

Nano-ZnO-reinforced chitosan–alginate edible coating for postharvest preservation of banana

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Abstract: Banana is highly perishable at tropical ambient temperatures, with postharvest losses of 25–50% along the supply chain. This work reports a chitosan–alginate/ZnO nanocomposite edible coating for banana. Nano-ZnO prepared by co-precipitation and calcined at 400 °C was phase-pure hexagonal wurtzite (JCPDS 36-1451), quasi-spherical and ≈ 20 nm, with a BET surface area of $36.74 \text{ m}^2 \text{ g}^{-1}$. Alginate at 5–20% w/w of chitosan plasticised the blend, lowering tensile strength from 26.97 MPa (neat chitosan) to 9.29 MPa at 15% while raising elongation at break from 2.09 to 9.38%. Against that baseline nano-ZnO acted as a reinforcing filler with a sharp optimum: 10% w/w (CA15Z10) gave 38.75 MPa and 12.86% elongation, whereas 15% reduced both to 16.87 MPa and 5.92%, electron microscopy showing even dispersion at 10% and clustering at 5% and 15%. All ZnO-containing films inhibited *Staphylococcus aureus*, but none inhibited *Escherichia coli*. Banana dip-coated with CA15Z10 and stored at 30 °C and 80% relative humidity for 14 days showed 10% decay incidence against 50% for uncoated fruit, peel a^* of 5.8 against 8.5, pulp firmness at day 11 of ≈ 2 N against ≈ 0.4 N and total organic acid of 0.55% against 0.67%, but only 21% against $\approx 24\%$ weight loss. Ten per cent w/w ZnO is the compositional optimum for this matrix, and the coating delays ripening rather than merely restricting water loss.

Keywords: Chitosan–Alginate; Zinc Oxide Nanoparticles; Edible Coating; Banana; Postharvest; Nanocomposite Film

1. Introduction

Banana (*Musa* spp.) is among the most widely traded tropical fruits and, at the same time, among the most fragile. It is a climacteric fruit with a short green life, and a recent review of the banana supply chain reports losses of 25–50% between harvest and retail, with high storage temperature and relative humidity alone accounting for 25–40% of the loss [1]. In tropical producing regions, where fruit routinely moves through ambient conditions close to 30 °C, the interval between harvest maturity and the onset of full yellowing and decay can be shorter than a week. Refrigerated chains would solve much of this, but they are capital-intensive and largely unavailable to smallholders, so interventions that work at ambient temperature and cost little per fruit remain the practical target.

Edible coatings are one such intervention. A thin polysaccharide layer deposited on the peel modifies the gas exchange at the fruit surface, restricts transpiration, and can carry antimicrobial agents to the site where spoilage begins. Chitosan is the most studied polysaccharide for this purpose. Obtained by alkaline deacetylation of chitin from crustacean shells, it carries primary amino groups that become protonated below its pK_a of about 6.3, which makes it the only common natural polysaccharide that is cationic in mildly acidic media. That positive charge underlies its intrinsic antibacterial activity: electrostatic interaction between $-\text{NH}_3^+$ groups and anionic components of the bacterial envelope is now regarded as the dominant mechanism, with cell penetration and metal chelation as secondary routes [2]. Activity scales with the degree of deacetylation, which governs the density of available amino groups [3], and the same charge that confers activity also confines it to acidic conditions. Neat chitosan films, moreover,

are brittle and moisture-sensitive, and lose homogeneity near physiological pH - limitations that have driven the search for blends rather than single-component films.

Sodium alginate is a natural partner. Its guluronate and mannuronate blocks carry carboxylate groups that complex electrostatically with the protonated amino groups of chitosan; turbidity, zeta-potential and interfacial studies of chitosan–alginate interpolyelectrolyte complexes show maximum association close to stoichiometric charge equivalence [4]. Alginate films in isolation give good oxygen and lipid barriers but are brittle and highly water-permeable [5], and when blended into chitosan the effect on mechanical behaviour is not simply additive: plasticisation of an ionically complexed chitosan–alginate network acts on the complex itself in ways that are not predicted from either component alone [6], and ionic crosslinking of the blend can raise flexibility while lowering tensile strength [7]. In practice, adding alginate to chitosan softens the film useful for a coating that must follow the curvature of a fruit without cracking, but costly in strength. Zinc oxide nanoparticles offer a way to recover that strength while adding a second, non-pH-dependent antimicrobial function. Their antibacterial action is usually attributed to three concurrent routes — direct membrane disruption, generation of reactive oxygen species, and release of Zn^{2+} [8] — and the relative weight of these routes is still being resolved: membrane damage can be detected in 70% of cells within 15 min of exposure [9], while experiments using chelation and physical separation of particles from bacteria indicate that soluble Zn^{2+} dominates toxicity in standard broth assays [10]. Particle size and specific surface area modulate all three routes, and capping agents that control size and surface chemistry change

antibacterial performance accordingly [11]. As a filler, ZnO frequently reinforces biopolymer films, but only up to a threshold: in a chitosan–xanthan matrix, tensile strength rose from 5.68 to 24.42 MPa between 0 and 3 wt% and then fell at 5 wt% through nanofiller agglomeration [12]; in chitosan–lecithin–montmorillonite films, improvement was confined to loadings of 3 wt% or less [13]; a response-surface optimisation of chitosan/ZnO/stearic acid films landed on 1% ZnO [14]. The direction of the effect is not even universal — in one chitosan system ZnO plasticised rather than reinforced [15] — which indicates that dispersion state and interfacial chemistry, not ZnO content alone, determine the outcome in a given matrix. On banana specifically, ZnO-bearing coatings have produced benefits that are clearest on decay and ripening rather than on water loss. A chitosan/gum arabic coating loaded with ZnO kept fruit marketable for more than 17 days at 35 °C, against fewer than 13 days for uncoated fruit [16]; chitosan carrying 0.3% green-synthesised ZnO reduced weight loss at day 20 from 21% to 12% at 28 °C [17]; a ZnO/Ag₂O composite treatment held decay incidence at 44.4% after 12 days at 25 °C where untreated fruit reached 100% [18]. What has not been reported is the combination examined here. Chitosan–alginate matrices loaded with metal oxides have been studied as packaging films — a sodium alginate–chitosan film containing CuO/ZnO achieved more than 90% bacterial growth inhibition [19], but such films have not, to our knowledge, been formulated as a dip coating and applied to banana, and the ZnO loading that optimises a chitosan–alginate matrix has not been established. This study therefore had three objectives: to synthesise and characterise nano-ZnO by a simple co-precipitation route suitable for scale-up; to determine the alginate and ZnO contents that give a chitosan-based film the combination of strength, extensibility and antibacterial activity required of a fruit coating; and to test the optimised formulation on banana under the ambient conditions (30 °C, 80% relative humidity) that actually prevail in tropical distribution. Both the compositional optimum and the limits of the storage trial are reported explicitly.

2. Materials and methods

2.1. Plant material

Green “tieu” banana (*Musa acuminata*, Cavendish subgroup) was used. Hands were harvested 115–120 days after flower emergence, corresponding to 80–90% maturity, and fruit were selected for uniform dark-green peel, full finger size, rounded rather than angular cross-section, and absence of insect damage, mechanical injury, scarring or visible rot. Fruit was transported to the laboratory on the day of harvest and processed immediately.

2.2. Chemicals

Chitosan (99% purity, white powder, viscosity-average molecular weight 82 kDa) was supplied by Duc Giang Chemical Joint Stock Company (Viet Nam). Sodium alginate (90%) was from Shanghai Macklin Biochemical Co., Ltd (China). Acetic acid (CH₃COOH) and zinc chloride dihydrate (ZnCl₂·2H₂O) were from Xilong Scientific Co., Ltd (China). Cetyltrimethylammonium bromide (CTAB, C₁₉H₄₂BrN, M = 364.46 g mol⁻¹) was from Oxford Lab Fine Chem LLP (India). Ammonia solution (NH₃, 30%) was from Shantou (Guangdong, China). Glycerol was used as plasticiser. All reagents were used as received without further purification, and distilled water was used throughout.

