., 2023).

Color properties

The effect of *Sideritis pisidica* Boiss. & Heldr. nanoparticle treatments on the surface color of grape berries, in the CIELAB color space (L*, a*, b*), is presented in Figure 4. The value for lightness (L*) was not significantly affected by treatments during the 20-day cold storage. All groups had similar L* values (32.08–33.36) with overlapping standard errors on day 0, indicating no difference at the initial stage. On day 20, L* values ranged from 32.65 ± 0.74 (50 mg L^{-1}) to 33.75 ± 1.04 (100 mg L^{-1}), which was still not statistically different due to overlapping standard errors. No statistically significant or consistent trend among treatments was observed during the storage period, suggesting that SPNP treatment had no detectable effect on grape skin lightness.

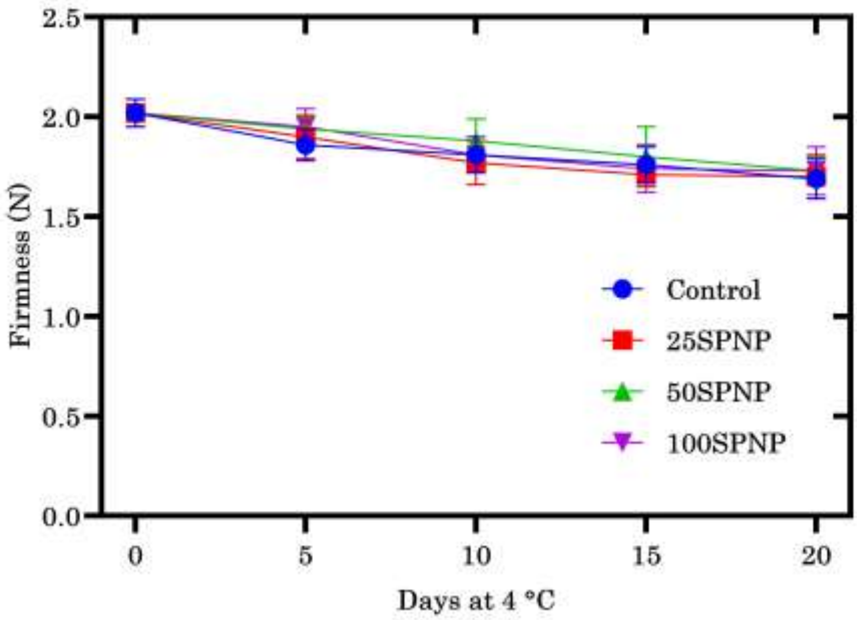

Figure 3. Firmness of grape berries treated with 25, 50 or 100 mg L^{-1} calcium-based nanoparticle prepared from *Sideritis pisidica* Boiss. & Heldr., and stored at 4 °C for 20 days. Vertical lines represent the standard error of the means.

Şekil 3. Sideritis pisidica Boiss. & Heldr.'dan hazırlanan 25, 50 veya 100 mg L^{-1} kalsiyum bazlı nanopartikül ile muamele edilen ve 20 gün boyunca 4 °C'de saklanan üzüm tanelerinin sertliği. Dikey çizgiler ortalamaların standart hatasını temsil etmektedir.

The a* values, which correspond to the green-red axis (negative values for greenness), were in the negative range for all samples and time points, indicating that the grapes did not alter their green colors during storage (Figure 4). On day 0, a* values ranged from –0.92 ± 0.18 to –1.44 ± 0.20, and on day 20, values ranged from –0.72 ± 0.40 to –0.97 ± 0.19. While small variations toward less negative values occurred in a few treatments over time (indicating a loss of greenness), these were within overlapping error ranges and cannot be deemed statistically significant.

The b* values (blue-yellow spectrum) increased slightly in all groups, with control samples rising significantly from 4.66 ± 0.44 to 5.76 ± 0.29, suggesting yellow pigment accumulation (day 15: $F(4, 16) = 3.57$ $p < .05$; day 20: $F(4, 16) = 4.03$ $p < .03$; Figure 4). The SPNP treatments showed the least b* value increase starting from day 15, potentially indicating better color preservation, especially at higher concentrations.

