International Journal of Food Science +Technology

Original article

The effects of chitosan-TiO_2 and chitosan-TiO_2-ZnO-MgO hybrid coatings on the shelf life of jackfruit bulbs (*Artocarpus heterophyllus* Lam)

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(Received 1 March 2023; Accepted in revised form 20 May 2023)

Summary This study evaluated the effect of chitosan hybrid coatings on the shelf life and quality of jackfruit bulbs. The treatments include control bulbs, bulbs with the chitosan coating (CS bulbs), bulbs with the chitosan-TiO_2 hybrid coating (CSTiO_2 bulbs) and bulbs with the chitosan-TiO_2-ZnO-MgO hybrid coating (CSTZM bulbs). Bulbs were stored at 4 °C. Shelf life of control, CS, CSTiO_2 and CSTZM bulbs were 4, 10, 13 and 15 days, respectively. The CSTZM bulbs exhibited < 1% weight loss, a decrease in the pattern of respiration, the highest levels of vitamin C (28.27 mg/100 g), carotenoids (10868.91 μg/100 g), phenolic compounds (31.31 mg/100 g) and antioxidant capacity (283.49–444.36 mmol/100 g). According to the *Artemia salina* test, all coatings were non-toxic. In conclusion, the chitosan-TiO_2-ZnO-MgO hybrid coating was the best alternative to extend the shelf-life of fresh-cut jackfruit.

Keywords bioactive compounds, Fresh-cut jackfruit, hybrid coatings, nutrients.

Introduction

Production of jackfruit (*Artocarpus heterophyllus* Lam) in the world is 3.7 million tons/year, and in the last 10 years, it increased by 400%. It is a highly perishable climacteric fruit, resulting in significant postharvest loss (30%–40%) (SIAP, 2021). This loss is particularly concerning given the growing demand for jackfruit, both domestically and internationally. Despite the increasing popularity of jackfruit, its short shelf life of <3 days at 25 °C (Anaya-Esparza *et al.*, 2018). Therefore, the extension of shelf life and the preservation of the quality of fresh-cut jackfruit need to be investigated, and the application of coatings and refrigerated storage are suitable alternatives.

Coatings act as a barrier to gases, and microorganisms, decrease physiological changes and extend the shelf-life of different whole or fresh-cut fruits. In jackfruit bulbs, 12 days of shelf-life at 4 °C is the most extended shelf-life reported when treated with edible coatings (Vargas-Torres *et al.*, 2017; Anaya-Esparza *et al.*, 2018). In the last decade, a promising polymer is chitosan; however, due to its high hygroscopicity and poor mechanical properties, it has been improved by obtaining hybrid coatings to broaden its large-scale deployment (Anaya-Esparza *et al.*, 2020; Oladzadabbasabadi *et al.*, 2022).

Hybrid coatings based on chitosan are obtained by combining this polymer with other chemical compounds between them, TiO_2 plus carbohydrates, essential oils and oxide nanoparticles. These combinations are to improve the gas or water barrier, antimicrobial and antioxidant properties, and mechanical, chemical, physical and thermal resistance of hybrid coatings (Oladzadabbasabadi *et al.*, 2022). It has been demonstrated that these hybrid coatings are the best to decrease oxygen permeability. Some generate hydroxyl radicals (−OH) and other reactive oxygen species in UV light, which can react with organic molecules such as ethylene and decompose them into CO_2 and water (Kaewklin *et al.*, 2018). This type of coating has extended the shelf life of fresh-cut melon (Qiao *et al.*, 2019) and whole fruits such as mango (Xing *et al.*, 2020; Wang *et al.*, 2022), apple (Liu *et al.*, 2021),

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doi:10.1111/ijfs.16516

blueberries (Rokayya *et al.*, 2021), litchi (Luo *et al.*, 2022) and strawberry (Yuan *et al.*, 2023).

Our working group developed a new coating using a ternary system with chitosan-titanium dioxide-zinc oxide-magnesium oxide (CSTZM) (Anaya-Esparza *et al.*, 2021, 2022). It was concluded that CSTZM coating exhibited excellent thermal stability, mechanical and barrier properties, low solubility and without toxicity effects on *Artemia salina* (Anaya-Esparza *et al.*, 2022). However, the impact of CSTZM hybrid coating on the quality and shelf life of fresh-cut jackfruit has not been investigated. Therefore, the objective of this study was to evaluate the effect of $CSTiO_2$ and CSTZM hybrid coatings on the shelf life and quality parameters of jackfruit bulbs during storage.

Materials and methods

Materials

Magnesium oxide (MgO), zinc oxide (ZnO) and glacial acetic acid (CH_3COOH), citric acid ($C_6H_8O_7$), calcium chloride ($CaCl_2$), nutrient agars, methanol (CH_3OH (CH_4O)), petroleum ether (C_6H_6) were analytical grades and obtained from Jalmek Scientific SA, Guadalajara, Jalisco, Mexico. Chitosan, dioxide titanium (TiO_2), standards of vitamins, HPLC solvents, carotenoids, 2,2-diphenyl-1-picrylhydrazyl (DPPH), 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) and phenolic compounds were purchased from Sigma Aldrich (St. Louis, MO, USA).

Coating preparation

All coatings were prepared according to Anaya-Esparza *et al.* (2022). Briefly, chitosan coating (CS coating) was prepared using 1 g of chitosan dissolved in 100 mL of 0.6% (v/v) glacial acetic acid solution under magnetic stirring. Chitosan-TiO_2 ($CSTiO_2$) hybrid coating was synthesised with the chitosan solution and TiO_2 nanoparticles (500 μg). Chitosan-TiO_2-ZnO-MgO (CSTZM) hybrid coating was developed with the chitosan solution and TiO_2-ZnO-MgO nanoparticles (500 μg) containing 90% of TiO_2, 5% of ZnO and 5% of MgO. All coatings were sterilised at 121 °C for 15 min and 1.5 atm pressure and stored at 4 °C until their use.

