SYNERGISTIC EFFECT OF SALTS AND CELLULOSE BASED COATING ON SHELF LIFE OF KINNOW (CITRUS RETICULATA BLANCO)

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ABSTRACT

Kinnow (Citrus reticulata Blanco) is achieving core consideration globally owing to its nutrition and high juice content. In Pakistan, kinnow export is limited due to inadequate postharvest handling and lack of appropriate storage practices. Considering these issues, the present study was designed to evaluate the effect of hydroxy-propyl-methylcellulose (HPMC) coating in combination with CaCl₂ and MgSO₄ on shelf life extension of kinnow. Fruits were treated with different levels of CaCl₂ and MgSO₄ followed by HPMC coating (3 and 5%) and stored at 10°C with 80% relative humidity for 6 weeks. Fruits were analyzed for various physico-chemical parameters on weekly basis. During this study lower fruit firmness (0.24Nmm⁻²), loss in weight (0.64%) and ethylene production (0.039 µL·kg⁻¹·hr⁻¹) was observed in fruits treated with 1% CaCl₂ + 1% MgSO₄ + 5% HPMC (T₅) during storage of 42 days. Minimum chilling injury indices 0.22% and 0.61% were recorded in treatments T₄ and T₆, respectively. T₆ showed higher values of titerable acidity (0.29%) and ascorbic acid contents (39.82mg/100g). Minimum TSS (9.62°Brix) were found in fruits of T₆. Overall, T₆ showed significantly better results for various parameters as compared to all other treated and control fruits.

Key words: Citrus coating, Post-harvest, Hydroxy-propyl-methylcellulose, Physicochemical, shelf life.

INTRODUCTION

Among the tropical and subtropical countries, Pakistan is blessed with best agro-climatic as well as food topographic conditions which provides a promising environment for successful cultivation of various fruits. Today, Pakistan has great potential for citrus production which is grown in more than 52 countries of the world. Citrus species produced in Pakistan are well known due to their best quality and good sensory profile. Citrus occupies 29.55% area of total fruit cultivation in Pakistan. While land under kinnow cultivation accounts 60% of total citrus acreage (GOP, 2003-2004).

Sargodha district in Punjab shows more potential in citrus production, processing, storage and exports. Citrus fruits are highly nutritious due to organic acids and carotenoids (Ansari and Feridoon, 2008). In Pakistan magnitude of postharvest losses ranges from 35 to 40% (PHDEB, 2006) due to lack of appropriate pre and post-harvest management, insufficient packing houses, inappropriate transport infrastructure and limited cold chain facilities with traditionally slow marketing system. The quality of kinnow starts deteriorating right after harvesting if not properly managed.

In modern ages, coatings and edible films have received extraordinary attention for their use over synthetic coatings. Edible coatings retard dehydration, inhibit respiration, retain volatile compounds, improve texture and reduce micro-flora. Coatings and edible films can be categorized in to three different types on the basis of their components i.e. composites, lipids and hydrocolloids. Different types of flavors, pigments, antioxidants, antimicrobial agents, firming agents and vitamins can be incorporated into edible coatings to improve and modify their functional properties. In the past few years, polysaccharides have been used as coating material for different fruits. These polymers proved to be having advantages due to their low cost, availability and biodegradability. Many derivatives of cellulose like carboxy-methylcellulose, methyl cellulose and hydroxy-propyl cellulose are produced commercially (Olivas and Barbosa-Canovas, 2005).

Cellulose based wax coating like (HPMC) is widely used on different species of the fruits, due to its non-toxic effects. Cellulose based compounds are water soluble and semi synthetic in nature which improve color and maintain fruit firmness. They are usually used as thickening agents, binders, film formers, water retention agents, protective colloids and emulsifiers. Cellulose coating with emulsions of salts and plant material showed prominent results to extend the shelf life of fruit (Adetunji et al. 2012).

Different salts such as KCl, CaCl₂ and Na₂HCO₃ along with cellulose based coating are supportive to maintain firmness and reduce physiological weight loss. Decrease in weight loss of oranges was observed when treated with emulsion of HPMC, beeswax and shellac along with calcium chloride. In various experiments, MgSO₄ was mostly used for tomato, cucumber and carrot but 1st time in this project it was used on citrus along with HPMC and CaCl₂ to extend the shelf life. The aim of the present study was to find out the effect of cellulose based coating assimilated with MgSO₄ and CaCl₂ on kinnow to extend the shelf life and improve quality parameters.
MATERIALS AND METHODS

Raw material: Kinnow mandarin (Citrus reticulata Blanco) used in this study were harvested in mid-February from the orchard of University of Agriculture, Faisalabad. Mature fruits were harvested from four directions of the plant canopy to get homogeneous fruit samples. Kinnows were harvested at commercially mature stage when the upper part of the fruit skin showed glossy appearance with soft skin which was easy to peel off. Harvesting was performed by clipping the fruit stalk just above the point of attachment to the fruit. After that kinnow fruits were placed in plastic buckets (100 fruits per bucket) and transported to the laboratory of National Institute of Food Science and Technology University of Agriculture, Faisalabad. Cellulose based coating HPMC (Sigma-Aldrich) and salts of calcium and magnesium (Fluka) were used with different concentrations.