Overall, the color stability observed under SPNP treatment is consistent with earlier reports that plant-based nanoparticle coatings can provide some degree of protection from discoloration without greatly influencing natural pigment development. Phenolic compounds found in *Sideritis* spp. can particularly influence a* and b* values by inhibiting polyphenol oxidase and preserving carotenoids (Shan et al., 2023). However, the non-linear response across concentrations suggests complex interactions between nanoparticle dosage and color stability mechanisms that could involve both direct pigment protection and indirect metabolic effects (Dhall, 2013). These findings demonstrate the potential of SPNP treatments for maintaining postharvest color quality, though further research is needed to optimize concentration and elucidate underlying biochemical pathways.

Respiration rate

Respiration rates of the *Sideritis pisidica* Boiss. & Heldr. nanoparticles-treated grape berries under 4 °C storage for 20 d are presented in Figure 5. The control on day 5 had a respiration rate of 32.33 ± 6.24 ml CO_2 kg^{-1} h^{-1}, while the treatments ranged from 35.30 ± 11.09 to 39.30 ± 6.70 ml CO_2 kg^{-1} h^{-1}. However, no statistically significant differences were observed on day 5. The same trend was observed on day 10, where respiration rates ranged from 33.00 ± 6.18 ml CO_2 kg^{-1} h^{-1} (control) to 45.33 ± 6.70 ml CO_2 kg^{-1} h^{-1} (100 mg L^{-1} SPNP), but the overlap among error intervals prevents claims of statistically significant differences as well.

After 15 days, the mean respiration rate in the SPNP-treated fruit increased more steeply than in the control group, particularly in the 100 mg/l treatment group (69.00 ± 11.10 ml CO_2/kg/h compared to 39.00 ± 4.60 ml CO_2/kg/h in the control group; Figure 5). This resulted in a significant difference between the control and the 100 mg/l SPNP treatment group ($F(4, 16) = 4.25$ $p < .04$). On day 20, there was a further increase in separation, with