2.3. Synthesis of nano-ZnO

Nano-ZnO was prepared by co-precipitation (Fig. 1A). ZnCl₂·2H₂O was dissolved completely in distilled water to give solution A, and CTAB was dissolved separately in distilled water to give solution B. Solution A was added dropwise into solution B under continuous stirring while the pH of the system was adjusted to 10 with 30% NH₃. The reaction mixture was then stirred at 80 °C for 2 h, yielding a white suspension. The precipitate was separated by centrifugation, washed twice with distilled water and once with ethanol to remove impurities and residual ions, dried at 80 °C for 12 h to give the anhydrous ZnO precursor, and finally calcined at 400 °C for 4 h to obtain nano-ZnO.

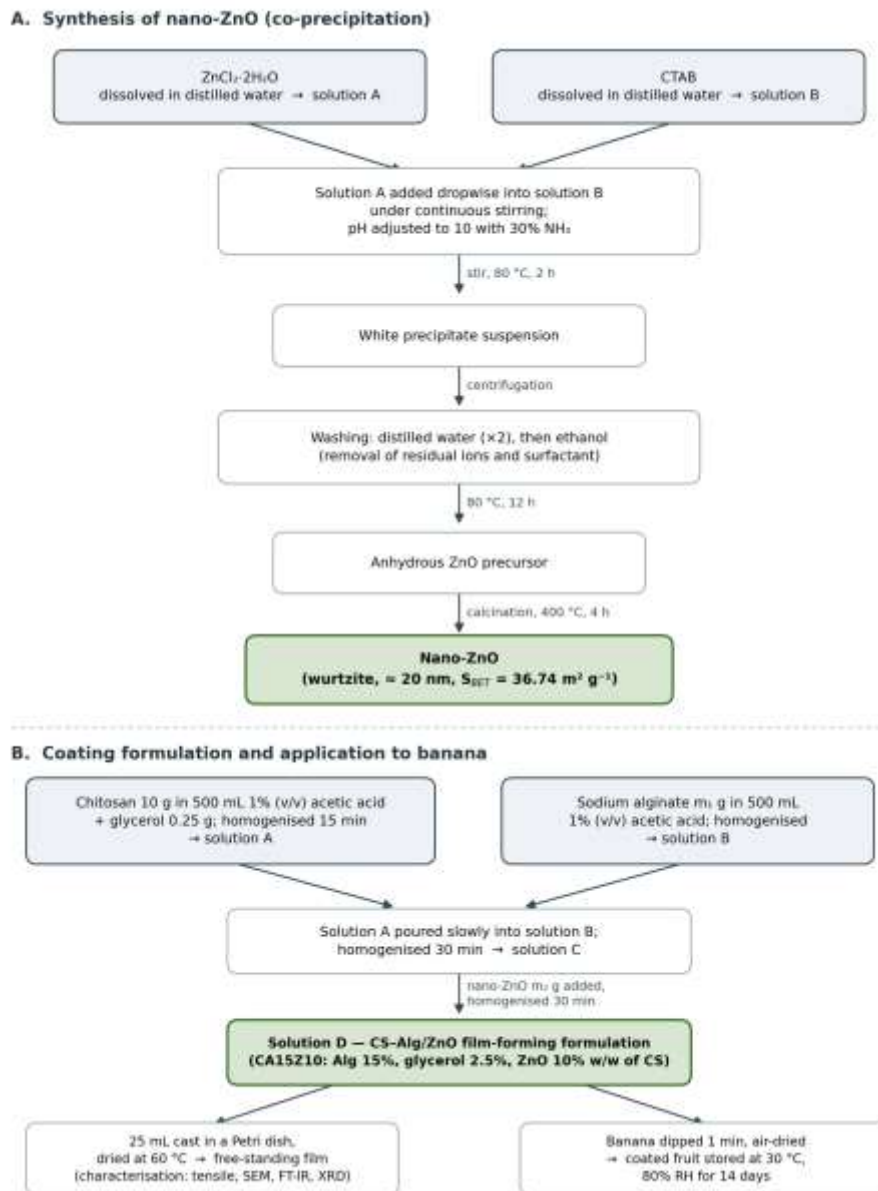


Figure 1. Process flow diagram of material preparation. (A) Synthesis of nano-ZnO by co-precipitation. (B) Preparation of the chitosan–alginate/ZnO film-forming formulation and its two uses: casting as a free-standing film for characterisation, and dip coating of banana for the storage trial.

2.4. Preparation of the CS–Alg/ZnO film-forming formulation

The formulation was prepared in three steps (Fig. 1B). *Step 1:* chitosan (10 g) was dissolved in 500 mL of 1% (v/v) acetic acid; glycerol (0.25 g, i.e. 2.5% w/w relative to chitosan) was added and the mixture homogenised for 15 min to give solution A. In parallel, alginate (m_1 g) was dissolved in 500 mL of 1% (v/v) acetic acid and homogenised to give solution B. Solution A was poured slowly into solution B and the mixture homogenised for 30 min to give solution C. *Step 2:* nano-ZnO (m_2 g) was weighed and added directly to solution C, and the suspension homogenised for a further 30 min to give solution D, the film-forming formulation. Because the two 500 mL solutions are

combined, the final formulation contains chitosan at 1% (w/v). *Step 3:* for characterisation of the material as a free-standing film, 25 mL of solution D was cast in a Petri dish and dried at 60 °C. The whole process was carried out at an ambient temperature of 30 °C.

Alginate and ZnO contents are expressed throughout as percentages by mass relative to chitosan. Alginate was varied at 5, 10, 15 and 20% (samples CA5–CA20) at constant glycerol content, and ZnO at 5, 10 and 15% on the selected CA15 base (samples CA15Z5, CA15Z10, CA15Z15). Compositions are given in Table 1. For the optimised formulation CA15Z10, this corresponds to $m_1 = 1.5$ g of alginate and $m_2 = 1.0$ g of nano-ZnO per 10 g of chitosan.

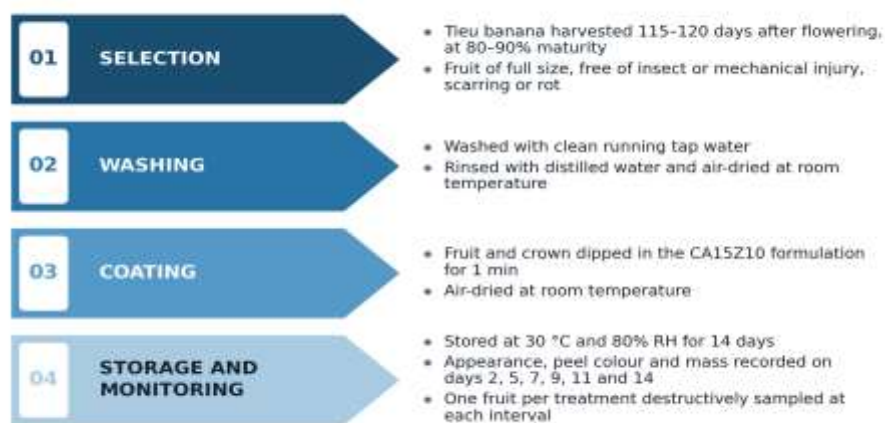
Table 1. Composition of the chitosan–alginate and chitosan–alginate/ZnO film-forming formulations. All contents are expressed as percentages by mass relative to chitosan.