Jackfruit bulbs

Ripened jackfruits were harvested in San Blas, Nayarit, Mexico and were cut manually. Jackfruit bulbs were then immersed in a cold solution (4 °C) of 1% (w/v) citric acid and 0.1% (w/v) calcium chloride for 1 min (Vargas-Torres *et al.*, 2017) and maintained at 4 °C until the coating application.

Coating application

The bulbs were divided into four batches: (i) uncoated control bulbs (Control bulbs), (ii) coated with CS coating (CS bulbs), (iii) coated with the $CSTiO_2$ hybrid coating ($CSTiO_2$ bulbs) and (iv) coated with the CSTMZ hybrid coating (CSTMZ bulbs). The coatings were applied by immersing the bulbs for 1 min at 4 °C. The bulbs were drained at 4 °C, packed in disinfected polypropylene containers, and the container was covered with perforations (2 mm diameter) and stored at 4 °C (Qiao *et al.*, 2019).

Microbiological analysis

Decimal dilutions were made to evaluate microorganisms (Total coliforms, aerobic mesophilic, psychrophilic and lactic bacteria, moulds and yeast) using pour-plate inoculation and FDA methods (FDA, 2021). All the results were expressed as CFU/g.

Physiological analysis

Weight loss was obtained by weighing 10 bulbs of each treatment daily using an analytical balance (Velab, Model VE-204B, Pharr, Texas, USA) and was reported as a percentage. The respiration rate (RR) was performed by gas chromatography (HP model 6890, Hewlett Packard, Palo Alto, CA, USA, fitted with thermal conductivity detector) (Vargas-Torres *et al.*, 2017). Results were expressed as mL CO_2/kg·h.

Physicochemical parameters

The firmness of the bulbs (without seed) was obtained by using a texturometer (TA XT plus, London, England). The results were expressed in Newton (N). Total soluble solids (TSS) were evaluated with a refractometer (Abbe 315RS, Royal Tunbridge Wells, UK), and results were expressed as °Brix. Titratable acidity (TA) was measured using an automatic titrator (SCHOTT Instruments; Berlin, Germany), and the results were reported in percentage of citric acid. The pH values were measured in homogenised pulp with a pH meter (HANNA Instruments Ltd, HI 221, Bedford, UK). The colour was measured as Hue angle (°Hue) on the surface of the samples using a colorimeter (Colorimeter NH300, Shanghai, China). All measurements of physicochemical parameters were made following the AOAC methods (2005).

Nutritional parameters

Nutritional parameters (protein, moisture, fat and ash) were evaluated using official AOAC methods (AOAC, 2005). Soluble carbohydrates were quantified by the Anthrone method (Loewus, 1952). Soluble dietary

fibre (SDF), insoluble dietary fibre (IDF) and total dietary fibre (TDF) were analysed according to Mañas & Saura-Calixto (1995). The data were reported as g/100 g FW. Vitamin C and vitamin E were quantified according to Barbosa-Gámez *et al.* (2017) using an HPLC-DAD (Agilent Technologies, Waldbronn, Germany). The results were expressed as mg/100 g FW.

Phenolic compounds

Total soluble phenols (TSP) were extracted with an organic aqueous extraction (Pérez-Jiménez *et al.*, 2008). The supernatants were used to measure the TSP (Montreau, 1972) and the residues to determine the hydrolysable polyphenols (Hartzfeld *et al.*, 2002) and condensed tannins (Reed *et al.*, 1982). The results were reported in mg/100 g FW. The identification of phenolic compounds was determined in the extracts from TSP by HPLC-DAD (Agilent Technologies 1260 Infinity, Waldbronn, Germany) (Nolasco-González *et al.*, 2022). The results were expressed in μg/100 g FW.

Carotenoids

The content of total carotenoids was determined using the method reported by Philip & Chen (1988) and registered as μg/100 g FW. A partial profile of carotenoids was performed in an HPLC-DAD (Agilent Technologies 1260 Infinity, Wald Bronn, Germany) (Meléndez-Martínez *et al.*, 2009). The results were expressed as μg/100 g FW.

Antioxidant capacity

The antioxidant capacity (AOX) of the TSP extracts and carotenoid extracts was determined by the (DPPH) radical assay (Prior *et al.*, 2005) and the ferric ion reduction method (FRAP) (Benzie & Strain, 1996). AOX was reported in mmol Trolox equivalents/100 g FW.

Toxicity evaluation with the *Artemia salina* test

Ten adult organisms (3 weeks of growth, 2 cm in length) were used with 500 μL of a solution of lyophilized jackfruit pulp (100, 500, 600, 800, 1000 or 1500 μg/mL) from control and coatings bulbs. The number of live and dead organisms was counted after 24 h of exposition. Organisms were considered dead when they did not show internal or external movements during 1 min of observation, and the percentage of survival rate was determined (Anaya-Esparza *et al.*, 2019).

Statistical analysis

The results were expressed as the mean ± standard deviation in triplicate. Data were analysed with a one-way analysis of variance (ANOVA) ($P < 0.05$) and Fisher-LSD means test ($\alpha = 0.05$). The Statistica v.12 software (Statsoft, Tulsa, Oklahoma, USA) was used.