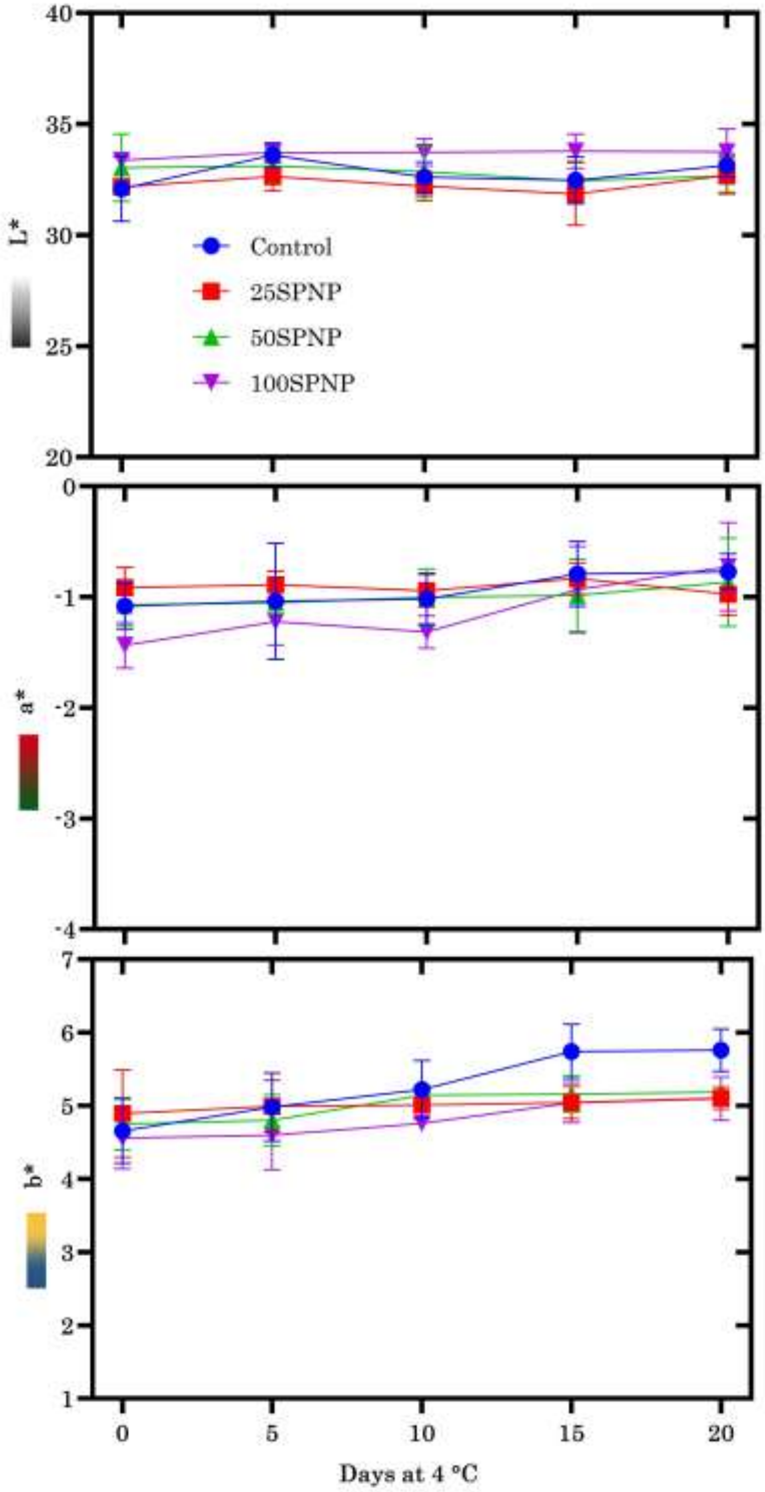

Figure 4. CIE L*, a* and b* color space values of grape berries treated with 25, 50 or 100 mg L^{-1} calcium-based nanoparticle prepared from *Sideritis pisidica* Boiss. & Heldr., and stored at 4 °C for 20 days. Vertical lines represent the standard error of the means.

Şekil 4. Sideritis pisidica Boiss. & Heldr.'dan hazırlanan 25, 50 veya 100 mg L^{-1} kalsiyum bazlı nanopartikül ile muamele edilen ve 20 gün boyunca 4 °C'de saklanan üzüm berrelerinin CIE L, a* ve b* renk değerleri. Dikey çizgiler ortalamaların standart hatasını temsil etmektedir.*

the control at 47.67 ± 6.02 ml CO_2/kg/h, while the treatment with 100 mg/l SPNP reached 80.33 ± 14.43 ml CO_2/kg ·/h (F(4, 16) = 4.10 p < .03). This indicated a statistically significant rise in respiration rate due to high SPNP levels.

Previous studies have reported that nanoparticle-based coatings or plant extract treatments could influence respiration rates of fruit by altering gas exchange or impacting metabolism (Han et al., 2017; Zhou et al., 2008). While the majority of food coatings reduce respiration rate by creating semi-permeable barriers (Valverde et al., 2005), some nanoparticle treatments have been associated with increased metabolic activity due to the material's bioactivity and concentration (de Oliveira et al., 2023). The bioactive and antioxidant potential of *Sideritis* extracts (González-Burgos et al., 2011) may influence enzymatic activities related to respiration, although in this work, no repeated increases were observed except at the highest SPNP concentration (100 mg L^{-1}).