Sample	Alginate (% w/w of CS)	Glycerol (% w/w of CS)	Nano-ZnO (% w/w of CS)
CA5	5	2.5	0
CA10	10	2.5	0
CA15	15	2.5	0
CA20	20	2.5	0
CA15Z5	15	2.5	5
CA15Z10	15	2.5	10
CA15Z15	15	2.5	15

2.5. Coating procedure and storage trial

The coating and storage protocol is summarised in Fig. 2. Fruit were sorted, washed with running tap water followed by distilled water, and air-dried at room temperature. Individual fingers, including the crown, were dipped in the CA15Z10 formulation for 1 min and then air-dried. Coated fruit and

uncoated controls (designated C) were stored side by side at an ambient temperature of 30 °C and 80% relative humidity for 14 days. Fruit appearance, peel colour and mass were recorded on days 2, 5, 7, 9, 11 and 14. At each of these intervals one fruit per treatment was destructively sampled for pulp firmness and total organic acid content.

**Figure 2.** Coating and storage protocol applied to banana with the CA15Z10 formulation.

2.6. Fourier-transform infrared spectroscopy

Chitosan, alginate and the CA15Z10 nanocomposite were analysed on a Nicolet iS10 spectrometer (Thermo Fisher Scientific, USA) at the Institute of Materials Science, Vietnam Academy of Science and Technology.

2.7. X-ray diffraction

Nano-ZnO powder and the CA15Z10 film were analysed on a D8 Advance diffractometer (Bruker, Germany) at the Institute of Chemistry, Vietnam Academy of Science and Technology. Diffractograms were indexed against JCPDS card 36-1451 for hexagonal wurtzite ZnO.

2.8. Mechanical testing

Tensile strength and elongation at break were measured on a Zwick Z2.5 universal testing machine (ZwickRoell, Germany) at the Institute of Materials Science. Specimens were cut into rectangles of 2 × 5 cm according to ASTM D882 and clamped at both ends, and the crosshead speed was 25 mm min⁻¹.

2.9. Electron microscopy and nitrogen physisorption

Surface morphology of the CS–Alg/ZnO films was examined by scanning electron microscopy (JSM-6510V, JEOL, Japan) at the Institute of Materials Science. Nano-ZnO was examined by transmission electron microscopy (JEOL 210, Japan) at the

same institute. Specific surface area, pore volume and mean pore diameter were determined by nitrogen physisorption at 77 K on a Micromeritics gas adsorption analyser (USA) at the Institute of Chemistry, applying the Brunauer–Emmett–Teller model; both the nano-ZnO powder and the CA15Z10 film were analysed, and the nano-ZnO results are reported here.

2.10. Antibacterial assay

Antibacterial activity of the nanocomposite films was determined by agar diffusion against *Escherichia coli* (Gram-negative) and *Staphylococcus aureus* (Gram-positive). A 0.2 mL aliquot of bacterial suspension at approximately 10⁶ CFU mL⁻¹ was spread evenly over tryptone soya agar in a Petri dish. Film discs of 7 mm diameter were placed on the inoculated surface, and plates were incubated at 25–27 °C for 24–48 h. Filter-paper discs served as the negative control. Inhibition zones, where formed, were measured across their diameter

2.11. Quality indices of stored fruit

Weight loss. Fruit were weighed on days 2, 5, 7, 9, 11 and 14 on an analytical balance with a weighing range of 0.50–2200.00 g and a readability of 0.01 g. Weight loss was calculated as:

$$(\%) = \left[\frac{M_0 - M_t}{M_0} \right] \times 100 \quad (1)$$

where M_0 is the initial fruit mass (g) and M_t the mass after t days of storage (g). Each determination was repeated at least three times.

Peel colour. Colour was measured with a portable NR3000 colorimeter at five positions per fruit — adjacent to the crown, at three points around the mid-section, and at the blossom end — and the mean taken as the value for that fruit. Results are expressed in the CIE $L^*a^*b^*$ system (Fig. 3), in which L^* runs from 0 (black) to 100 (white), a^* from green (negative) to red (positive) and b^* from blue (negative) to yellow (positive).

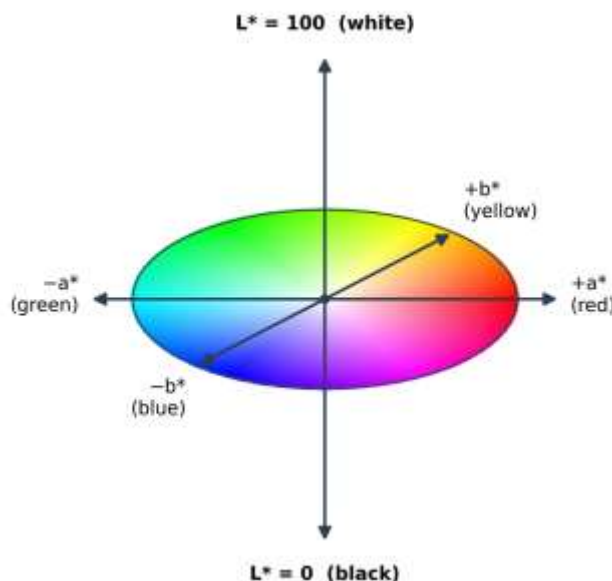


Figure 3. The CIE $L^*a^*b^*$ colour space used to express peel colour. L^* is the lightness axis; a^* runs from green to red and b^* from blue to yellow in the chromatic plane.

Pulp firmness. Firmness was measured with an FHM-5 fruit hardness penetrometer (Japan) and is reported as the maximum penetration force, in newtons (N), recorded as the probe was driven into the pulp.

Decay incidence. Decay incidence was calculated as $P/Q \times 100$, where P is the number of fruit showing rot during storage and Q the total number of fruit monitored in that treatment.

Total organic acid content. Total organic acid was determined by neutralisation titration according to TCVN 5483:2007. Banana pulp was ground with 40 mL of water, the mixture centrifuged at 6000 rpm for 10 min and the supernatant filtered. A 5 mL aliquot of the filtrate was treated with 2–3 drops of 0.1% phenolphthalein and titrated with 0.1 N NaOH until a stable pink colour appeared. Content was calculated as

$$A (\%) = \frac{(V \times N \times V_1 \times 0.067 \times 100)}{(m \times V_0)} \quad (2)$$

where V is the volume of NaOH used (mL), N its normality (0.1 N here), V_1 the total volume of the sample extract (mL), V_0 the

volume of extract titrated (mL) and m the mass of the fruit sample (g); the factor 0.067 g mL⁻¹ is the mass of malic acid equivalent to 1 mL of 1 N NaOH.

3. Results

3.1. Structure and texture of the synthesised nano-ZnO

The X-ray diffractogram of the calcined powder (Fig. 4) shows sharp reflections at $2\theta \approx 32.0^\circ, 34.9^\circ, 36.6^\circ, 47.8^\circ, 56.7^\circ, 62.8^\circ$ and 68.2° , corresponding to the (100), (002), (101), (102), (110), (103) and (112) planes of hexagonal wurtzite ZnO. The pattern matches JCPDS card 36-1451 closely and contains no additional reflections, indicating that co-precipitation produced a single, well-crystallised phase with no detectable residue of the precursor or of the surfactant. The precipitation pH and the calcination temperature used here lie within the ranges reported to yield phase-pure, well-crystallised ZnO by precipitation routes [20].

