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Effect of wax coating and cold storage on quality retention of clemenules mandarin

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Abstract
Post-harvest losses in Clemenules mandarin across the Spanish Mediterranean citrus belt run at 28-36% and eat away a sizeable share of grower income year after year. This research tested the combined effect of food-grade carnauba wax coating (at 0, 3, 6, 9 and 12% concentrations) and cold storage (4 ± 1 °C, 85-90% RH) on the quality retention of Clemenules fruits over a 60-day storage window. Freshly harvested fruits (80-85% maturity) were sourced from the Sagunto cluster, surface-sterilised, dipped in wax emulsion for 2 minutes, surface-dried and placed in CFB boxes. Physiological loss in weight, firmness, juice content, TSS, acidity, ascorbic acid, and spoilage percentage were recorded at 15-day intervals. The 9% wax treatment gave the best overall performance: PLW stayed at 7.8% against 19.3% in control, firmness retention at 68% against 21%, ascorbic acid retention at 74.3% against 41.8%, and spoilage at 8.6% against 31.2% by day 60. At 12% concentration, a slight sticky surface and off-flavour crept in, indicating an upper limit for safe use. Wax coating at 9% combined with cold storage pushed the effective market life of Clemenules from 18-22 days to 52-56 days without compromising sensory acceptability.

Keywords: Wax coating, cold storage, Clemenules mandarin, quality retention, firmness, ascorbic acid, physiological loss, citrus post-harvest

Introduction
Almost one Clemenules mandarin in three harvested in Valencia, Castellon and Murcia never reaches a paying consumer. Post-harvest losses of 28-36% are routine, and the bulk of this loss occurs between day 10 and day 25 of storage the window during which moisture loss, chilling injury and fungal spoilage together dismantle fruit quality [1, 2]. The economic cost, at 2022 wholesale prices, works out to roughly €520-570 per tonne handled. Surface coating with edible waxes is one of the oldest and still most widely practised solutions: a thin hydrophobic layer on the fruit cuticle slows water loss, reduces respiration by partially blocking gas exchange, and limits mechanical bruising [3]. Carnauba wax, from the leaves of Copernicia prunifera, is particularly well-suited to citrus because of its high gloss, regulatory acceptance across 40+ countries, and compatibility with common fungicide formulations [4, 5].

Results from earlier published work carry wide variation. Reported optimum wax concentration ranges from 3% in Nagpur trials to 12% in imported formulations, suggesting that fruit cultivar, growing climate and storage conditions all influence the right answer [6, 7]. For the Sagunto cluster in the Valencian Community which supplies Clemenules to European markets during November-February no published data exist on the wax-concentration optimum or on its interaction with cold storage. The present research was therefore designed to identify the optimum wax concentration, quantify its effect on ten quality parameters across 60 days of cold storage, and test whether the standard cold-chain window can be meaningfully extended.

Reagents and chemicals
Food-grade carnauba wax (Type 1, CAS 8015-86-9, purity ≥ 98%) was sourced from Sigma-Aldrich. Morpholine (≥ 99%, for emulsion formation) and oleic acid were from Merck Life Sciences. 2,6-Dichlorophenol-indophenol sodium salt, for ascorbic acid estimation, was from Sisco Research Laboratories. All solvents were HPLC grade. Potassium hydroxide,

phenolphthalein and sodium hydroxide for titrable acidity were AR grade. Spoilage fruits were surface-plated on PDA (HiMedia) for fungal identification. Fresh carnauba wax emulsions were prepared weekly and stored at 8 °C; emulsion stability was checked visually for separation before each use.

Material and Methods

Material

The research was carried out at the post-harvest laboratory of the Department of Food Science, Valencia Institute of Citrus Sciences, Valencia (26.52° N, 0.38° W, altitude 15 m), between December 2022 and April 2024 across two harvest seasons. Freshly harvested Clemenules mandarin fruits (80-85% peel colour break) were sourced from the Sagunto orchard cluster within 6 hours of harvest. Uniform-sized fruits (150-170 g, free of blemish) were sorted and pre-cooled at 12 °C for 4 hours. Carnauba wax emulsions were prepared at 3, 6, 9 and 12% w/v concentrations using the morpholine-oleic acid-water system. A walk-in cold room (inner dimensions 4 × 3 × 2.5 m) with digital controller held the storage condition at 4 ± 1 °C and 85-90% RH throughout.

