



Plant Archives

Journal homepage: <http://www.plantarchives.org>

DOI Url : <https://doi.org/10.51470/PLANTARCHIVES.2026.v26.supplement-1.253>

INFLUENCE OF OZONE FUMIGATION ON RAISINS TO SUBSTITUTE SULPHUR FUMIGATION

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(Date of Receiving : 09-10-2025; Date of Acceptance : 15-12-2025)

ABSTRACT

The study entitled “*Influence of ozone fumigation on raisins to substitute sulphur fumigation*” was conducted in the Department of Postharvest Management, College of Horticulture, Bagalkot, Karnataka during 2024–25 to evaluate the effect of ozone fumigation on the shelf life of raisins as an alternative to sulphur dioxide fumigation. The experiment comprised seven treatments, viz., sulphur dioxide fumigation at 3 g kg⁻¹ (T₁), dipping of grape bunches in ozonated water for 3 minutes (T₂), and ozone fumigation for 10 (T₃), 15 (T₄), 20 (T₅), 25 (T₆) and 30 minutes (T₇). The treated raisins were stored under ambient (29 ± 1 °C) and cold (5 ± 2 °C) storage conditions. Values in parentheses represent mean values under ambient storage followed by cold storage. Among the treatments, T₄ and T₅ (ozone fumigation for 15 and 20 minutes, respectively) proved most effective in retaining quality during storage. The highest TSS was recorded in T₄ (69.89, 69.95 °Brix) and T₅ (69.84, 69.90 °Brix), and titratable acidity was better retained in T₄ (0.68, 0.70%) and T₅ (0.69, 0.71%). Instrumental colour analysis revealed significantly higher lightness values in T₄ (15.15, 17.38) and T₅ (14.27, 16.66), along with improved yellowness in T₄ (24.43, 26.62) and T₅ (23.53, 25.55). Moisture content and water activity were optimally maintained in T₄ (12.37, 13.07%; 0.31, 0.30) and T₅ (12.57, 13.30%; 0.31, 0.30). The findings clearly establish that ozone fumigation for 15–20 minutes effectively preserves the physicochemical and colour quality of raisins under both storage environments and can be recommended as a safe and efficient alternative to sulphur fumigation for raisin processing.

Keywords : Raisins, ozone fumigation, sulphur dioxide, shelf life.

Introduction

Raisins, defined as dehydrated form of grape berries, are categorized under traditional dried fruits due to the absence of exogenous sugar incorporation during their processing. From a nutritional standpoint, raisins represent a concentrated source of energy and micronutrients, as dehydration leads to a substantial reduction in moisture content, thereby concentrating intrinsic bioactive and nutritive constituents. Sulphur dioxide (SO₂) fumigation is conventionally applied in raisin processing to inhibit microbial spoilage, prevent enzymatic browning and extend shelf life. This

treatment typically involves exposing the dried grapes to SO₂ gas, followed by storage in controlled conditions to maintain microbial stability. However, the process faces significant challenges due to the difficulty in regulating SO₂ concentrations, as variations in relative humidity can lead to uncontrolled gas release and accumulation of residues exceeding the maximum permissible limit of 10 mg/kg. Excessive SO₂ residues are of particular concern in international trade, where stringent regulations have led to the rejection of export consignments in several cases due to non-compliance with residue limits. In addition to potential health risks such as sulphite-induced allergic

reactions, high SO₂ concentrations can cause bleaching of the berries and rachis, deterioration of organoleptic properties and reduced consumer acceptance (de Aguiar *et al.*, 2023). In response to these limitations, there is increasing interest in the development of residue-free and environmentally sustainable alternatives for raisin preservation. Alternative treatments such as modified atmosphere packaging, ethanol vapor application, chitosan-based coatings, heat treatments and ozone (O₃) fumigation have been explored for their efficacy in controlling microbial contamination without the drawbacks associated with SO₂. Among these, ozone fumigation is emerging as a promising technology due to its strong oxidative antimicrobial action and lack of chemical residues.

Ozone (O₃), a triatomic form of oxygen, is generated when molecular oxygen (O₂) reacts with oxygen free radicals. It is characterized by a sharp, pungent odor and appears as a blue gas when produced from dried air, though it remains colorless when generated from high-purity oxygen. At concentrations typically used in food processing, its color is not perceptible. Ozone holds Generally Recognized as Safe (GRAS) status for applications in the food industry and can be utilized either in its gaseous form or dissolved in water, depending on the intended use.

With the rising health consciousness among Indian consumers, the long-term sustainability of sulphur dioxide (SO₂) treatment in raisin processing has come under scrutiny, raising the possibility of future regulatory restrictions on its use for certain commodities. In response to these challenges, ozone fumigation has emerged as a promising and technologically advanced alternative to conventional SO₂ fumigation. Ozone offers multiple benefits, functioning as a safer and healthier option for both the product and end consumers. Additionally, its residue-free nature and environmentally sustainable application make it a simpler yet effective solution for addressing postharvest preservation challenges in raisins.

Materials and Methods

Preparation of fruits

Grapes of the cultivar Manik Chaman, harvested from JMK Farms, Mashal, were utilized in this study. Fully matured and ripened grape bunches were collected and shifted to the commercial raisin processing unit established at the farmer's field.

Pre-treatment

The grape bunches underwent a pre-treatment process involving immersion in a standardized dipping

solution comprising of 1.5 per cent ethyl oleate and 2.5 per cent potassium carbonate. The bunches were submerged in this solution for a controlled duration of three minutes to facilitate wax layer disruption and enhance subsequent drying efficiency.