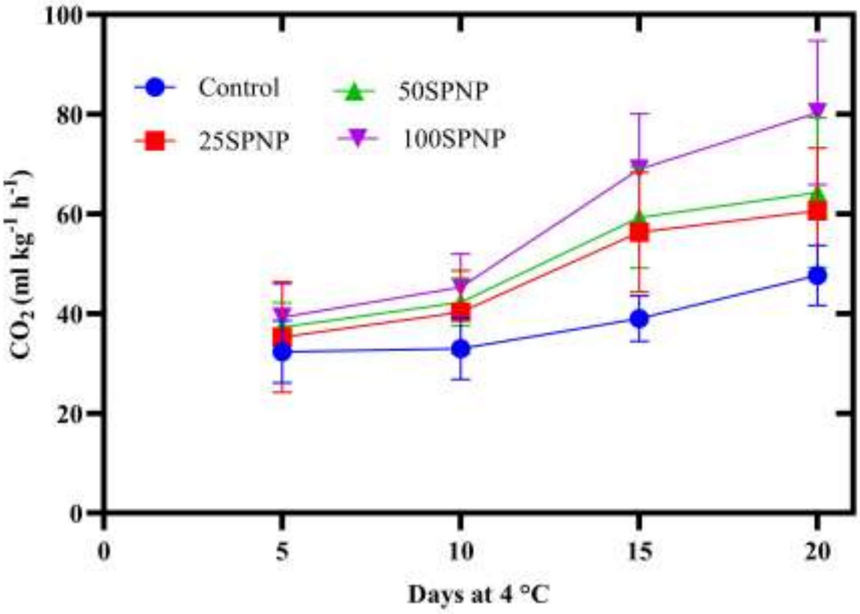

Figure 5. Respiration rate of grape berries treated with 25, 50 or 100 mg L^{-1} calcium-based nanoparticle prepared from *Sideritis pisidica* Boiss. & Heldr., and stored at 4 °C for 20 days. Vertical lines represent the standard error of the means.

Şekil 5. Sideritis pisidica Boiss. & Heldr.'dan hazırlanan 25, 50 veya 100 mg L-1 kalsiyum bazlı nanopartikül ile muamele edilen ve 20 gün boyunca 4 °C'de saklanan üzüm tanelerinin solunum hızı. Dikey çizgiler ortalamaların standart hatasını temsil etmektedir.

Total soluble solids content

Total soluble solids (TSS) content is an important index of grape sugar concentration and flavor, prevalently used to estimate fruit maturity and postharvest quality (Figure 6). At the beginning of the experiment, the TSS was 2.02 ± 0.07% for all treatments. During storage, TSS values decreased very slightly in all treatment. On day 5, the values ranged from 1.86 ± 0.08% for the control to 1.95 ± 0.09% for the treatment with 100 mg L^{-1} SPNP, with no statistically significant differences among treatments. The results regarding insignificant differences among treatments remained consistent on days 10, 15, and 20. On day 10, TSS ranged from 1.77 ± 0.11% (25 mg L^{-1} SPNP) to 1.88 ± 0.11% (50 mg L^{-1} SPNP), on day 15 from 1.71 ± 0.06% (25 mg L^{-1} SPNP) to 1.80 ± 0.15% (50 ml L^{-1} SNPP), and on day 20 from 1.69 ± 0.10% (control) to 1.73 ± 0.12% (100 mg L^{-1} SPNP).

The time-dependent decrease in TSS that was observed is consistent with the common metabolic processes that take place during storage, such as respiration and enzymatic hydrolysis of sugars (Ali et al., 2010; Valverde et al., 2005). In contrast to certain earlier work where the use of edible coating or nanoparticle treatment can lead to the retention of TSS after cold storage (Han et al., 2017; Singh & Packirisamy, 2022), employing SPNP in the presented concentration could not significantly affect values of TSS under these experimental conditions. Despite *Sideritis* extracts having long-identified antioxidant attributes (González-Burgos et al., 2011), the latter was not met regarding changing quantifiable levels of TSS for this group of experimental conditions.