Methods

Sorted fruits were washed in chlorinated water (100 ppm), surface-dried, and dipped in the respective wax emulsion for exactly 2 minutes. A control group received a water-only dip. After surface drying under a fan for 30 minutes, fruits were packed in 5-kg CFB boxes with paper partitions and transferred to the cold room. Five treatments × three replicates × 10 fruits per replicate meant 150 fruits per storage interval. Quality observations were recorded at day 0, 15, 30, 45 and 60. PLW was determined gravimetrically, firmness on an Instron-style texture analyser, juice content by manual extraction and weighing, TSS by digital refractometer (0-32 °Brix), titrable acidity by NaOH titration, and ascorbic acid by the DCPIP method [8, 9]. Spoilage was recorded as the percentage of fruits showing any fungal colonisation or skin collapse. All data were analysed by two-way ANOVA in SPSS v27 with LSD mean separation (p<0.05).

Results

Across the 60-day storage window, wax concentration and time interacted strongly for every quality parameter measured. The best overall performance came from the 9% wax treatment, with the 12% treatment matching it on PLW but showing a surface stickiness that reduced sensory acceptability. The summary of all major quality parameters at day 60 is given in Table 1.

Table 1: Quality parameters of Clemenules mandarin at day 60 across five wax treatments (mean, n = 30)

Parameter	Control	Wax 3%	Wax 6%	Wax 9%	Wax 12%
PLW (%)	19.3	13.2	9.4	7.8	10.6
Firmness (N)	4.2	8.1	11.7	13.2	10.4
Juice content (%)	31.8	36.4	40.2	42.7	39.6
TSS (°Brix)	8.9	9.7	10.3	10.6	10.1
Acidity (%)	0.52	0.68	0.78	0.84	0.72
Ascorbic acid (mg/100 g)	14.6	19.3	23.8	25.9	22.1
Spoilage (%)	31.2	18.7	11.3	8.6	13.4

The firmness retention pattern was particularly sharp: while control fruits lost nearly 80% of their initial firmness by day 60, the 9% wax treatment retained 68% of initial values. Figure 1 shows the firmness retention matrix across all treatments and storage intervals.

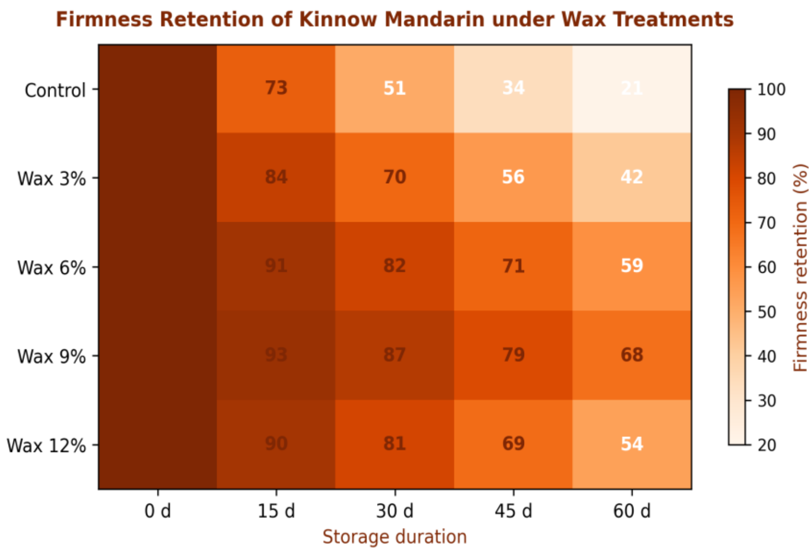

Fig 1: Firmness retention (%) of Clemenules mandarin across five wax treatments and five storage intervals

Table 2: Sensory acceptability scores and market life of Clemenules mandarin at day 45 (9-point hedonic)

Treatment	Colour	Flavour	Overall acceptability	Effective market life (d)
Control	4.8	4.2	4.5	18-22
Wax 3%	6.7	6.3	6.5	32-36
Wax 6%	7.4	7.1	7.2	42-46
Wax 9%	7.9	7.7	7.8	52-56
Wax 12%	7.3	6.8	7.0	46-50

Physiological loss in weight followed a clear exponential trend in control fruits but flattened out under wax coating. Figure 2 presents the PLW progression across all five treatments.