Treatment imposition

The first treatment involved sulphur dioxide fumigation at a rate of three g/kg on the 7th day of drying, while the second treatment consisted of dipping the grape bunches in ozonated water for 3 minutes. Treatments 3, 4, 5, 6, and 7 involved ozone fumigation for durations of 10, 15, 20, 25, and 30 minutes, respectively.

Table 1 : Treatment details.

Treatment	Postharvest treatment
T ₁	Sulphur dioxide fumigation @3g/kg
T ₂	Dipping in ozonated water for 3 minutes
T ₃	Ozone fumigation for 10 minutes
T ₄	Ozone fumigation for 15 minutes
T ₅	Ozone fumigation for 20 minutes
T ₆	Ozone fumigation for 25 minutes
T ₇	Ozone fumigation for 30 minutes

Observations recorded:

Total soluble solids (°B)

The Total Soluble Solids (TSS) content of raisin samples was determined using a digital refractometer. A representative quantity of the sample was homogenised with an equal volume of distilled water (1:1 w/v ratio) to facilitate extraction. The homogenate was then filtered, and the clear extract was used for measurement. The TSS was recorded directly as °Brix from the refractometer scale.

Titrateable acidity (%)

The titrateable acidity of grape berries was determined using the titration method. A 5 ml aliquot of juice was titrated with a standard 0.1N NaOH solution, using phenolphthalein as the indicator. The titration endpoint was noted by the appearance of a stable pink colour, corresponding to a pH of 8.1. The titrateable acidity was then calculated and expressed as a percentage, in terms of tartaric acid equivalent (Srivastava and Sanjeevkumar, 1998).

Titrateable

$$\text{acidity (\%)} = \frac{\text{Titre value} \times \text{N of NaOH} \times \text{Vol. made up} \times \text{Eq. weight of acid}}{\text{Vol. of aliquot} \times \text{Vol. of sample taken} \times 1000} \times 100$$

Colour (L*, a*, b*)

The colour characteristics of raisin samples were determined using a Hunter Colour Meter (Model: Color Flex® EZ Standard Box) equipped with an 8

mm diameter aperture. Raisin samples were evenly spread to cover the measurement aperture, and three consecutive readings were recorded for each sample, with the average value reported.

Moisture (%) and water activity

The moisture content of raisins was determined using a Radwag moisture analyser (Model: MAC 50, Make: Poland). Exactly one gram of raisins was placed in the sample dish for analysis. A water activity meter (Model: Labswift-aw, Make: Novasina) was employed for the determination of water activity in raisin samples.

Results and Discussion

Total soluble solids (TSS)

Total soluble solids of raisins declined progressively under both ambient and cold storage, with a slower reduction at low temperature (Fig. 1). Across treatments, the overall mean TSS decreased from about 69.7 °B initially to 65.26 °B in ambient storage and 67.36 °B in cold storage by 150 days. This decline is primarily attributed to non-enzymatic browning reactions wherein reducing sugars participate in Maillard reactions, gradually lowering soluble solids (Winkler *et al.*, 1974). Cold storage minimized these losses by suppressing metabolic activity. Among the treatments, ozone fumigation for 15 minutes (T_4) and 20 minutes (T_5) consistently retained the highest TSS values in both environments, with mean values of 69.89–69.95 °B in cold storage and 69.84–69.90 °B in ambient storage. Their effectiveness may be linked to ozone's antimicrobial and oxidative action, which limits microbial growth and retards biochemical degradation of sugars.

Titrateable acidity (%)

Titrateable acidity (TA) of raisins exhibited a consistent declining trend under both ambient and cold storage, with cold conditions moderating the rate of reduction (Fig 2). Across treatments, overall TA decreased from an initial mean of about 0.73–0.74% to 0.51% in ambient storage and 0.55% in cold storage by 150 days. The decline in acidity is primarily attributed to the utilization of organic acids as respiratory substrates through the tricarboxylic acid cycle, coupled with moisture loss and compositional changes during storage (Kays and Paull, 2004). Lower temperatures slowed these metabolic processes, resulting in better acid retention under cold storage (Wills *et al.*, 2007). Among the treatments, ozone fumigation for 15 minutes (T_4) and 20 minutes (T_5) maintained the highest acidity in both storage environments, recording mean values of 0.68–0.70% and 0.69–0.71%,

respectively. The superior stability of these treatments is associated with ozone's antimicrobial and antioxidative action, which reduces microbial load and slows biochemical degradation of organic acids.

Instrumental Colour (L^* , a^* , b^*)

Instrumental L^* values of raisins declined progressively under both storage conditions (Fig 3), reflecting surface darkening associated with non-enzymatic browning, including Maillard reactions and oxidation of phenolics (Miranda and Berna, 2021). Despite this natural decrease, clear treatment differences were evident. Ozone fumigation for 15 minutes (T_4) and 20 minutes (T_5) maintained the highest brightness, with mean L^* values of 15.15 and 14.27 in ambient storage and 17.38 and 16.66 in cold storage, respectively. Their superior colour retention is attributed to effective suppression of oxidative reactions and reduced microbial activity (Karaca and Velioglu, 2007). Across all treatments, a^* values (red–green axis) increased steadily during storage, rising from an overall mean of 7.32 initially to 12.05 at 150 days, indicating a gradual shift toward red–brown tones (Fig 4). Among treatments, T_2 (ozonated water dip) recorded the highest mean a^* value (11.54), reflecting enhanced red pigmentation linked to mild oxidative priming. In contrast, T_4 and T_5 consistently showed the lowest a^* values (8.86 and 9.19, respectively), demonstrating effective suppression of red–brown colour development. A similar trend was observed for b^* values, where the yellow chromatic component declined gradually during the storage owing to pigment degradation typical of dried fruits (Fig 5). Treatments T_4 and T_5 again recorded the highest b^* values of 24.43 and 23.53 under ambient storage and 26.62 and 25.55 under cold storage, indicating better preservation of yellow pigments.