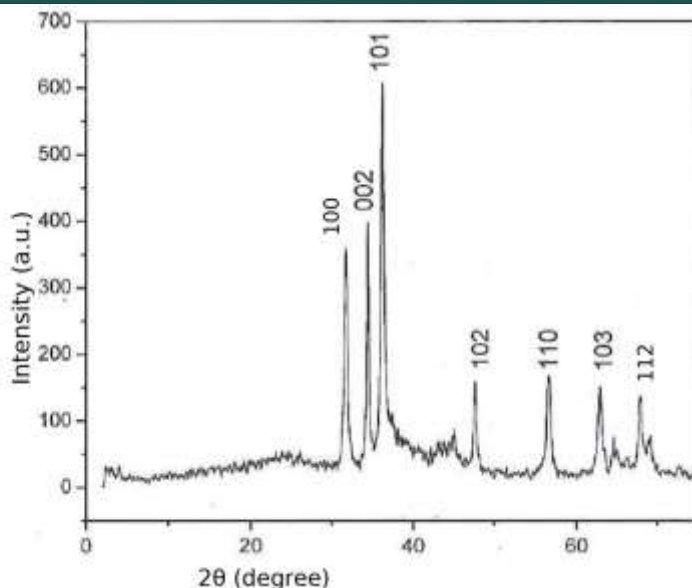


Figure 4. X-ray diffractogram of the nano-ZnO obtained by co-precipitation, indexed to hexagonal wurtzite ZnO (JCPDS 36-1451). The nitrogen adsorption–desorption isotherm (Fig. 5) is of type IV in the IUPAC classification, the signature of a mesoporous solid. Three regions are distinguishable: monolayer adsorption at low relative pressure ($P/P_0 \approx 0-0.3$), multilayer development at intermediate pressure ($0.3-0.7$), and capillary condensation within the pore network above $P/P_0 \approx 0.7$, where the isotherm rises steeply. The BET specific surface area was $36.74 \text{ m}^2 \text{ g}^{-1}$, the pore volume $0.133 \text{ cm}^3 \text{ g}^{-1}$ and the mean pore diameter 6.75 nm , the last value placing the material firmly in the mesoporous range. Type IV isotherms and mesoporous texture are characteristic of nano-ZnO; reported BET areas for ZnO nanostructures span approximately $3-84 \text{ m}^2 \text{ g}^{-1}$ depending on morphology, and the highest areas are associated with the strongest antibacterial effect [21].

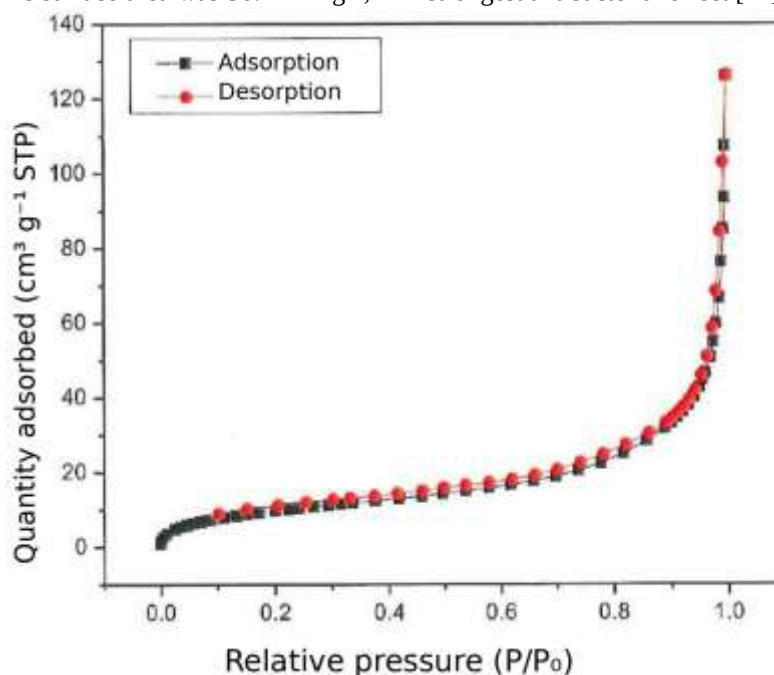


Figure 5. Nitrogen adsorption–desorption isotherm of nano-ZnO at 77 K.

Scanning and transmission electron microscopy (Fig. 6) show quasi-spherical particles with a mean size of approximately 20 nm . The particles are distributed fairly evenly with limited aggregation, and distinct particle boundaries are visible in the transmission images, confirming the nanometric dimension of

the product. The equivalent spherical diameter derived from the BET surface area, $d = 6/(\rho S)$ with $\rho = 5.606 \text{ g cm}^{-3}$ and $S = 36.74 \text{ m}^2 \text{ g}^{-1}$, is approximately 29 nm ; that this exceeds the microscopic estimate is consistent with the limited aggregation visible in the images.

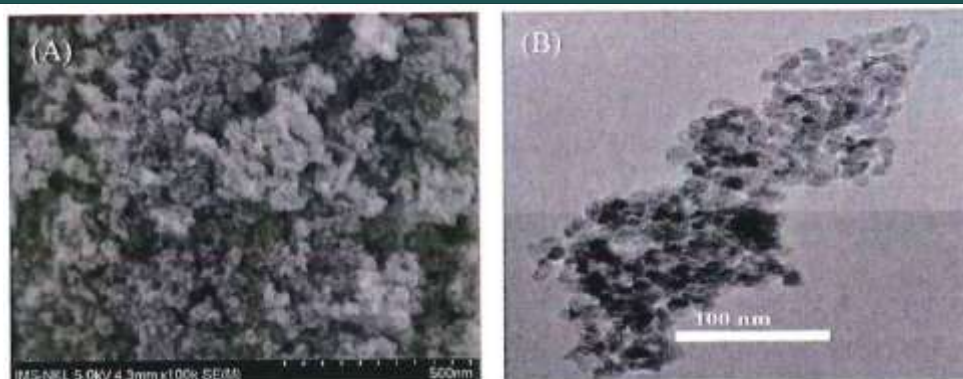


Figure 6. Scanning electron micrograph (A) and transmission electron micrograph (B) of the nano-ZnO.

3.2. Mechanical properties of the CS–Alg and CS–Alg/ZnO films

Tensile strength and elongation at break of the cast films are given in Table 2.

Table 2. Tensile strength and elongation at break of the cast chitosan–alginate and chitosan–alginate/ZnO films. Values are means $\pm$ standard deviation.

Sample	Alginate (% w/w of CS)	Nano-ZnO (% w/w of CS)	Tensile strength, σ (MPa)	Elongation at break, ϵ (%)
CS	0	0	26.97 ± 1.73	2.09 ± 3.21
CA5	5	0	11.95 ± 2.43	5.33 ± 2.85
CA10	10	0	10.34 ± 1.70	7.38 ± 0.74
CA15	15	0	9.29 ± 1.16	9.38 ± 2.79
CA20	20	0	8.68 ± 1.15	7.72 ± 2.62
CA15Z5	15	5	22.83 ± 4.04	7.23 ± 1.76
CA15Z10	15	10	38.75 ± 0.76	12.86 ± 3.96
CA15Z15	15	15	16.87 ± 2.71	5.92 ± 1.63

Neat chitosan was strong but almost inextensible (26.97 ± 1.73 MPa; 2.09% elongation). Adding alginate lowered tensile strength progressively and monotonically — to 11.95, 10.34, 9.29 and 8.68 MPa at 5, 10, 15 and 20% w/w respectively — while elongation at break rose from 2.09% to a maximum of 9.38% at 15% alginate. The loss of strength is consistent with alginate chains interrupting the hydrogen-bond network between chitosan chains and loosening the polymer network, while the gain in extensibility reflects the added flexibility of the complexed blend. CA15 was carried forward as the base composition because it combined the highest elongation at break with a tensile strength adequate for a flexible, crack-resistant coating.

Nano-ZnO reversed the softening trend, but only within a narrow window. At 5% w/w, tensile strength recovered to 22.83 ± 4.04 MPa with elongation at break falling slightly, from 9.38 to 7.23%, a difference within the scatter of the measurement. At 10% (CA15Z10), tensile strength reached 38.75 ± 0.76 MPa — 4.2 times that of the unfilled CA15 blend and 1.4 times that of neat chitosan, and elongation at break simultaneously reached its highest value of $12.86 \pm 3.96\%$. Raising the loading to 15% reduced both properties sharply, to 16.87 ± 2.71 MPa and 5.92

$\pm 1.63\%$. A simultaneous increase in strength and extensibility, as seen at 10%, indicates genuine load transfer from matrix to well-dispersed filler rather than simple stiffening; the collapse of both properties at 15% is the classic signature of nanofiller agglomeration disrupting the continuity of the polymer network.