Results and discussion

Microbiological analysis

It was evident that control bulbs exhibited the highest microbial growth from the beginning of storage until final storage (Table S1). Also, these bulbs exceed the permissible limits (FDA, 2021). In contrast, all the coated bulbs remained within the stipulated standards till the end of storage while the lowest microbial counts were found in $CSTiO_2$ and CSTZM bulbs. The antimicrobial activity of CS is attributed to the interaction of their amino groups with the bacterial cell wall causing damage (Mohandas *et al.*, 2017). However, adding TiO_2 into the CS matrix enhances antimicrobial activity due to the generation of photoreactive radicals, causing cell death (membrane deformation, cell disruption, and cell wall structural changes) by oxidative stress (Sami *et al.*, 2020). The CSTZM hybrid coating was an antimicrobial more effective because the doping of TiO_2 with ZnO and MgO reduced the electron pair–hole recombination, which increased the catalytic activity of the ternary system, in addition to increasing the contact area and reactivity of the CSTZM system on microorganisms by electrostatic attraction causing irreparable cell disruption (Raut *et al.*, 2016). The shelf life of the bulbs was dependent on the type of treatment applied, being 5, 10, 13 and 15 days at 4 °C for CSTZM, $CSTiO_2$, CS and control bulbs, respectively.

Physiological parameters

The control bulbs exhibited the most significant weight loss (1.5%) at 5 days, while CSTZM bulbs presented the lowest weight loss (0.86%) after 15 days of storage (Fig. 1a). Anaya-Esparza *et al.* (2022) reported that CSTZM hybrid coating exhibited lower water vapour permeability than CS coating and $CSTiO_2$ hybrid coatings, which was attributed to the presence of the mixed oxides bound to the CS matrix forming a dense structure that slowed the passage of water molecules through the coating, which delayed the dehydration of the jackfruit bulbs.

In control bulbs, an accelerated decrease in RR was observed, suggesting the deterioration by microorganisms (Fig. 1b). Conversely, a slowest decreased pattern of RR was shown in CSTZM bulbs. The CS coating reduces water vapour exchange, O_2 and CO_2 permeabilities (USDA, 2016). However, the CS coating is unstable during refrigerated storage due to changes in the acetyl groups and deprotonation of the amino

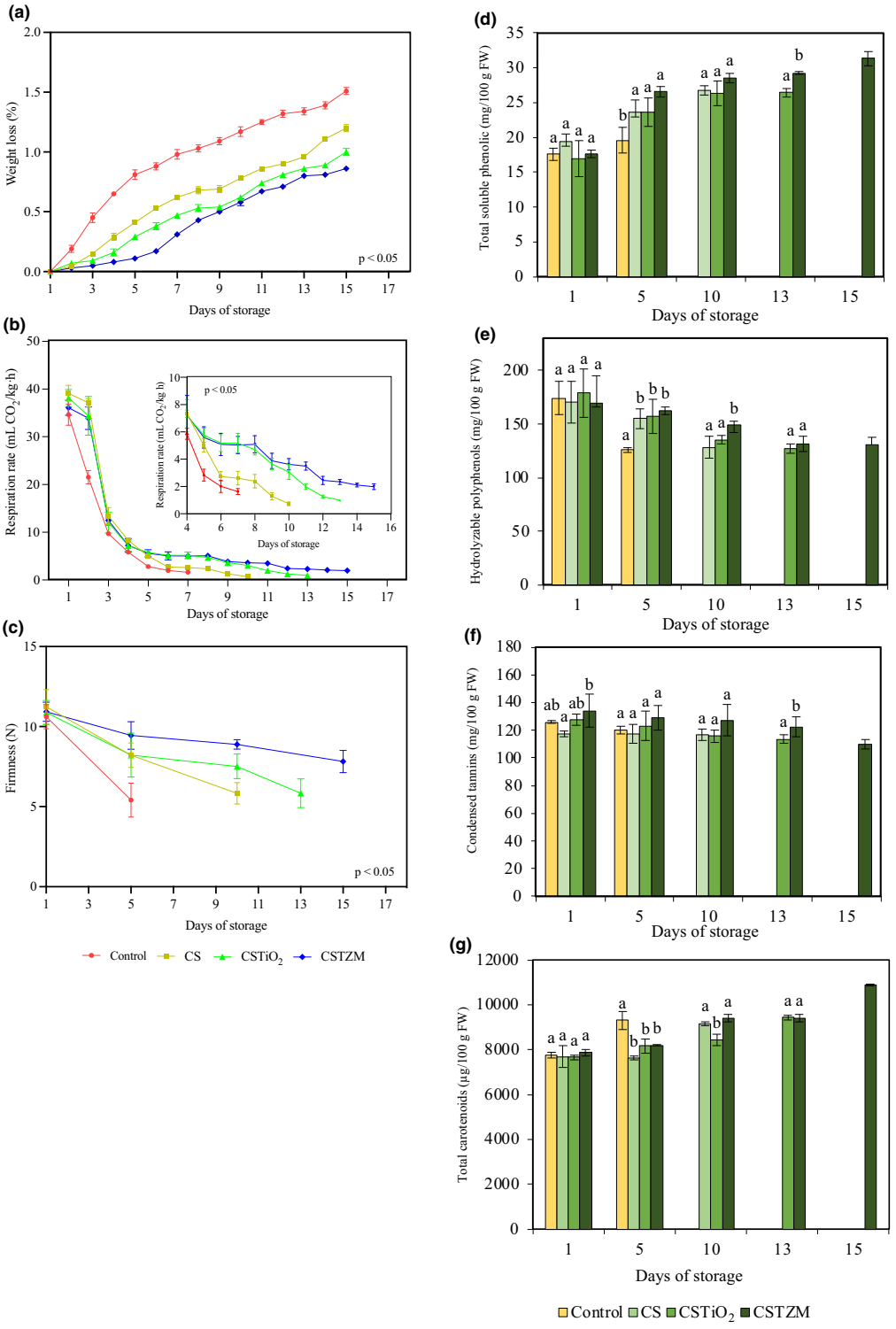

Figure 1 Weight loss (a), respiration rate (b) and firmness (c), soluble phenols (d), hydrolyzable phenols (e), condensed tannins (f) and total carotenoids (g) of uncoated jackfruit bulbs (control bulbs), bulbs coated with chitosan (CS), with chitosan-TiO2 hybrid (CSTiO2) and with Chitosan-TiO2-ZnO-MgO hybrid (CSTZM), during storage at 4 °C. Lowercase letters indicate significant statistical differences ($P < 0.05$) between treatments.

group by the evaporation of water (Anaya-Esparza *et al.*, 2021). In the $CSTiO_2$ hybrid coating, the TiO_2 acts as an anti-plasticizing agent absorbing water molecules from the environment or the coating itself. However, the coating becomes brittle for long storage at low relative humidity (Anaya-Esparza *et al.*, 2021). The CSTZM hybrid coating is more resistant due to the modification of the interfacial regions of the polymeric matrix of chitosan with the interaction of TiO_2, ZnO and MgO (crosslinking effect), carboxyl groups ($-COOH$), and amine groups ($-NH_2$), limiting the exchange of CO_2, O_2 and water vapour and providing the most excellent stability to this coating during storage (Anaya-Esparza *et al.*, 2021, 2022).