Moisture (%)

The moisture content of raisins declined gradually under both ambient and cold storage (Fig 6), with reductions more controlled under refrigeration (14.29% to 13.66%) than under ambient conditions (14.29% to 11.74%), as lower temperatures suppress transpiration and metabolic activity in dried fruits (Canellas *et al.*, 1993). Across both environments, ozone fumigation for 15 minutes (T_4) and 20 minutes (T_5) maintained the most desirable moisture levels, with means of 13.07% and 13.30% in cold storage and 12.37% and 12.57% in ambient storage, ranges considered optimal for microbial safety and long-term stability (Kaya *et al.*, 2007).

Water activity

Water activity (a_w), showed a gradual yet controlled rise in raisins during storage (Fig 7), increasing from 0.30 to 0.35 under ambient conditions and remaining comparatively lower under cold storage (0.28–0.34), reflecting the reduced moisture equilibration achieved at low temperature (Roos, 2010). Across both storage conditions, ozone

fumigation for 15 minutes (T_4) and 20 minutes (T_5) consistently maintained the lowest a_w values (0.30–0.31), indicating superior control of surface moisture, reduced microbial activity, and restricted water mobility, likely due to ozone-induced microstructural stabilization and suppression of surface microbiota (Bialka *et al.*, 2004; Piechowiak *et al.*, 2022).