3.3. Film morphology

Scanning electron micrographs of the three ZnO-containing films (Fig. 7) show the same ordering as the mechanical data. At 5% w/w (Fig. 7A) the particles are unevenly distributed: discrete clusters of micrometre scale are visible against large areas of bare film, so that the loading is simultaneously too low to cover the surface and prone to local aggregation. At 10% (Fig. 7B) the surface is densely and uniformly populated with fine features, with no large clusters. At 15% (Fig. 7C) coverage is dense, but the uniformity is lost, and larger aggregates reappear, consistent with particle–particle attraction outcompeting particle–matrix interaction once the optimum loading is exceeded. CA15Z10 thus shows the most even distribution of the three, in agreement with its mechanical behaviour.

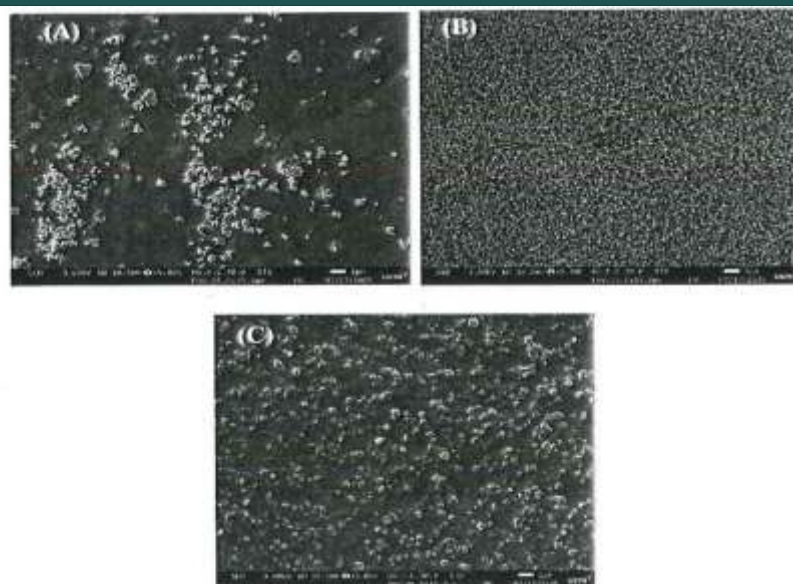


Figure 7. Scanning electron micrographs of the nanocomposite films: (A) CA15Z5, (B) CA15Z10, (C) CA15Z15.

3.4. Antibacterial activity

Agar diffusion results are shown in Fig. 8. Against *S. aureus* (Fig. 8A) the negative control produced no zone, whereas all three ZnO-containing films generated visible inhibition zones; CA15Z10 gave the clearest zone of the three, although the differences between loadings were not pronounced. Against *E.*

coli (Fig. 8B) none of the films produced an inhibition zone under the conditions used. The contrast between the two strains is consistent with the different architecture of the Gram-positive and Gram-negative envelope and the resulting difference in susceptibility to the antibacterial species released from the film.

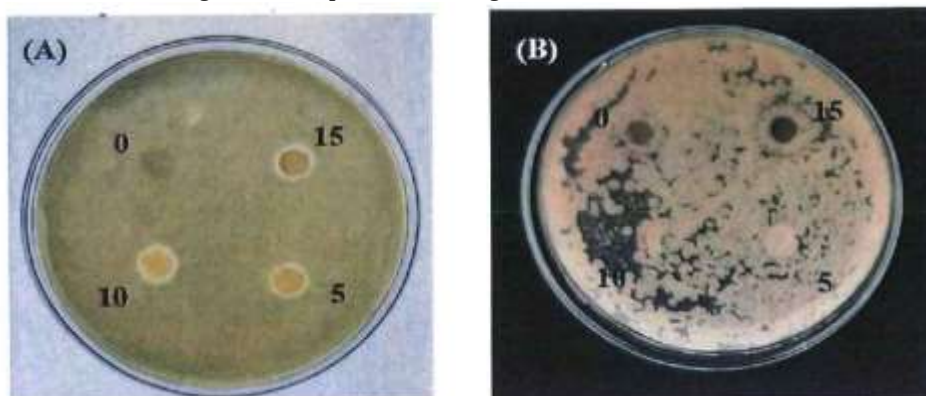


Figure 8. Agar diffusion assay of the CA15Z5, CA15Z10 and CA15Z15 films, marked 5, 10 and 15 respectively, with a filter-paper negative control marked 0, against *Staphylococcus aureus* (A) and *Escherichia coli* (B).

On the combined evidence of dispersion, mechanical performance and activity against *S. aureus*, CA15Z10 was selected for the fruit trial.

3.5. Structural characterisation of CA15Z10

The infrared spectra of chitosan, alginate and the nanocomposite are compared in Fig. 9. Chitosan shows a broad band at 3358.82 cm^{-1} (overlapping --OH and --NH stretching) together with amide I at 1642.84 cm^{-1} and amide III at 1420.59 cm^{-1} . Alginate shows --OH stretching at 3228.23 cm^{-1} and the asymmetric and symmetric carboxylate stretches at 1594.50 and 1407.31 cm^{-1} . In the CA15Z10 spectrum the corresponding

chitosan bands are displaced to lower wavenumber — the --OH/--NH envelope to 3290.17 cm^{-1} , the carbonyl/carboxylate region to 1584.53 cm^{-1} and the 1420.59 cm^{-1} band to 1375.34 cm^{-1} — and the intensity of the glycosidic C--O--C region at $1020\text{--}1030\text{ cm}^{-1}$ changes. Displacement of the hydroxyl, amine and carboxylate bands indicates hydrogen bonding and surface coordination between ZnO and the functional groups of the CS–Alg network. Bands near $600\text{--}660\text{ cm}^{-1}$ appear in all three spectra and therefore cannot be assigned unambiguously to Zn–O stretching in this data set.

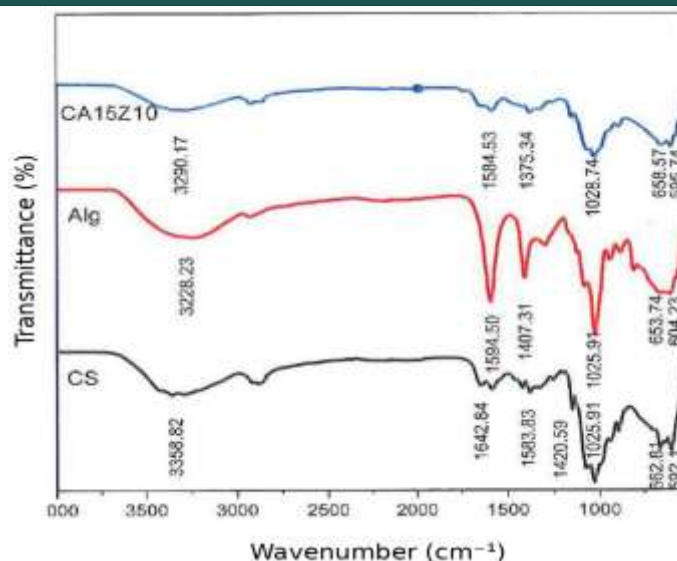


Figure 9. Fourier-transform infrared spectra of chitosan (CS), sodium alginate (Alg) and the CA15Z10 nanocomposite.