Physicochemical parameters

Control bulbs had a 49% loss of firmness at 5 days, CS bulbs had a 48% loss at 10 days, $CSTiO_2$ bulbs had a 46% loss at 13 days and CSTZM bulbs had a 28% loss at 15 days (Fig. 1c). TSS and TA slightly increased, while pH decreased during storage time (Table S2). The colour of the bulbs changed significantly ($P < 0.05$) from yellow-orange colour (55.62–57.34 °Hue) to intense orange colour when storage ended. The behaviour of TSS, TA, pH and colour is characteristic of the 'Agüitada' genotype during its ripening (Morelos-Flores *et al.*, 2021). However, the significant effect ($P < 0.05$) of the coatings was observed by the delay in the changes of physicochemical parameters of the coated bulbs compared to the control bulbs. It has been reported that the CS hybrid coatings delayed the changes in TSS, TA, pH and colour in fresh-cut apples and melons (Qiao *et al.*, 2019). The CSTZM hybrid coating delayed the physicochemical changes in jackfruit bulbs during a more extended period by its lowest O_2 permeability (Anaya-Esparza *et al.*, 2022).

Nutritional parameters

Statistically, there was a significant effect ($P > 0.05$) due to storage time and coatings on nutritional parameters (Table 1). The highest moisture loss was observed in the control bulbs (1.54%), followed by CS bulbs (1.36%), $CSTiO_2$ bulbs (1.08%), and finally in CSTZM bulbs (0.91%), coinciding with the physiological weight loss. The soluble carbohydrates and fat increased

Table 1 The nutritional parameters (g/100 g FW) of control bulbs, CS bulbs, $CSTiO_2$ bulbs and CSTZM bulbs were measured at the beginning and end of storage at 4 °C

Days of storage	Control (5 days)	CS (10 days)	$CSTiO_2$ (13 days)	CSTZM (15 days)
Moisture				
Initial	$76.24 \pm 0.80^{a,X}$	$77.03 \pm 0.04^{b,X}$	$75.44 \pm 0.16^{c,X}$	$76.26 \pm 0.18^{ab,X}$
Final	$75.06 \pm 0.77^{a,Y}$	$75.98 \pm 0.43^{a,Y}$	$74.62 \pm 0.27^{b,Y}$	$75.56 \pm 0.10^{ab,Y}$
Soluble carbohydrates				
Initial	$21.40 \pm 0.56^{a,X}$	$20.80 \pm 0.05^{a,X}$	$21.26 \pm 0.01^{a,X}$	$21.31 \pm 0.25^{a,X}$
Final	$23.46 \pm 0.46^{a,Y}$	$22.49 \pm 0.38^{a,Y}$	$23.50 \pm 0.11^{a,Y}$	$23.10 \pm 0.16^{a,Y}$
Total protein				
Initial	$1.31 \pm 0.32^{a,X}$	$1.40 \pm 0.14^{a,X}$	$1.33 \pm 0.10^{a,X}$	$1.40 \pm 0.08^{a,X}$
Final	$1.41 \pm 0.20^{a,X}$	$1.51 \pm 0.04^{a,X}$	$1.52 \pm 0.07^{a,X}$	$1.47 \pm 0.03^{a,X}$
Total ashes				
Initial	$0.66 \pm 0.01^{a,X}$	$0.66 \pm 0.01^{a,X}$	$0.72 \pm 0.02^{b,X}$	$0.78 \pm 0.04^{c,X}$
Final	$0.63 \pm 0.01^{a,Y}$	$0.63 \pm 0.02^{a,X}$	$0.70 \pm 0.02^{a,X}$	$0.73 \pm 0.09^{a,X}$
Total fat				
Initial	$0.13 \pm 0.01^{a,X}$	$0.14 \pm 0.01^{a,X}$	$0.14 \pm 0.01^{a,X}$	$0.15 \pm 0.03^{a,X}$
Final	$0.16 \pm 0.02^{a,Y}$	$0.29 \pm 0.01^{a,Y}$	$0.29 \pm 0.04^{ab,Y}$	$0.39 \pm 0.02^{c,Y}$
Total dietary fibre				
Initial	$3.08 \pm 0.06^{a,X}$	$3.07 \pm 0.09^{a,X}$	$3.04 \pm 0.04^{a,X}$	$3.24 \pm 0.04^{b,X}$
Final	$2.54 \pm 0.07^{a,Y}$	$2.75 \pm 0.03^{b,Y}$	$2.62 \pm 0.05^{c,Y}$	$2.91 \pm 0.06^{d,Y}$
Vitamin E (mg/100 g FW)				
Initial	$1.62 \pm 0.04^{a,X}$	$1.59 \pm 0.05^{a,X}$	$1.68 \pm 0.05^{a,X}$	$1.59 \pm 0.02^{a,X}$
Final	$1.51 \pm 0.01^{a,Y}$	$2.03 \pm 0.01^{b,Y}$	$2.06 \pm 0.03^{b,Y}$	$2.04 \pm 0.02^{b,Y}$
Vitamin C (mg/100 g FW)				
Initial	$27.84 \pm 0.14^{a,X}$	$27.26 \pm 1.53^{a,X}$	$28.13 \pm 1.12^{a,X}$	$28.53 \pm 1.37^{a,X}$
Final	$21.87 \pm 0.30^{b,Y}$	$25.26 \pm 0.22^{b,X}$	$27.63 \pm 0.05^{a,X}$	$28.27 \pm 1.32^{a,X}$

Values are mean ± standard deviation ($n = 4$). Lowercase letters indicate significant statistical differences ($P < 0.05$) between treatments on the same day of storage. Capital letters indicate statistical differences ($P < 0.05$) between the initial and final days of storage between treatments.