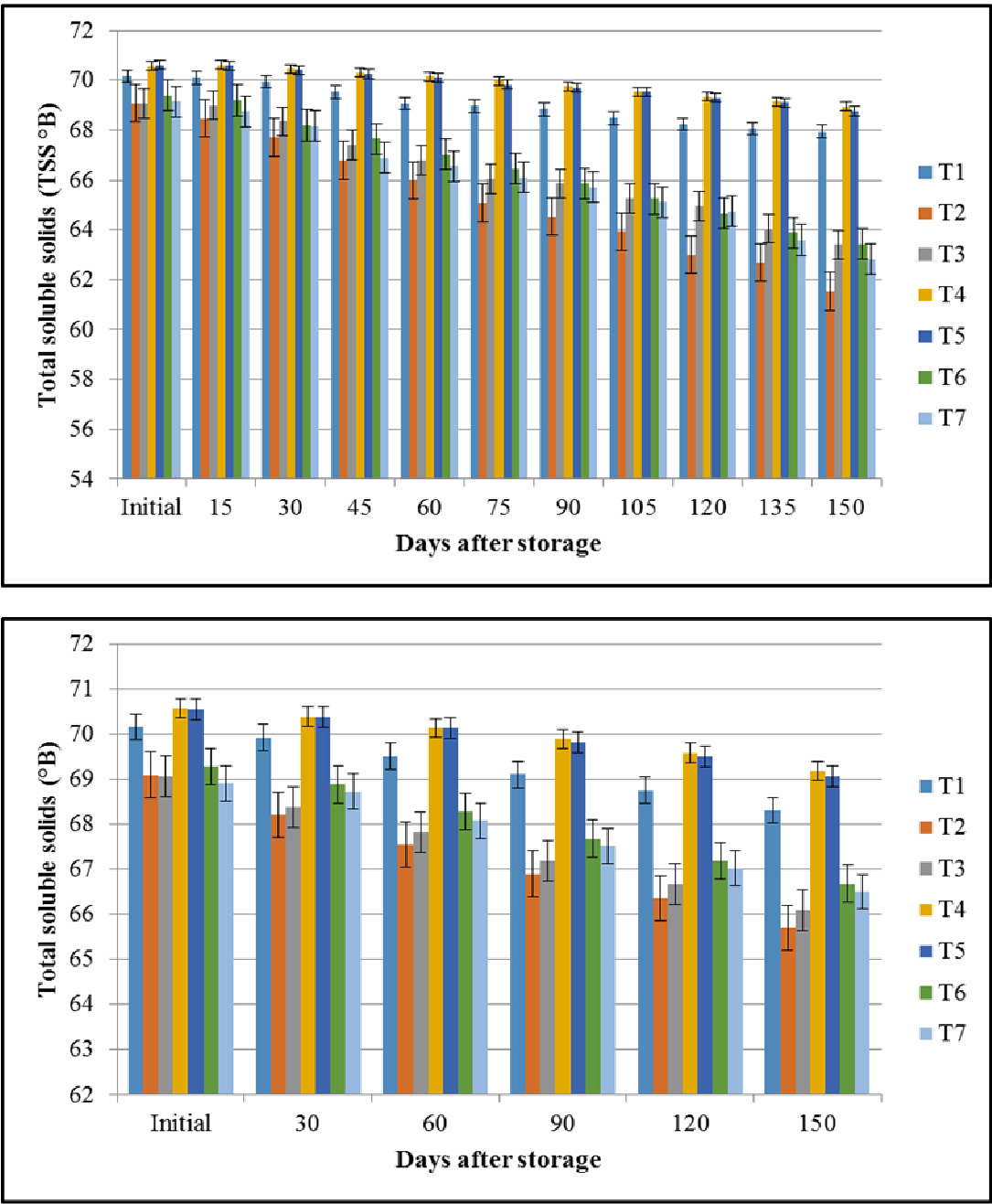


Fig. 1 : Effect of different postharvest treatments on total soluble solids of raisins under ambient storage ($29\pm1^{\circ}\text{C}$, $43\pm1\%$ RH) and cold storage ($5\pm2^{\circ}\text{C}$, RH- 85-95%)
 T_1 :Sulphur dioxide fumigation at 3g/kg
 T_2 :Dipping in ozonated water for 3 minutes
 T_3 :Ozone fumigation for 10 minutes
 T_4 :Ozone fumigation for 15 minutes
 T_5 :Ozone fumigation for 20 minutes
 T_6 :Ozone fumigation for 25 minutes
 T_7 :Ozone fumigation for 30 minutes

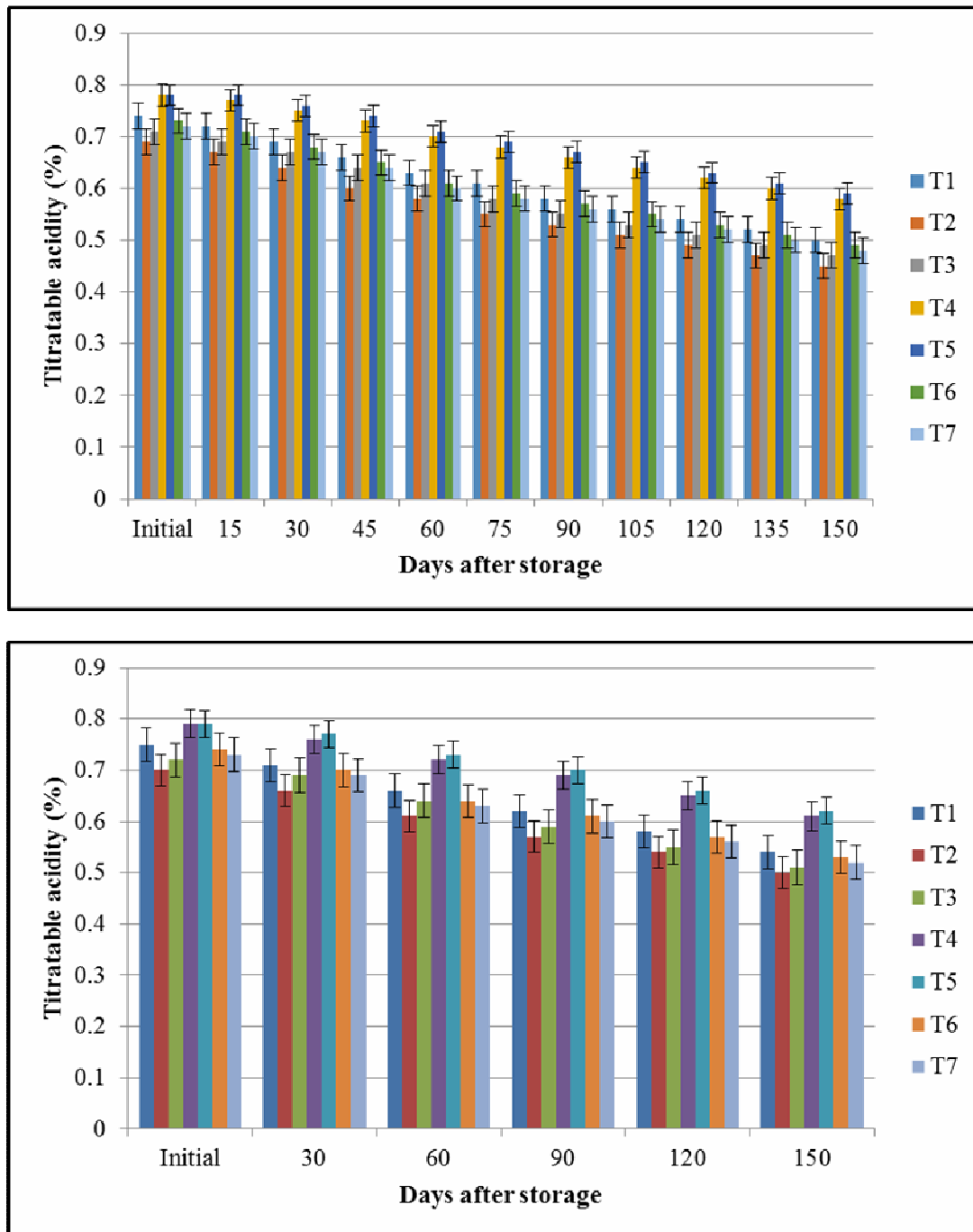


Fig. 2 : Effect of different postharvest treatments on titratable acidity of raisins under ambient storage (29±1°C, 43±1% RH) and cold storage (5±2°C, RH- 85-95%)