The diffractogram of CA15Z10 (Fig. 10) retains the chitosan reflections, marked with asterisks, together with reflections at $2\theta \approx 32.5^\circ$, 35.1° , 37.5° , 57.7° , 62.8° and 68.2° indexed to the (100), (002), (101), (110), (103) and (112) planes of wurtzite ZnO. Retention of the full wurtzite pattern shows that the

nanoparticles are incorporated into the polymer matrix without loss of crystalline structure, while the persistence of the chitosan reflections shows that the polysaccharide network is not disrupted by the filler.

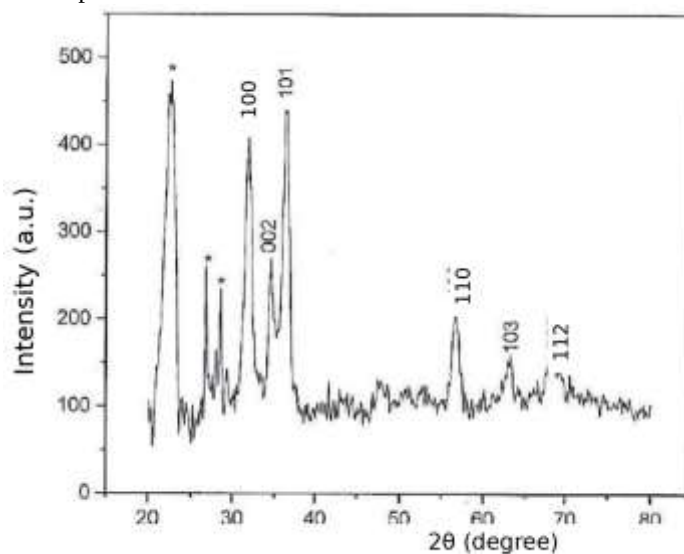


Figure 10. X-ray diffractogram of the CA15Z10 nanocomposite film. Asterisks mark the reflections of chitosan; the remaining reflections are indexed to wurtzite ZnO.

3.6. Storage performance on banana

Appearance and ripening. Visual changes over 14 days are shown in Fig. 11. Uncoated fruit turned from green to stages 4–5 of the standard banana colour scale (more yellow than green,

to yellow) from day 7 onwards, and by day 14 the peel was heavily covered in brown senescent spotting. Coated fruit retained a uniformly green peel through to day 14.

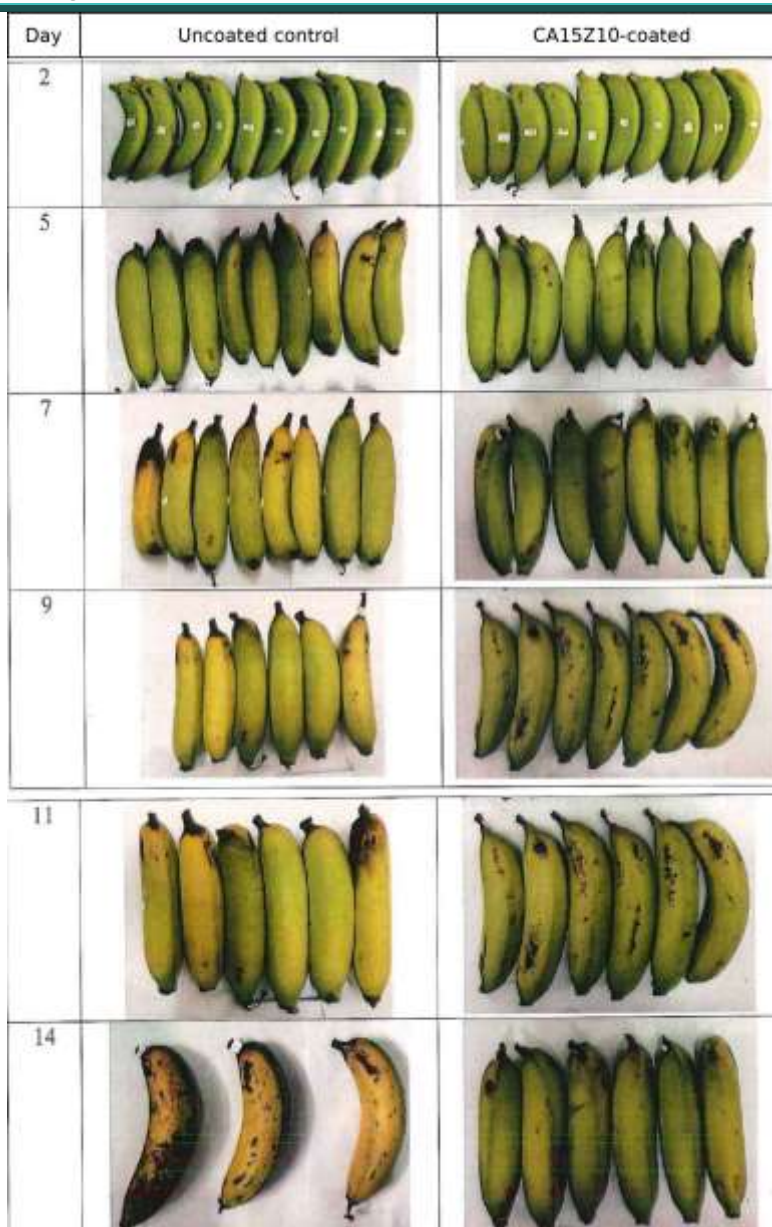


Figure 11. Appearance of uncoated control bananas and bananas coated with the CA15Z10 formulation over 14 days of storage at 30 °C and 80% relative humidity.

Decay incidence. Decay data are given in Table 3.

Table 3. Decay incidence of banana coated with the CA15Z10 formulation and of uncoated control fruit during storage at 30 °C and 80% relative humidity.

Storage time (days)	Decay incidence, control (%)	Decay incidence, CA15Z10 (%)
2	0	0
5	5	0
7	10	0
9	15	0
11	20	5
14	50	10

Coated fruit showed no decay at all through the first week, at which point the uncoated control had already reached 10%. The gap widened thereafter: 15% versus 0% at day 9, 20% versus

5% at day 11, and 50% versus 10% at day 14 — a five-fold difference at the end of the trial.

Peel colour. Peel lightness increased in both treatments (Fig. 12) as chlorophyll was degraded, but the two treatments

differed markedly in rate. Uncoated fruit rose steeply to $L^* \approx 65.5$ by day 11 before declining slightly to 64.3 at day 14, the decline reflecting the onset of brown spotting. Coated fruit remained 3–7 units darker throughout and reached only $L^* \approx 57$ at day 14. The a^* coordinate (Fig. 13) rose in both treatments as chlorophyll was replaced by carotenoids, from approximately -5.2 at day 2; uncoated fruit crossed into positive values by day 5 and reached ≈ 8.5 at day 14, whereas coated fruit remained negative until about day 9 and reached only ≈ 5.8 . Measurements of b^* followed the same pattern, the uncoated control reaching approximately 46 at day 11 against 37.5 for coated fruit.

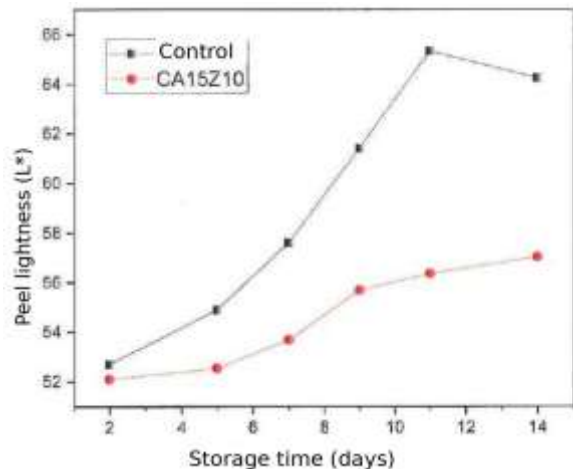


Figure 12. Change in peel lightness (L^*) of uncoated control and CA15Z10-coated bananas during storage.