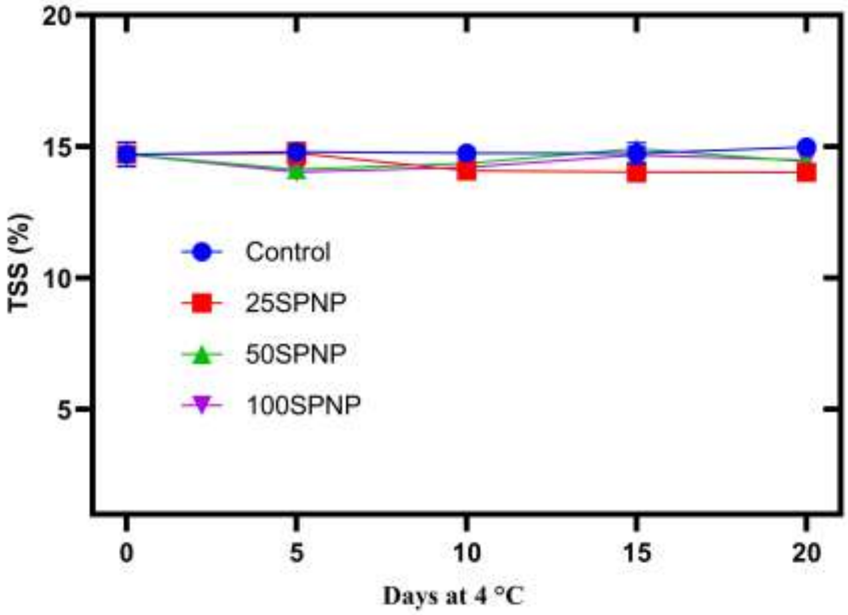

Figure 6. Total soluble solids (TSS) content of grape berries treated with 25, 50 or 100 mg L^{-1} calcium-based nanoparticle prepared from *Sideritis pisidica* Boiss. & Heldr., and stored at 4 °C for 20 days. Vertical lines represent the standard error of the means.

Şekil 6. Sideritis pisidica Boiss. & Heldr.'dan hazırlanan 25, 50 veya 100 mg L^{-1} kalsiyum bazlı nanopartikül ile muamele edilen ve 20 gün boyunca 4 °C'de depolanan üzüm tanelerinin toplam çözünür kuru madde (TÇKM) içeriği. Dikey çizgiler ortalamaların standart hatasını temsil etmektedir.

Titratable acidity and pH

Titratable acidity (TA) and pH changes of grape berries during 20 d of cold storage at 4 °C after *Sideritis pisidica* Boiss. & Heldr. nanoparticle treatments are shown in Figure 7. Initial TA and pH values were 0.42 ± 0.01% and 3.52 ± 0.05, respectively, on day 0. Titratable acidity slightly increased while pH initially rose and subsequently fell during storage, reflecting normal metabolic and ripening-related physiological changes in grapes.

On day 5, the TA in all the SPNP-treated samples (0.40–0.41%) was comparable to the control (0.42 ± 0.01%), with no statistical differences (Figure 7). The same pattern was observed on day 10. Although the 50 mg L^{-1} SPNP treatment had slightly lower titratable acidity (0.39 ± 0.01%), the difference was still within the error range compared to the other treatments. From day 15 onwards, acidity values in all the samples increased. On day 20, the titratable acidity of the SPNP-treated grapes ranged from 0.48 ± 0.01 to 0.50 ± 0.03%, slightly higher than the control (0.46 ± 0.01%) but again showed no statistical differences in treatment at this stage.

pH levels followed the opposite trajectory (Figure 7). From day 0 to day 10, pH rose in all groups, reaching its highest at day 10 for both control (3.71 ± 0.03) and SPNP treatments (3.58 ± 3.67) with very small differences between concentrations. Although the average values varied in these aspects, all groups had overlapping standard errors, preventing any statistically significant interpretation. From days 15 to 20, pH dropped in all treatments, consistent with the accumulation of organic acids typically observed during late storage. pH values dropped to 3.43–3.55 by day 20, with the lowest pH observed in the 100 mg L^{-1} SPNP (3.43 ± 0.03) and the highest in the control (3.55 ± 0.03). Again, although differences appeared obvious, standard error ranges were close enough to suggest no statistically significant deviations.