T₁ :Sulphur dioxide fumigation at 3g/kg

T₂ :Dipping in ozonated water for 3 minutes

T₃ :Ozone fumigation for 10 minutes

T₄ :Ozone fumigation for 15 minutes

T₅ :Ozone fumigation for 20 minutes

T₆ :Ozone fumigation for 25 minutes

T₇ :Ozone fumigation for 30 minutes

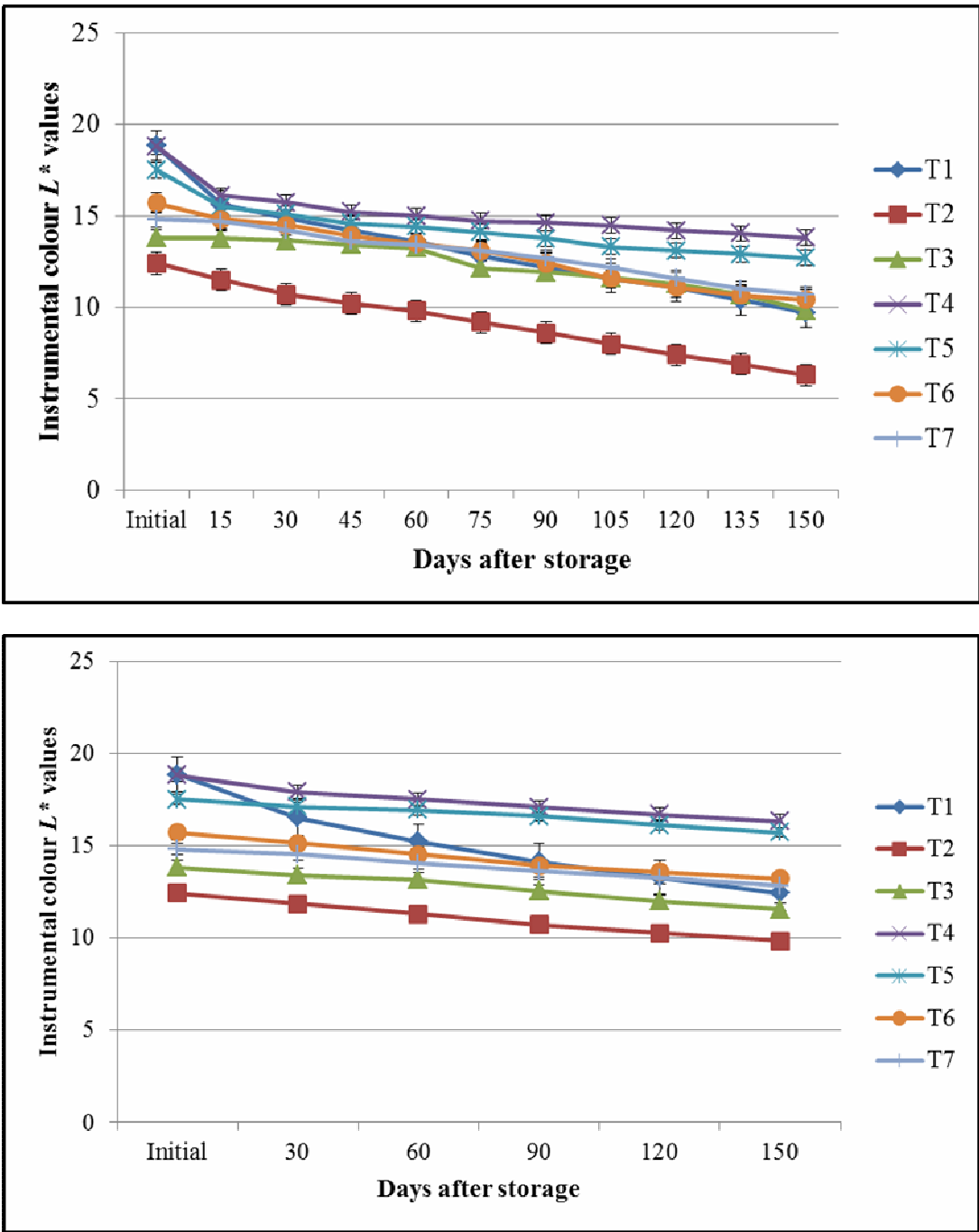


Fig. 3 : Effect of different postharvest treatments on instrumental L^* values of raisins under ambient storage (29±1°C, 43±1% RH) and cold storage (5±2°C, RH- 85-95%)

T₁ :Sulphur dioxide fumigation at 3g/kg T₅ :Ozone fumigation for 20 minutes
T₂ :Dipping in ozonated water for 3 minutes T₆ :Ozone fumigation for 25 minutes
T₃ :Ozone fumigation for 10 minutes T₇ :Ozone fumigation for 30 minutes
T₄ :Ozone fumigation for 15 minutes