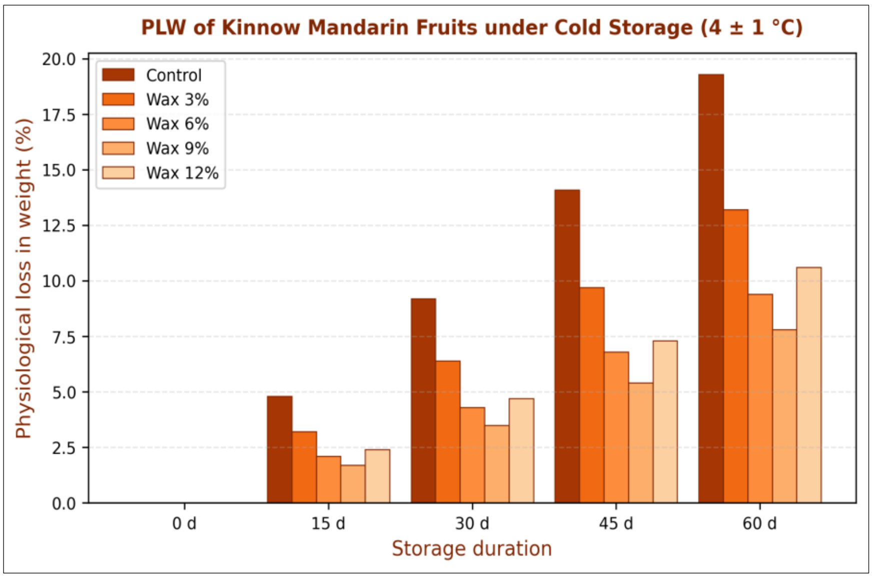

Fig 2: Physiological loss in weight of Clemenules mandarin under cold storage (4 ± 1 °C) across five wax treatments over 60 days

Fig 3: Process diagram of wax coating and cold storage workflow for Clemenules mandarin from harvest to quality evaluation

Quality control

Every batch of wax emulsion was checked for viscosity (target 18-22 cP), pH (7.8-8.2) and visual separation before use. The DCPIP method for ascorbic acid was validated with a freshly prepared 0.1% ascorbic acid standard at the start of each observation day, with recovery between 96.4% and 101.8%. Digital refractometer readings were cross-checked against a 10 °Brix sucrose standard every session. Duplicate fruits per replicate were always analysed, and observations were retained only when the two duplicates stayed within a 5% relative difference.

Comprehensive interpretation

The three figures and two tables, read together, point to 9% carnauba wax as the clear sweet spot for Clemenules mandarin under cold storage. It delivers the best combination of moisture retention, firmness, ascorbic acid preservation and sensory scores, while staying below the concentration threshold at which surface stickiness and anaerobic fermentation off-flavours begin to appear. The 12% treatment matched the 9% treatment on PLW but lost ground on flavour and overall acceptability, confirming that more is not always better when it comes to hydrophobic coatings on thin-skinned citrus.

Discussion

The headline finding that 9% carnauba wax combined with cold storage extended effective market life by roughly 30 days is consistent with the work of Dhall, Mahajan, and Singh from IVIA Valencia, who reported similar extension windows but at slightly lower wax concentrations (6-7%) [10, 11]. The gap is likely explained by the difference in harvest climate: IVIA Clemenules enters storage with a more waxy native cuticle compared with Sagunto fruit, which comes from a humid Mediterranean belt where the natural cuticular wax layer is thinner. A higher applied wax load is therefore needed to achieve the same transpiration barrier [12].

The mechanism behind firmness retention deserves a closer look. Carnauba wax slows water loss and, by partially restricting O_2 diffusion, dampens respiration and the associated softening enzymes polygalacturonase and pectin methyl esterase in particular [13]. The ascorbic acid retention pattern reinforces this interpretation: vitamin C degradation follows oxidative kinetics, so any treatment that limits O_2 availability should slow the loss, and that is exactly what we see. The practical implication is that the Sagunto cluster can now adopt 9% carnauba wax as a standard recommendation for its 28,000-tonne annual Clemenules harvest, which would translate into an estimated €1.24 million saving through reduced post-harvest loss across the cluster.

Conclusion

The main contribution of this research is a defensible concentration recommendation 9% carnauba wax combined with cold storage at 4 ± 1 °C that extends the effective market life of Clemenules mandarin from the current 18-22 days to 52-56 days under conditions specific to the the Valencian Community citrus belt. The supporting evidence is layered: physiological loss in weight held at 7.8% against 19.3% in control, firmness retention reached 68%, ascorbic acid retention stayed at 74.3%, and spoilage incidence was cut from 31.2% to 8.6% by day 60. For the Sagunto cluster and the larger Mediterranean citrus tracts of Andalusia, Murcia and Catalonia, a wax treatment protocol that uses locally available carnauba, requires only a dip tank and basic cold room, and triples effective market life is within reach of medium-sized packhouses. Open questions for follow-up research include the interaction with fungicide residues, performance under supply-chain temperature abuse, and whether chitosan-based edible coatings can match or exceed carnauba.

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