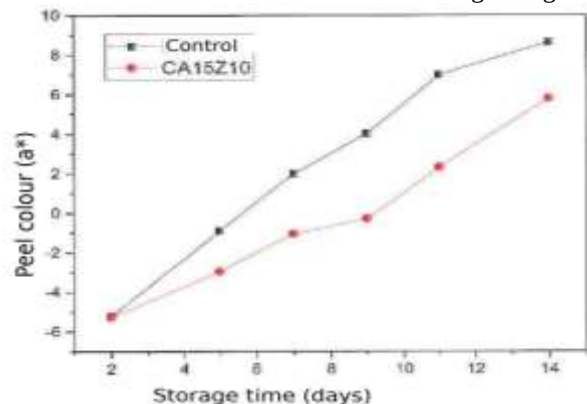


Figure 13. Change in peel colour coordinate a^* of uncoated control and CA15Z10-coated bananas during storage.

Weight loss. Weight loss rose steadily in both treatments (Fig. 14) and was indistinguishable between them for the first week. The curves separated from day 9 onwards, and at day 14 the uncoated control had lost approximately 24% of its initial mass against 21% for coated fruit — a relative reduction of about one eighth.

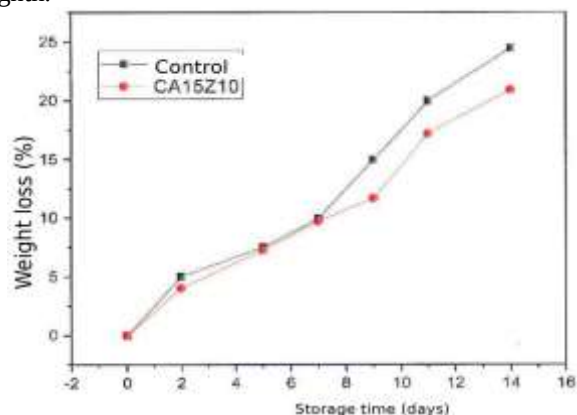


Figure 14. Cumulative weight loss of uncoated control and CA15Z10-coated bananas during storage.

Pulp firmness. Firmness fell from an initial 7.8 N in both treatments (Fig. 15) but at very different rates. Uncoated fruit softened rapidly, dropping to 5.4 N by day 2 and to essentially zero by day 11 (≈ 0.4 N on the plotted curve), at which point the pulp had lost structural integrity. Coated fruit held 7.0 N at day 5, 5.1 N at day 9 and ≈ 2.1 N at day 11, reaching zero only at day 14.

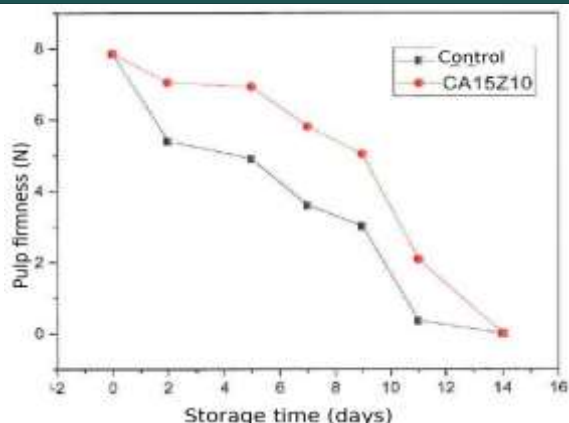


Figure 15. Change in pulp firmness of uncoated control and CA15Z10-coated bananas during storage.

Total organic acid. Total organic acid rose continuously in both treatments from an initial 0.335% (Fig. 16). The uncoated control climbed steeply to 0.67% at day 14, whereas coated fruit reached only 0.55%, about 18% lower. The coating did not change the direction of the trend, but it slowed it throughout the storage period.

4. Discussion

4.1. A sharp compositional optimum in a plasticised matrix

The mechanical data locate a narrow optimum at 10% w/w ZnO relative to chitosan, equivalent to 7.8 wt% of total film solids. Both tensile strength and elongation at break peak there and both collapse at 15%, and the micrographs supply the reason: even dispersion at 10%, clustering at 5% and 15%. The pattern itself is familiar: in a chitosan–xanthan matrix, tensile strength rose to a maximum at 3 wt% ZnO and collapsed at 5 wt% through agglomeration [12]; in chitosan–lecithin–montmorillonite films improvement was confined to 3 wt% or below [13]; and a response-surface optimisation of chitosan/ZnO/stearic acid films converged on 1% [14]. What differs here is the position of the optimum, two to eight times higher than in those chitosan-only systems. The position of that threshold is not fixed even within chitosan-only matrices: in a chitosan coating on cherry tomato, performance improved particle attraction dominates. Consistent with this, neither the direction nor the magnitude of the ZnO effect is universal: in one chitosan system ZnO plasticised rather than reinforced, lowering tensile strength from 46.7 to about 32 MPa while raising ductility [15], whereas at a fixed 1% loading green-

4.2. Selective antibacterial activity

Inhibition zones formed against *S. aureus* but not against *E. coli*. The same ordering has been reported for ZnO nanostructures assayed by zone of inhibition, where complete kill of *S. aureus* was achieved at lower doses than for *E. coli* [24], and it is usually rationalised by the outer membrane and lipopolysaccharide layer of Gram-negative bacteria. The evidence is, however, less settled than it is often presented. A carboxymethyl chitosan/gelatin/ZnO film killed approximately 99% of *E. coli* against 85% of *S. aureus* [25]; an optimised chitosan/ZnO film produced near-identical zones against the two organisms [14]; and the closest published comparator to the

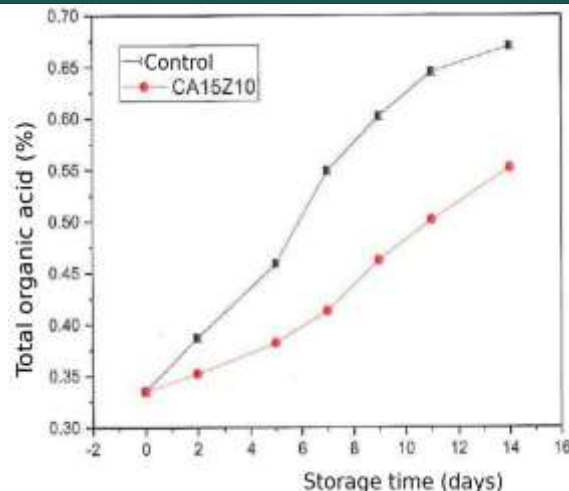


Figure 16. Change in total organic acid content of uncoated control and CA15Z10-coated bananas during storage.

monotonically up to the highest loading tested — 0.9% w/v ZnO in a 1.5% w/v chitosan solution, equivalent to 60% w/w of chitosan — with no reversal [22]. Loadings expressed on different bases are not directly comparable and the substrate differs, but the absence of an upper limit in that system supports the view that the threshold reflects dispersion state and interfacial chemistry rather than a universal ZnO content. The most likely explanation is the state of the matrix into which the filler is placed. Alginate at 15% w/w had already reduced tensile strength from 26.97 to 9.29 MPa by interrupting inter-chain hydrogen bonding, and the carboxylate groups it introduces provide coordination sites for the ZnO surface in addition to the hydroxyl and amino groups of chitosan — an interpretation supported by the band displacements in the infrared spectra (Fig. 9). A network that is both looser and richer in coordinating groups can accommodate more filler before particle–

synthesised ZnO raised tensile strength by 13–39% across cellulose acetate, chitosan and gelatin [23]. The 4.2-fold gain reported here should therefore be read against the softened CA15 baseline, not as a general claim about ZnO in chitosan.

present system — a chitosan film carrying green-synthesised ZnO, applied to banana — gave a 15 mm zone against *E. coli* at both 0.1% and 0.3% loading, equal to its zone against *S. aureus* [17]. Selectivity is therefore better regarded as dependent on morphology, dose, matrix and assay format than as a property of ZnO. Two features of the present assay reinforce that caution. First, agar diffusion measures only what leaves the film, and in a solid polysaccharide matrix that is essentially dissolved Zn^{2+} — the species shown, by chelation and physical-separation experiments, to dominate ZnO toxicity in standard media [10]. A film that immobilises its particles will therefore appear weaker here than a particle suspension, and

absence of a zone is not absence of activity on contact. Second, ZnO prepared with CTAB and combined with sodium alginate has been reported active against both organisms [26], so the selectivity seen here is unlikely to be intrinsic to the CTAB-derived particles. Finally, the decay that limits banana shelf life

Table 4. Performance of the present coating relative to recently reported polysaccharide and ZnO-based coatings on banana and other fruits. Values are as reported in the cited sources.