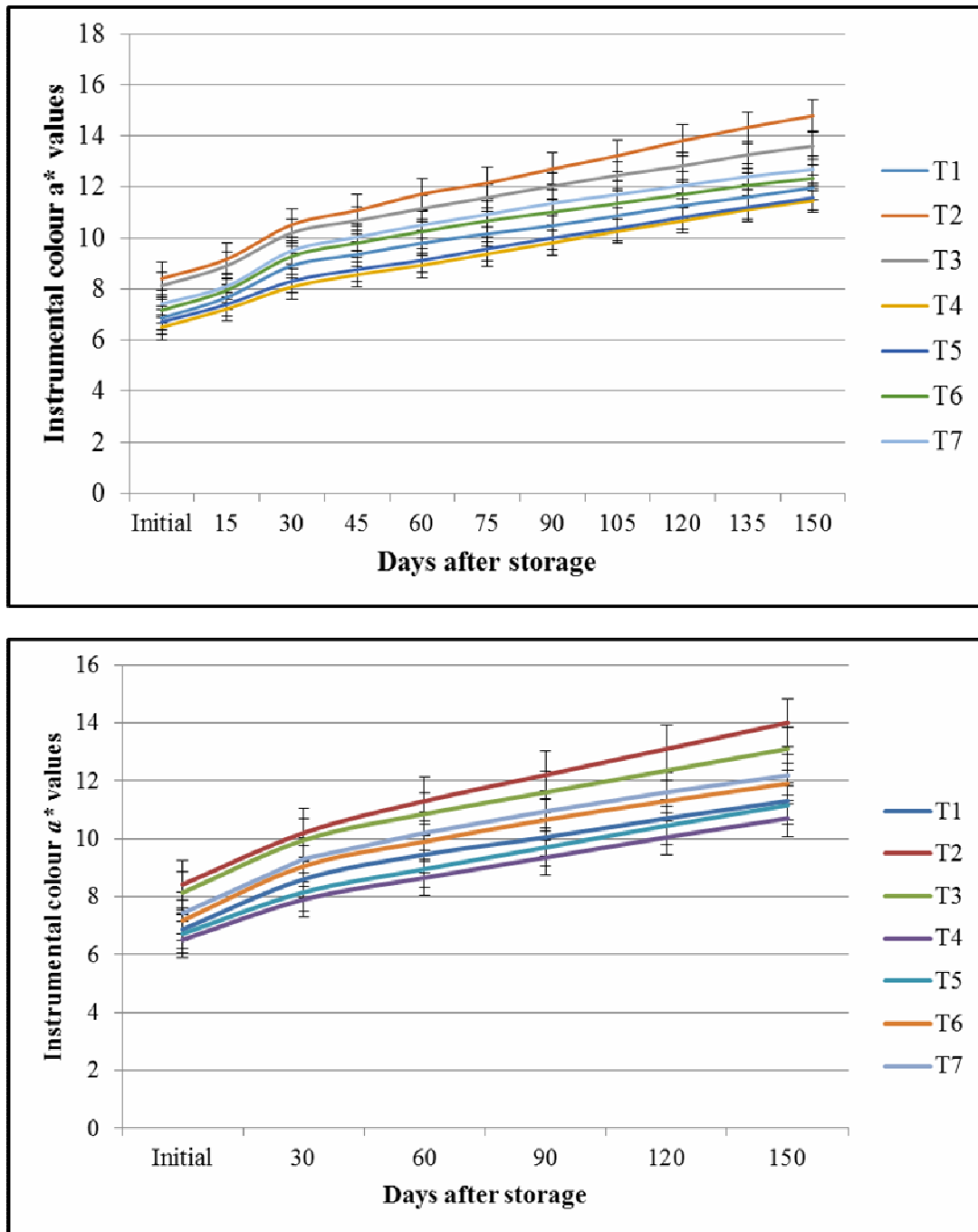


Fig. 4 : Effect of different postharvest treatments on instrumental a^* values of raisins under ambient storage ($29\pm 1^\circ\text{C}$, $43\pm 1\%$ RH) and cold storage ($5\pm 2^\circ\text{C}$, RH- 85-95%)