($P < 0.05$), while protein and ashes protein remained unchanged ($P > 0.05$) on the last day of storage. It is attributed to the main changes in minimally processed fruits are in sugars, firmness and colour (Anaya-Esparza *et al.*, 2018). $CSTiO_2$ and CSTZM bulbs presented the highest content ($P < 0.05$) of ashes by the presence of TiO_2, MgO and ZnO in the coatings. The initial values (3.08–3.24 g/100 g FW) of total dietary fibre decreased (2.54–2.91 mg/100 g FW) during storage, which coincided with the loss of firmness, where CSTZM bulbs conserved the highest total dietary fibre. Previous studies suggest that chitosan hybrid coatings modify the internal atmosphere of fruits, which decreases the degradation process of pectin and cellulases (Kumarihami *et al.*, 2021).

The vitamin E increased at the end of storage (2.03–2.06 mg/100 g FW) without significant differences between coatings ($P > 0.05$), except for the control

Table 2 Phenolic compound profile of uncoated jackfruit bulbs (control), CS bulbs, $CSTiO_2$ bulbs and CSTZM bulbs at 4 °C storage (initial and final)

N°	Phenolic compounds	Day of storage	Treatment (μg/100 g FW)			
			Control (5 days)	CS (10 days)	$CSTiO_2$ (13 days)	CSTZM (15 days)
1	Shikimic acid	Initial	$165.84 \pm 0.02^{a,X}$	$182596.75 \pm 18.97^{b,X}$	$212378.74 \pm 36.20^{c,X}$	$270557.61 \pm 31.26^{d,X}$
		Final	$155696.36 \pm 26.28^{a,Y}$	$163309.33 \pm 38.28^{b,Y}$	$187511.79 \pm 18.98^{c,Y}$	$246601.64 \pm 5.21^{d,Y}$
Hydroxybenzoic acids						
2	Gallic	Initial	$219.13 \pm 9.91^{a,X}$	$201.76 \pm 1.57^{b,X}$	$142.15 \pm 5.21^{c,X}$	$170.30 \pm 1.05^{d,X}$
		Final	$369.79 \pm 8.20^{b,Y}$	$236.99 \pm 8.86^{a,Y}$	$230.45 \pm 18.29^{a,Y}$	$230.01 \pm 4.50^{a,Y}$
3	Protocatechuic	Initial	$31.64 \pm 0.38^{a,X}$	$30.01 \pm 1.48^{a,X}$	$30.01 \pm 1.08^{a,X}$	$45.37 \pm 0.35^{b,X}$
		Final	$47.46 \pm 1.15^{a,Y}$	$34.18 \pm 0.63^{b,Y}$	$68.40 \pm 2.94^{c,Y}$	$79.13 \pm 0.93^{d,Y}$
4	2-hydroxybenzoic	Initial	$21.41 \pm 0.11^{ab,X}$	$21.34 \pm 0.06^{ab,X}$	$21.25 \pm 0.42^{a,X}$	$21.86 \pm 0.01^{b,X}$
		Final	$18.71 \pm 0.51^{a,Y}$	$15.27 \pm 0.08^{b,Y}$	$19.69 \pm 0.07^{c,Y}$	$20.70 \pm 0.24^{d,Y}$
5	4-hydroxybenzoic	Initial	$113.63 \pm 21.11^{b,X}$	$153.81 \pm 10.75^{c,X}$	$196.57 \pm 1.23^{a,X}$	203.58 ± 0.19^{axe}
		Final	$26.86 \pm 1.16^{a,Y}$	$44.96 \pm 6.50^{b,Y}$	$64.26 \pm 0.39^{c,Y}$	$102.21 \pm 10.42^{d,Y}$
6	2,5-dihydroxybenzoic	Initial	$33.49 \pm 0.38^{a,X}$	$34.31 \pm 7.49^{a,X}$	$68.49 \pm 9.51^{b,X}$	$83.99 \pm 2.46^{b,X}$
		Final	$36.02 \pm 4.63^{a,X}$	$32.02 \pm 2.96^{a,X}$	$86.89 \pm 2.95^{b,X}$	$85.84 \pm 5.54^{b,X}$
7	3,4-Dihydroxyphenylacetic	Initial	$531.67 \pm 7.77^{b,X}$	$599.29 \pm 1.09^{a,X}$	$588.25 \pm 3.18^{a,X}$	$653.05 \pm 13.69^{c,X}$
		Final	$675.57 \pm 0.24^{b,Y}$	$634.33 \pm 8.50^{a,Y}$	$619.82 \pm 14.46^{a,X}$	$671.39 \pm 3.38^{b,X}$
8	4-Hydroxy-3-methoxyphenylacetic	Initial	$16.24 \pm 1.16^{a,X}$	$20.20 \pm 0.32^{b,X}$	$35.08 \pm 0.53^{c,X}$	$15.69 \pm 0.96^{a,X}$
		Final	$32.86 \pm 0.37^{a,Y}$	$22.00 \pm 0.88^{b,X}$	$25.14 \pm 1.64^{c,Y}$	$29.15 \pm 0.23^{d,Y}$
9	4-hydroxybenzaldehyde	Initial	$9.18 \pm 0.01^{a,X}$	$9.14 \pm 0.02^{a,X}$	$9.60 \pm 0.06^{b,X}$	$9.81 \pm 0.05^{c,X}$
		Final	$9.97 \pm 0.05^{a,Y}$	$9.86 \pm 0.04^{b,Y}$	$10.98 \pm 0.03^{c,Y}$	$10.39 \pm 0.01^{d,Y}$
Hydroxycinnamic acids						
10	Trans-cinnamic	Initial	$30.52 \pm 0.06^{a,X}$	$30.74 \pm 0.17^{a,X}$	$32.25 \pm 0.10^{b,X}$	$32.66 \pm 0.32^{b,X}$
		Final	$33.54 \pm 0.15^{ab,Y}$	$30.43 \pm 0.01^{c,X}$	$34.34 \pm 0.44^{b,Y}$	$32.75 \pm 0.40^{a,X}$