Trends were consistent with previous reports of small changes in pH and acidity due to natural fruit senescence or microbial interaction during storage. For example, Serrano et al. (2005) reported that postharvest treatments such as edible coatings or bioactive compounds can influence acidity and pH, but significant changes were more frequently dependent on treatment type and storage time. In the current study, even though SPNP treatments did not significantly alter titratable acidity or pH compared to control, their application did not disrupt natural ripening-associated trends, revealing a neutral effect on acid metabolism under cold storage conditions.

Toxicological considerations and safety assessments of nanoparticles

While the present research demonstrated promising results for SPNPs in maintaining postharvest color, the potential toxicological implications of nanoparticle application in food systems warrant careful consideration.

Plant-mediated synthesis, such as the use of *Sideritis pisidica* extract, is generally considered safer and more eco-friendly compared to chemical synthesis (Iravani, 2011; Mittal et al., 2013). However, nanoparticles can exhibit biological activity due to their nanoscale size and extreme reactivity in any context. Nanoparticles have been shown to cause oxidative stress, inflammation, and genotoxicity in biological systems depending on particle size, concentration, and surface chemistry (Manke et al., 2013; Hossain et al., 2015). This study did not identify acute phytotoxicity, but higher respiration rates at higher SPNP concentrations could indicate a metabolic response to mild stress. This finding underscores the need for ongoing safety testing, including assessments of cytotoxicity, bioaccumulation potential, and consumer acceptability, particularly when using nanomaterials in food-grade applications. Follow-up studies should also include comprehensive toxicological evaluations using in vitro gastrointestinal test systems to confirm the safety of SPNPs in industrial food applications (Ashfaq et al., 2022; Shah et al., 2024).