T₁ :Sulphur dioxide fumigation at 3g/kg

T₂ :Dipping in ozonated water for 3 minutes

T₃ :Ozone fumigation for 10 minutes

T₄ :Ozone fumigation for 15 minutes

T₅ :Ozone fumigation for 20 minutes

T₆ :Ozone fumigation for 25 minutes

T₇ :Ozone fumigation for 30 minutes

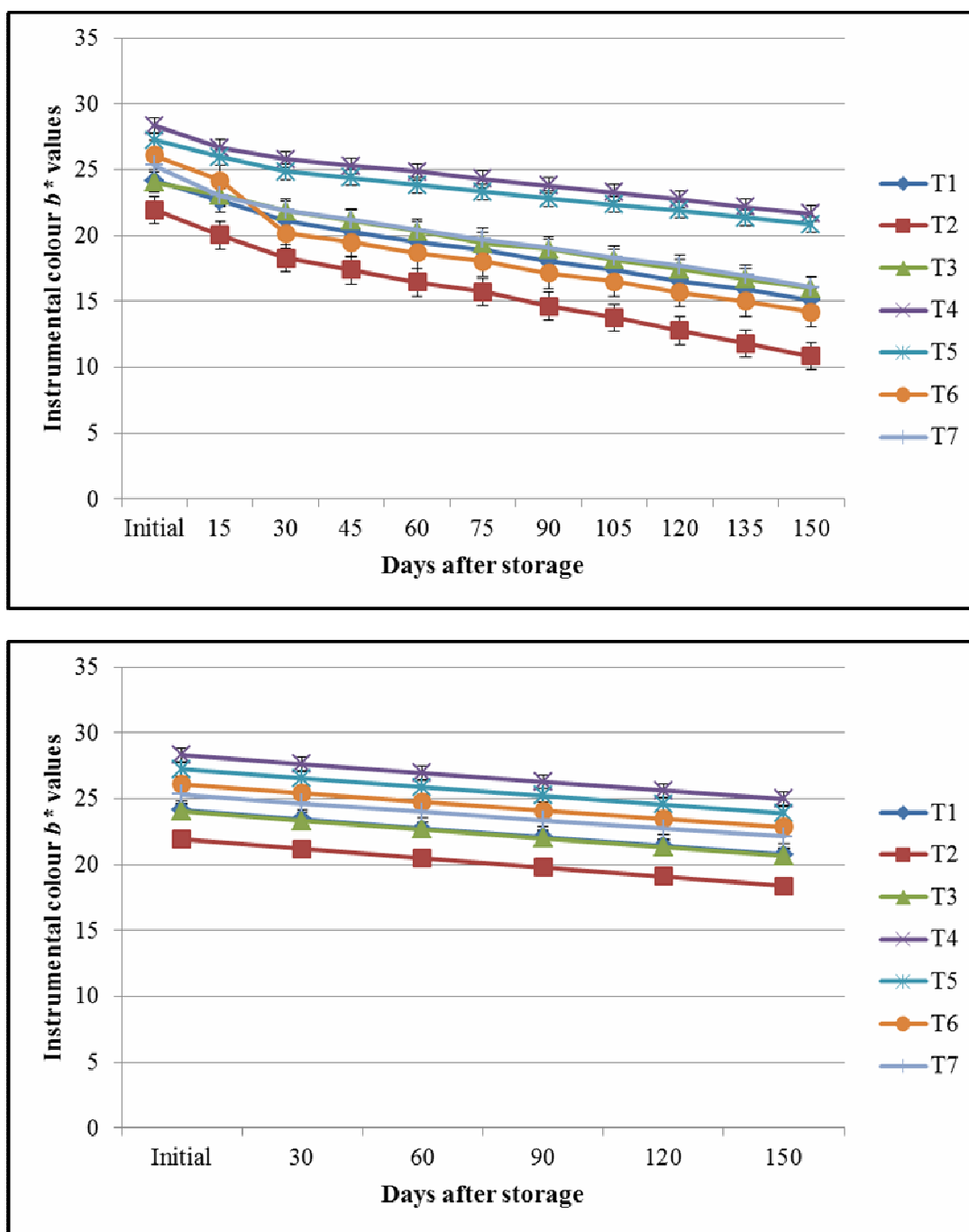


Fig. 5 : Effect of different postharvest treatments on instrumental b^* values of raisins under ambient storage ($29\pm 1^\circ\text{C}$, $43\pm 1\%$ RH) and cold storage ($5\pm 2^\circ\text{C}$, RH- 85-95%)

T₁ :Sulphur dioxide fumigation at 3g/kg

T₂ :Dipping in ozonated water for 3 minutes

T₃ :Ozone fumigation for 10 minutes

T₄ :Ozone fumigation for 15 minutes

T₅ :Ozone fumigation for 20 minutes

T₆ :Ozone fumigation for 25 minutes

T₇ :Ozone fumigation for 30 minutes

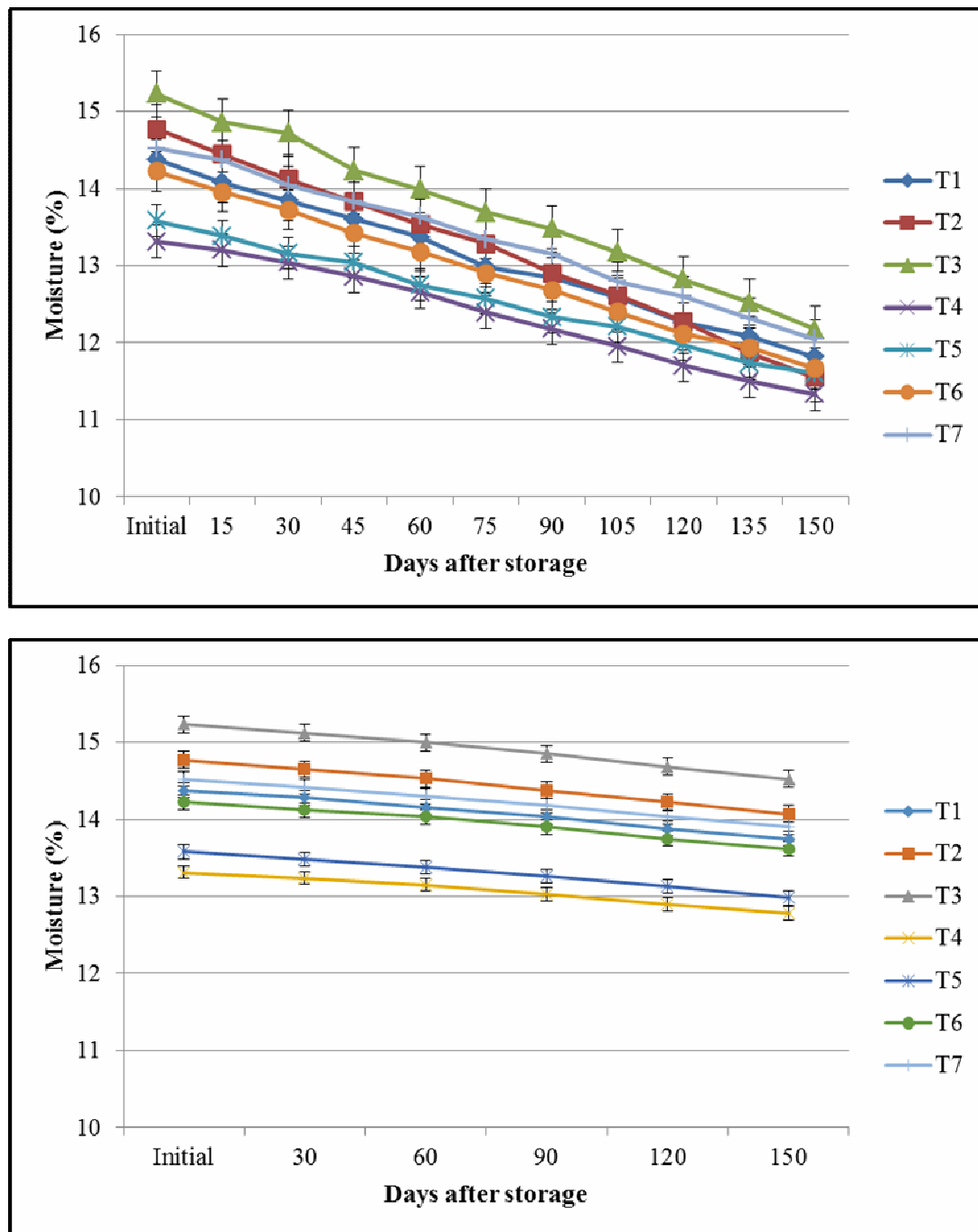


Fig. 6 : Effect of different postharvest treatments on moisture content of raisins under ambient storage (29±1°C, 43±1% RH) and cold storage (5±2°C, RH- 85-95%)