11	*p*-coumaric	Initial	$86.27 \pm 0.18^{c,X}$	$83.39 \pm 0.12^{b,X}$	$92.62 \pm 0.15^{a,X}$	$92.48 \pm 0.09^{a,X}$
		Final	$95.12 \pm 0.15^{b,Y}$	$91.74 \pm 0.04^{a,Y}$	$97.69 \pm 1.82^{c,X}$	$92.63 \pm 0.13^{ab,X}$
12	Caffeic	Initial	$18.85 \pm 0.14^{a,X}$	$20.11 \pm 0.34^{b,X}$	$25.22 \pm 0.06^{c,X}$	$21.77 \pm 0.25^{d,X}$
		Final	$20.44 \pm 0.25^{b,Y}$	$17.80 \pm 0.05^{a,Y}$	$21.40 \pm 0.37^{c,Y}$	$18.07 \pm 0.01^{a,Y}$
13	Chlorogenic	Initial	$270.10 \pm 0.71^{a,X}$	$264.30 \pm 1.55^{a,X}$	$188.03 \pm 7.01^{c,X}$	$123.52 \pm 3.06^{b,X}$
		Final	$71.70 \pm 1.35^{a,Y}$	$55.91 \pm 0.62^{b,Y}$	$97.25 \pm 0.05^{c,Y}$	$84.25 \pm 0.52^{d,Y}$
14	Trans-ferulic	Initial	$24.04 \pm 0.37^{b,X}$	$26.13 \pm 0.28^{a,X}$	$26.61 \pm 0.16^{a,X}$	$26.50 \pm 0.26^{a,X}$
		Final	$31.98 \pm 0.40^{c,Y}$	$29.15 \pm 0.01^{b,Y}$	$39.51 \pm 0.93^{a,Y}$	$40.21 \pm 0.64^{a,Y}$
Flavonoids						
15	Myricetin	Initial	$14.31 \pm 0.03^{a,X}$	$14.58 \pm 0.39^{a,X}$	$14.71 \pm 0.21^{a,X}$	$13.43 \pm 0.23^{b,X}$
		Final	$14.66 \pm 0.16^{a,X}$	$14.89 \pm 0.08^{ab,X}$	$15.75 \pm 0.29^{c,X}$	$15.15 \pm 0.01^{b,Y}$
16	Catechin	Initial	$1406.10 \pm 19.66^{a,X}$	$1962.68 \pm 11.01^{b,X}$	$1734.50 \pm 10.27^{c,X}$	$2702.93 \pm 27.84^{d,X}$
		Final	$2048.91 \pm 2.05^{b,Y}$	$2376.71 \pm 0.01^{a,Y}$	$2394.66 \pm 11.09^{a,Y}$	$2691.03 \pm 31.80^{c,X}$
17	Naringenin	Initial	$12.78 \pm 0.08^{c,X}$	$12.45 \pm 0.15^{b,X}$	$13.58 \pm 0.13^{a,X}$	$13.48 \pm 0.04^{a,X}$
		Final	$13.89 \pm 0.25^{a,Y}$	$13.93 \pm 0.02^{a,Y}$	$15.09 \pm 0.60^{b,X}$	$15.12 \pm 0.01^{b,Y}$
Total content of phenolic compounds			159.24	166.97	191.35	250.82

Values are mean ± standard deviation ($n = 4$). Lowercase letters indicate significant statistical differences ($P < 0.05$) between treatments on the same day of storage. Capital letters indicate statistical differences ($P < 0.05$) between the initial and final days of storage between treatments.

bulbs. The control bulbs exhibited a 21% loss of vitamin C, whereas in $CSTiO_2$ bulbs, the loss was 7%. $CSTiO_2$ and CSTZM bulbs maintained the vitamin C content until the end of storage. The loss of vitamin C in the control bulbs is due to oxidation during the ripening process (Morelos-Flores *et al.*, 2021). Nonetheless, the vitamin C preservation by $CSTiO_2$ and CSTMZ bulbs might be related to their higher ability to decrease O_2 permeability and, consequently, the reduction of vitamin C oxidation (Rokayya *et al.*, 2021). The jackfruit bulbs coated with the CSTZM hybrid coating preserved macro and micronutrients for the longest time.

Phenolic compounds

Fig. 1d demonstrates that TSP increased during storage, and CSTZM bulbs exhibited the highest values (31.31 mg/100 g FW) after 15 days. This result is explained because chitosan in coatings induced the defence system in fruits through the synthesis of phenolic compounds; also, CSTZM hybrid coating protected bulbs from oxidation better than other coatings (Xing *et al.*, 2020; Liu *et al.*, 2021). The hydrolysable polyphenols (Fig. 1e) and condensed tannins (Fig. 1f) decreased during storage in all treatments. The decrease of these polyphenols was most rapid in control bulbs. Hydrolysable polyphenols and tannins are non-extractable phenolic compounds bound to the cell wall of polysaccharides (Liu *et al.*, 2020). Therefore, their decrease in bulbs during storage is related to the loss of firmness by enzymatic degradation of the cell wall; the non-extractable polyphenols could be released and oxidised (Liu *et al.*, 2019).