Coating	Fruit	Conditions	Duration	Weight loss, coated vs control	Other key outcomes	Ref.
CS–Alg/ZnO (CA15Z10)	Banana	30 °C, 80% RH	14 d	21% vs ≈24%	Decay 10% vs 50%; a^* 5.8 vs 8.5; total organic acid 0.55% vs 0.67%	This work
Chitosan/gum arabic + ZnO	Banana	35 °C, 54% RH	>17 d	not reported	Marketable >17 d vs <13 d; firmness and titratable acidity retained	[16]
Chitosan + 0.3% ZnO	Banana	28 °C	20 d	12% vs 21%	Day-15 ripening index 0.016 vs 0.130	[17]
ZnO/Ag ₂ O composite	Banana	25 °C	12 d	14.07–17.95% across all treatments; n.s.	Decay 44.4% vs 100%; a^* –2.39 vs +2.22	[18]
Alginate hydrogel	Banana	20 °C	11 d extension	≈3–4 points lower	Rate of colour change halved; titratable acidity 0.20% vs 0.10%	[28]
Carboxymethyl cellulose 1.5%	Banana	20 °C	15 d	6% (coated)	Decay 9% at day 9; PME, PG and chlorophyllase activity reduced	[29]
Alginate + ZnO	Mango	13 °C	28 d	4.30% vs 5.75%	Firmness 22.65 N vs 14.17 N; titratable acidity 4.48% vs 2.56%	[30]
Chitosan + 3% ZnO	Strawberry	27 °C	9 d	22.9% (coated)	Firmness 21 N; infection incidence 6.2%	[31]

The clearest effects were on ripening rather than on water loss. Weight loss differed by only 3 percentage points at day 14 (21% coated versus ≈24% uncoated), whereas decay incidence differed five-fold (50% versus 10%), firmness at day 11 differed by a factor of five, peel a^* differed by 2.7 units and total organic acid by 18%. The ordering is informative: a coating acting chiefly as a moisture barrier would show the opposite pattern. Slower accumulation of organic acid, slower degreening and retained firmness together point to suppressed metabolic activity, consistent with modified gas exchange at the peel surface. Carboxymethyl cellulose coatings on banana lower pectin methylesterase, polygalacturonase, cellulase and chlorophyllase activity [29], and nanoparticle-enriched coatings on Cavendish banana reduced ethylene production

at 20 °C retained titratable acidity at 0.20% malic acid against 0.10% in uncoated fruit, both falling from an initial 0.40% [28].

is predominantly fungal, and no antifungal testing was performed, although chitosan–ZnO films have inhibited *Alternaria alternata* mycelial growth by 99% [27].

4.3. Fruit performance in context

Table 4 sets the storage results beside recent work.

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from 446 to 144 $\mu\text{L kg}^{-1} \text{h}^{-1}$ [32]; either route would produce the pattern observed here.

The absolute weight losses are high because the trial ran at 30 °C. Chitosan carrying 0.3% green-synthesised ZnO gave 12% loss against 21% for uncoated banana at 28 °C, but over 20 days [17]; ZnO/Ag₂O on banana at 25 °C gave weight losses ranging from 14.07 to 17.95% across the control and all coated treatments, with no significant difference between them, over 12 days [18]; alginate–ZnO on mango at 13 °C gave 4.30% against 5.75% over 28 days [30]. The decay figures compare more favourably: 10% at day 14 here against 44.4% at day 12 for ZnO/Ag₂O-treated banana, where untreated fruit reached 100% [18]. The organic acid trend also merits comment. Acidity rose throughout the present trial, whereas an alginate hydrogel coating on banana

The two observations are not in conflict: organic acids in banana rise to a maximum during ripening and decline

thereafter as they are converted to sugars [33], so fruit here were still on the ascending limb at day 14 while those in the cited work had passed the peak.

4.4. Limitations

Five limitations qualify these conclusions and should be addressed before the formulation is developed further. First, nanoparticles to the storage result cannot be separated from that of the chitosan–alginate matrix, and the fruit data therefore support the coating as a whole rather than ZnO specifically. Second, statistical treatment was not applied: replicate numbers for the mechanical and colour data are not reported, one fruit per treatment was sampled destructively at each interval for firmness and acidity, and no significance testing was performed, so differences of a few percent — including the weight-loss difference — should be regarded as indicative. Third, inhibition zone diameters were not quantified, and no antifungal assay was run. Fourth, the ZnO loading of 7.8 wt% of film solids exceeds the range for which migration data exist: food-simulant work against the Regulation (EU) No 10/2011 limit of 5 mg zinc kg⁻¹ recommends a maximum of 1% ZnO nanoparticles for acidic food contact [34], and although EFSA has concluded that zinc oxide does not migrate in nanoform, so that the assessment applies to soluble ionic zinc [35], zinc migration from this coating into banana pulp was not measured. Fifth, the trial ended at day 14 with coated fruit still green; whether such fruit subsequently ripen to acceptable eating quality was not determined, and no sensory evaluation was carried out.

5. Conclusions

Nano-ZnO was obtained by co-precipitation as phase-pure hexagonal wurtzite with a mean particle size of approximately 20 nm, a BET surface area of 36.74 m² g⁻¹, a pore volume of 0.133 cm³ g⁻¹ and a mean pore diameter of 6.75 nm. In a chitosan–alginate matrix plasticised with 15% w/w alginate and 2.5% w/w glycerol, nano-ZnO showed a sharp compositional optimum at 10% w/w relative to chitosan: tensile strength technologies, and strategies to reduce losses along the supply chain of banana: A review," Trends Food Sci. Technol., vol. 134, pp. 177–191, 2023, doi: 10.1016/j.tifs.2023.03.003.

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only CA15Z10 was applied to fruit; without a ZnO-free CA15-coated arm, the contribution of the

reached 38.75 ± 0.76 MPa and elongation at break 12.86 ± 3.96%, against 9.29 ± 1.16 MPa and 9.38 ± 2.79% for the unfilled blend, with both properties falling at 15% as agglomeration set in. The optimum lies well above the 1–3 wt% typically reported for chitosan-only matrices, which is attributed to the looser, carboxylate-rich network created by alginate. All ZnO-containing films inhibited *S. aureus*; none inhibited *E. coli* under the conditions used.

Applied as a dip coating to banana stored at 30 °C and 80% relative humidity, the optimised formulation held decay incidence to 10% at day 14 against 50% for uncoated fruit, retained measurable pulp firmness at day 11, kept the peel green (a^* 5.8 versus 8.5) and slowed the accumulation of total organic acid (0.55% versus 0.67%), while reducing weight loss only from approximately 24% to 21%. The pattern indicates that the coating acts primarily by slowing ripening rather than by restricting transpiration. Because the fruit trial included no ZnO-free coated arm and no statistical replication, and because the zinc loading exceeds the range covered by existing migration guidance, the next steps should be a factorial trial separating matrix from filler, measurement of zinc migration into the pulp, antifungal testing against the postharvest pathogens of banana, and extension of the approach to other tropical fruits.

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