T₁ : Sulphur dioxide fumigation at 3g/kg

T₂ : Dipping in ozonated water for 3 minutes

T₃ : Ozone fumigation for 10 minutes

T₄ : Ozone fumigation for 15 minutes

T₅ : Ozone fumigation for 20 minutes

T₆ : Ozone fumigation for 25 minutes

T₇ : Ozone fumigation for 30 minutes

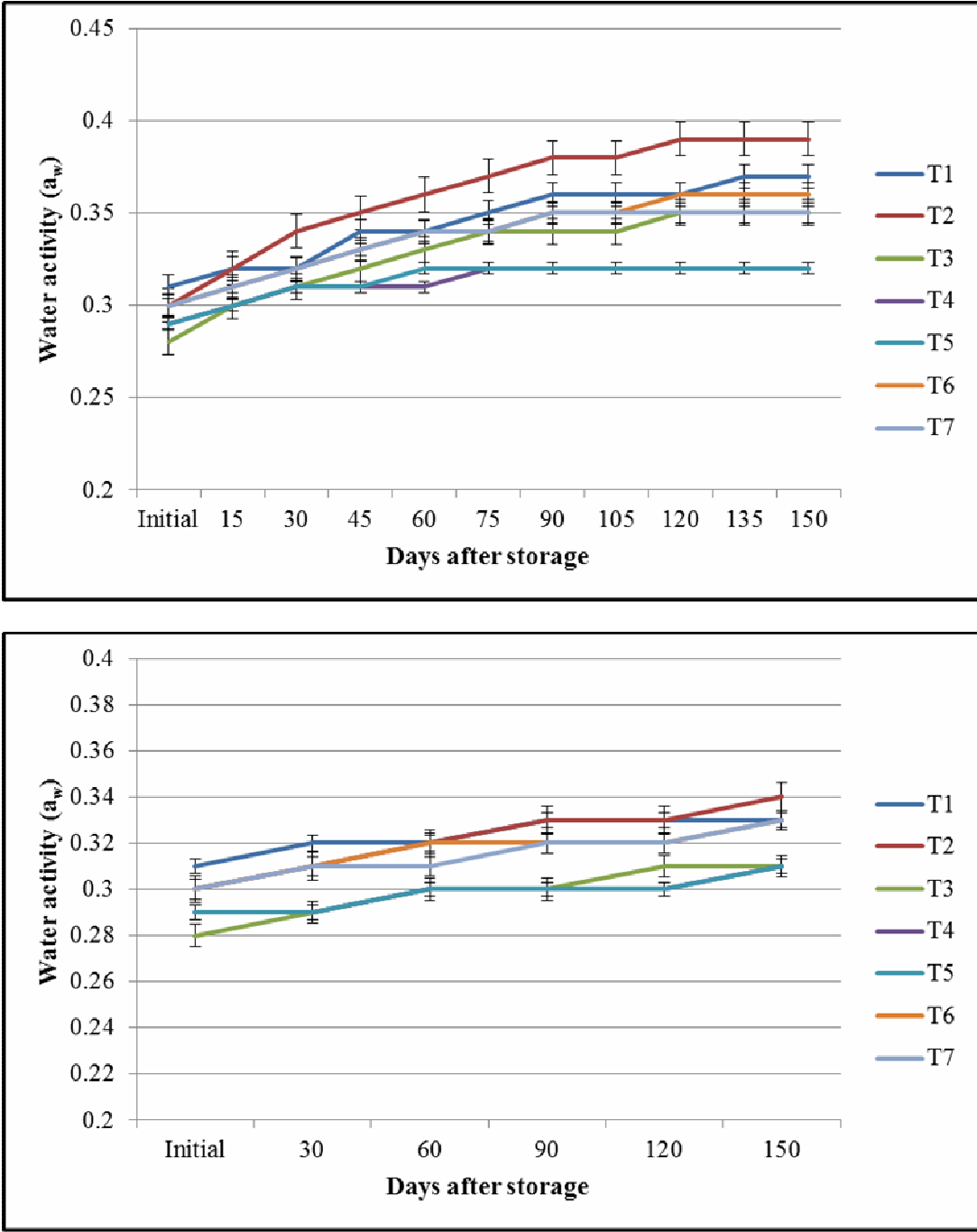


Fig. 7 : Effect of different postharvest treatments on water activity of raisins under ambient storage (29±1°C, 43±1% RH) and cold storage (5±2°C, RH- 85-95%)

Conclusion

Ozone fumigation, particularly at 15 and 20 minutes (T₄ and T₅), offers a safe and highly effective alternative to sulphur fumigation for extending raisin shelf life under both ambient and cold storage conditions. These treatments not only minimized quality deterioration but also ensured consumer acceptability over prolonged storage, thereby presenting themselves as viable and eco-friendly

postharvest management strategies for the raisin industry.

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