Fourteen phenolic acids and three flavonoids were identified (Table 2). The content of shikimic acid decreased in all treatments because this compound is the primary precursor for synthesising other phenolic acids through the shikimate pathway (Cai *et al.*, 2023). Hydroxycinnamic acids, flavonoids and most hydroxybenzoic acids increased on the last day of storage, except for 2-hydroxybenzoic and 4-hydroxybenzoic acids. The decrease in these hydroxybenzoic acids is attributed to the fact that they are the first phenolic compounds used as antimicrobials, initiating the defence response to the attack of microorganisms (Dey *et al.*, 2005). CSTZM bulbs exhibited higher content of phenolic compounds ($P < 0.05$), coinciding with TSP.

Carotenoids

Carotenoid levels increased rapidly in the control samples (Fig. 1g) due to the absence of a barrier accelerating their ripening (Anaya-Esparza *et al.*, 2018). The CSTZM hybrid coating delayed the carotenoid synthesis due to its gas barrier effect (Xing *et al.*, 2020). However, all coated samples had similar content as the control bulbs at the end of storage, except for CSTZM bulbs, which had the highest carotenoid content attributed to the protective effect of this coating on these pigments (Xing *et al.*, 2020). The content of violaxanthin, lutein, β-cryptoxanthin, α-carotene and β-carotene decreased in the control bulbs at the end of storage, except α-carotene and β-carotene (Table 3). In contrast, except for violaxanthin, the carotenoids increased in coated bulbs at the end of storage. Once again, it was demonstrated that coatings preserved the

Table 3 Carotenoid profile from uncoated jackfruit bulbs (control bulbs), CS bulbs, $CSTiO_2$ bulbs and CSTZM bulbs at initial and final storage at 4 °C

		Treatment (μg/100 g FW)			
Carotenoids	**Day of storage**	**Control (5 days)**	**CS (10 days)**	**$CSTiO_2$ (13 days)**	**CSTZM (15 days)**
Violaxanthin	Initial	$371.84 \pm 5.27^{a,X}$	$334.61 \pm 4.74^{b,X}$	$323.24 \pm 0.71^{c,X}$	$392.08 \pm 0.69^{d,X}$
	Final	$355.99 \pm 3.47^{c,Y}$	$313.25 \pm 3.87^{a,Y}$	$310.08 \pm 1.44^{a,Y}$	$327.56 \pm 0.01^{b,Y}$
Lutein	Initial	$11.87 \pm 0.14^{c,X}$	$6.70 \pm 0.06^{b,X}$	$6.74 \pm 0.01^{b,X}$	$6.94 \pm 0.06^{a,X}$
	Final	$2.44 \pm 0.27^{b,Y}$	$7.75 \pm 0.25^{a,Y}$	$7.65 \pm 0.59^{a,X}$	$7.57 \pm 0.31^{a,Y}$
β-cryptoxanthin	Initial	$47.58 \pm 0.19^{a,X}$	$36.7 \pm 0.01^{b,X}$	$48.39 \pm 0.25^{c,X}$	$43.19 \pm 0.18^{d,X}$
	Final	$43.10 \pm 0.07^{a,Y}$	$48.95 \pm 0.14^{b,Y}$	$56.90 \pm 0.39^{c,Y}$	$52.30 \pm 0.19^{d,Y}$
α-carotene	Initial	$67.72 \pm 0.60^{a,X}$	$60.26 \pm 0.95^{b,X}$	$65.28 \pm 0.36^{c,X}$	$73.81 \pm 0.26^{d,X}$
	Final	$81.94 \pm 0.30^{c,Y}$	$73.14 \pm 0.24^{b,Y}$	$98.41 \pm 0.16^{a,Y}$	$97.61 \pm 2.04^{a,Y}$
β-carotene	Initial	$1632.88 \pm 1.84^{a,X}$	$1644.81 \pm 0.58^{b,X}$	$1762.99 \pm 9.86^{c,X}$	$1793.99 \pm 1.88^{d,X}$
	Final	$1875.11 \pm 3.39^{a,Y}$	$1945.03 \pm 0.7^{b,Y}$	$1988.34 \pm 2.28^{c,Y}$	$1995.76 \pm 0.28^{d,Y}$

Values are mean ± standard deviation ($n = 4$). Lowercase letters (a–d) indicate significant statistical differences ($P < 0.05$) between treatments on the same day of storage. Capital letters (X, Y) indicate statistical differences ($P < 0.05$) between the initial and final days of storage between treatments.