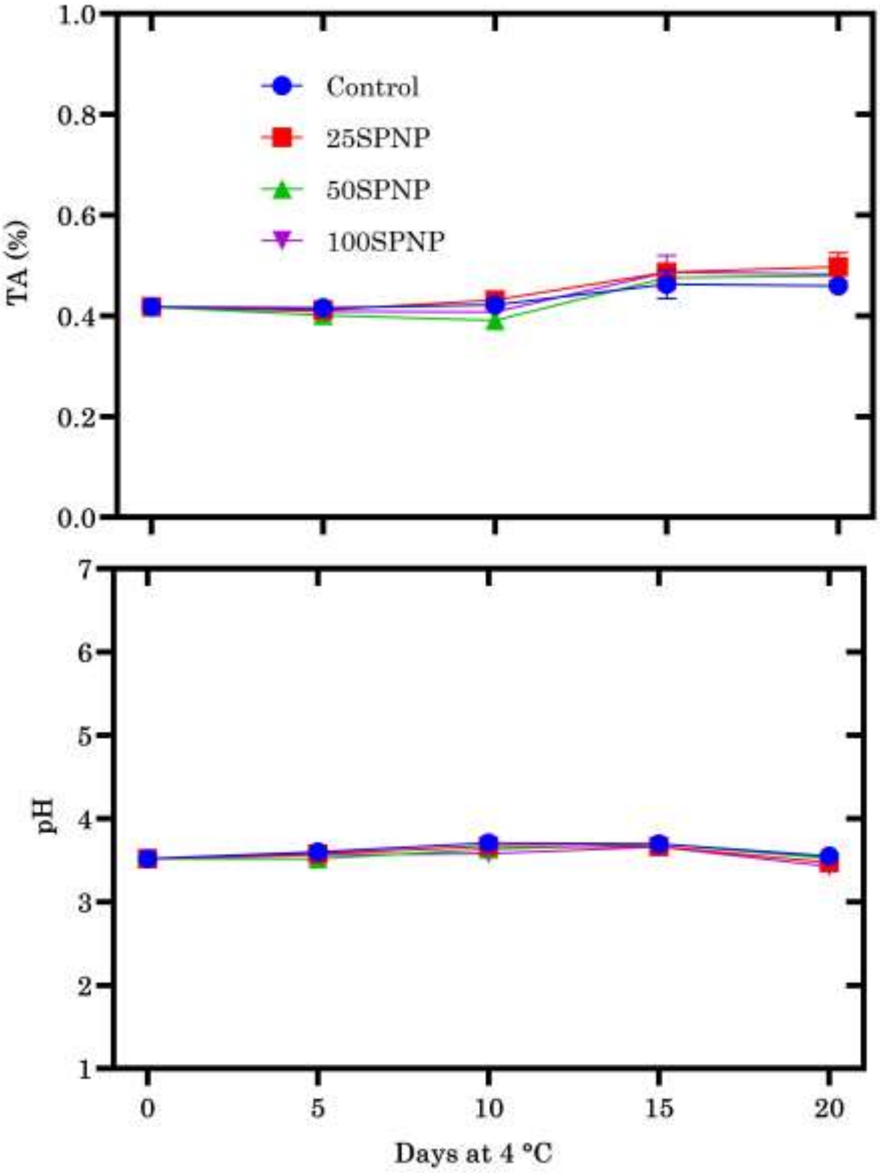

Figure 7. Titratable acidity (TA) and pH of grape berries treated with 25, 50 or 100 mg L^{-1} calcium-based nanoparticle prepared from *Sideritis pisidica* Boiss. & Heldr., and stored at 4 °C for 20 days. Vertical lines represent the standard error of the means.

Şekil. 7. Sideritis pisidica Boiss. & Heldr.'dan hazırlanan 25, 50 veya 100 mg L^{-1} kalsiyum bazlı nanopartikül ile muamele edilen ve 20 gün boyunca 4 °C'de depolanan üzüm tanelerinin titre edilebilir asitliği (TA) ve pH'ı. Dikey çizgiler ortalamaların standart hatasını temsil etmektedir.

CONCLUSION

This study showed that the efficient green synthesis of calcium-based nanoparticles using *Sideritis pisidica* Boiss. & Heldr. Leaf extract is feasible. Nanoparticle characterization using UV-visible and FTIR spectroscopy validated the efficient reduction and stabilization of calcium ions by the phytochemicals of *S. pisidica*. The presence of functional groups like phenolics and flavonoids not only enhanced the biosynthesis process but most likely also enhanced the biological activity of nanoparticles.

Postharvest quality attributes such as fruit firmness, respiration rate, TSS, TA, pH, and skin color were analyzed to evaluate the effectiveness of SPNP treatments at concentrations of 25, 50, and 100 mg L^{-1}. Fruit firmness, a key quality attribute, decreased during the storage period in all treatment groups, including the control, with no statistically significant differences. Similarly, SPNP treatment did not have a significant impact on TA, pH, or TSS content, indicating that under the tested conditions, SPNP did not inhibit or delay ripening or senescence-related metabolic processes.

However, significant variations were observed in the respiration rates and color parameters of treated grapes. Grapes treated with 100 mg L^{-1} SPNP showed significantly higher respiration rates during the later storage phase, possibly due to increased metabolic activity induced by the bioactive nanoparticle coating. In terms of color retention, SPNP treatments, particularly at higher levels, preserved yellow pigmentation by suppressing the increase in b* value, indicating possible protection against oxidative browning and pigment degradation.

In general, the utilization of SPNPs as a postharvest treatment has remained limited and has not reached the level of benefits that were anticipated. However, their role in regulating physiological processes, such as respiration and color retention, is encouraging. The use of *S. pisidica* extract in nanosynthesis offers a green and sustainable route for the synthesis of nanomaterials that can substitute conventional chemical preservatives. The study offers blueprints for the utilization of plant-mediated calcium nanoparticles for postharvest fruit preservation. Future studies should target maximizing the concentration of SPNP, mode of application, and combination of such treatments with others to maximize efficacy and ensure safety and consumer acceptability in commercial applications.

Contribution Rate Statement Summary of Researchers

The authors declare that they have contributed equally to the article.

Conflict of Interest

No conflict of interest was declared by the authors.

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