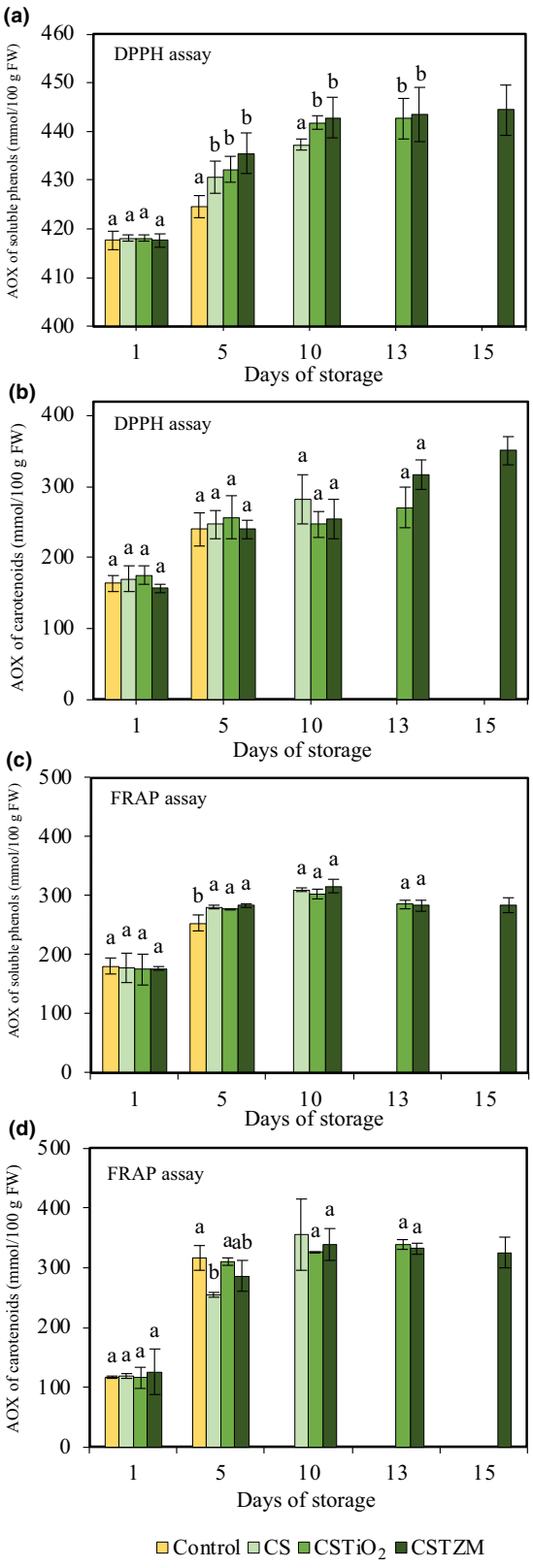

Figure 2 Antioxidant capacity measured by DPPH assay from phenolic (a) and carotenoid extracts (b) and by FRAP assay of phenolic (c) and carotenoid (d) extracts in uncoated jackfruit bulbs (control bulbs), bulbs coated with chitosan (CS), with chitosan-TiO2 hybrid (CSTiO2) and with chitosan-TiO2-ZnO-MgO hybrid (CSTZM), during storage at 4 °C. Lowercase letters indicate significant statistical differences ($P < 0.05$) between treatments.

bioactive compounds, although it depends on the type of coating.

Antioxidant capacity

Using the DPPH assay (Fig. 2a,b), the coated bulbs presented higher AOX than control bulbs, and CSTZM bulbs exhibited the highest AOX (444.36 mmol/100 g FW) from soluble phenol extract and carotenoid extract (350.6 mmol/100 g FW). According to Liu *et al.* (2019), the highest neutralisation capacity of the DPPH radical is from extractable phenolic compounds associated with proton donation; therefore, the AOX of the soluble phenol extract was higher than the carotenoid extract. In contrast, the AOX of both extracts determined by the FRAP assay (Fig. 2c,d) was lower than DPPH and increased at the end of storage. It can be related to polyphenols that are being released and carotenoid synthesis when bulbs are ripening; both have a metal chelating capacity, and they can donate protons and an electron to hydroxyl, peroxyl and peroxynitrite radicals (Liu *et al.*, 2019; Rey *et al.*, 2020).

Toxicity with *Artemia salina* test

No toxicity of coated samples was observed in any of the evaluated treatments. Anaya-Esparza *et al.* (2019) demonstrated that mixed oxide nanoparticles are not toxic to *A. salina* at 4000 µg/mL. In addition, bioaccumulation of TiO_2 nanoparticles inside the gut of *A. salina* has not been shown to induce mortality after 24 h of exposure (Cornejo-Garrido *et al.*, 2011).

Conclusions

The CSTZM hybrid coating preserved the microbiological quality, bioactive compounds and the content of nutrients of jackfruit bulbs. Also, it retarded the physicochemical and physiological changes. This same coating had a significantly longer shelf life (up to 15 days) than control bulbs, CS and $CSTiO_2$ bulbs. Additionally, the coated jackfruit bulbs exhibited no toxicity, as determined by the *Artemia salina* assay. The CSTZM hybrid coating could be used in future research as a new alternative for extending shelf life,

preserving bioactive compounds and the nutritional quality in other fresh-cut fruits or perishable whole fruits.

Acknowledgments

The authors thank CONACYT-Mexico for the scholarship (771579) awarded to Ramírez-Concepción, H.R.

Author contributions

Heidi Rubí Ramírez-Concepción: Formal analysis (equal); writing – original draft (equal). **Luis Miguel Anaya-Esparza:** Formal analysis (equal); writing – original draft (equal). **María de Lourdes García-Magaña:** Writing – review and editing (equal). **Elhadi M. Yahia:** Writing – review and editing (equal). **Libier Meza-Espinoza:** Data curation (equal); formal analysis (equal). **Efigenia Montalvo-González:** Conceptualization (lead); funding acquisition (lead); project administration (lead); validation (lead); writing – original draft (lead).

Conflict of interest

The authors reported no conflict of interest.

Ethical approval

Ethical approval was not required for this research.

Funding information

This research was supported by TecNM (grant no. 13795.22-P).

Peer review

The peer review history for this article is available at https://www.webofscience.com/api/gateway/wos/peer-review/10.1111/ijfs.16516.

Data availability statement

Research data are not shared.

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Supporting Information

Additional Supporting Information may be found in the online version of this article:

Table S1. Microbial counts from uncoated jackfruit bulbs (control bulbs), bulbs with chitosan coatings (CS bulbs), bulbs with chitosan-TiO_2 hybrid coating ($CSTiO_2$ bulbs) and bulbs with chitosan-TiO_2-ZnO-MgO hybrid coating (CSTZM bulbs), during of storage at 4 °C.

Table S2. Total soluble solids, pH, titratable acidity and colour from uncoated jackfruit bulbs (control bulbs), bulbs with chitosan coating (CS bulbs), bulbs with chitosan-TiO_2 hybrid coating ($CSTiO_2$ bulbs) and bulbs with chitosan-TiO_2-ZnO-MgO hybrid coating (CSTZM bulbs), during of storage at 4 °C.