Preparation of wax emulsion: Wax emulsion was prepared by dispersing HPMC in hot water at 80°C with glycerol (2 parts HPMC: 1 part glycerol) which is used as a plasticizer.

Pretreatment: Before wax coating kinnow fruits were sorted and graded to obtain uniform size for further postharvest processing. Then, kinnow fruits were disinfected by immersion in diluted bleach (0.1% sodium hypochlorite) for 3-5 minutes followed by rinsing with tap water to remove dirt, spray residues and extraneous matter from the field. Thereafter, Kinnows were placed on paper towels and allowed to dry in air at room temperature (25°C) under fan at moderate speed.

Treatment layout: Fruits were randomly divided into seven sets, each contained 63 fruits. Each set was treated by different treatments including: uncoated/control fruits (T0), fruits coated with 2% CaCl2 + 3% HPMC (T1), 2% CaCl2 + 5% HPMC (T2), 2% MgSO4 + 3% HPMC (T3), 2% MgSO4 + 5% HPMC (T4), 1% CaCl2 +1% MgSO4 + 3% HPMC (T5) and 1% CaCl2 +1% MgSO4 + 5% HPMC (T6). The fruits were dipped in above mentioned chemicals for 3 minutes at room temperature. Afterwards, they were dried at room temperature with natural air convection for 2–3 hours. After drying both, treated and untreated kinnow fruits were placed in small pre-cleaned perforated plastic baskets and stored at 10°C with 80% relative humidity in a chamber (Memmert ICH-260-C) equipped with automatic temperature and relative humidity control system.

Quality analysis: Physical properties like firmness and juice weight were determined by adopting the standard methods. Data concerning to fruit firmness was recorded with the help of digital penetrometer (53205 Digital Fruit Firmness Tester, Agri. Measurement Instrument, Turoni, Italy). Juice weight was obtained by subtracting peel weight, rag weight and seed weight from total fruit weight. Physiological loss in weight was expressed as percentage decrease in fruit weight as described by Thakur et al. (2002). Incidence of chilling injury was determined as percentage of the number of decayed fruits from the initial number after an indicated period of storage. Rates of ethylene production were measured by the static system. A known quantity of fruits per treatment was weighed and sealed with ethylene meter (Model # drager PAC-III) in 3L container for 1 hour. After 1 hour the meter was removed from container and the reading (C2H4) was displayed in ppm on screen. The rate of ethylene production was calculated in µL·kg⁻¹·h⁻¹.

Biochemical quality characteristics of the treated fruits were estimated from the juice samples taken from each treatment. The quality attributes such as total soluble solids, titratable acidity and ascorbic acid were determined by using standard procedures of AOAC (2006). Total soluble solids (TSS) were examined by using a portable hand Refractrometer (Model BS Eclipse 3-45) and the results were expressed as °Brix. Titratable acidity (TA) was measured by titration of juice sample with 0.1 N NaOH, and expressed as percentage of citric acid/100 ml of juice. Ascorbic acid was measured by 2, 6- dichlorophenol indophenol titration solution to end point, and expressed as ascorbic acid in mg per 100 mL of juice.

Statistical analysis: The obtained data for each parameter were analyzed through two way analysis of variance technique under Completely Randomized Design with factorial arrangements. Level of significance was set at P≤0.05 as described by Steel et al. (1997).

RESULTS AND DISCUSSION

Physical analysis: Means regarding the effect of physical parameters (firmness and juice weight) showed significant differences at P≤0.05 for treatment, storage and their interaction. Fruits treated with 1% CaCl2 + 1% MgSO4 + 5% HPMC (T6) showed significantly high fruit firmness (1.2Nm²) as compared to all other treatments. Lower fruit firmness was observed in untreated fruits (0.22Nm²). Firmness decreased significantly with the passage of time. Before storage it was 1.2Nm² and after 42 days it decreased up to 0.24Nm² in case of T0 as shown in Figure 1. According to results T6 showed significantly higher juice weight (44.18%) as compared to T0 (38.56%). Maximum juice weight 42.76% was observed in initial day of storage but after 42 days it reduced to 40.14% under applied storage conditions as depicted in Figure 2. With previous findings firmness of kinnow was slightly better with HPMC compared to uncoated samples and that difference could be due to cultivar and storage conditions (Contreras-Olivae et al. 2012). Alteration in firmness and juice weight were less in those fruits which were treated with HPMC + Moringa.
as compared to untreated (Adetunji et al. 2012). Generally, firmness of citrus decreases during the ripening processes. That is altered due to breakdown of starch molecules to form sugars in the presence of hydrolytic enzymes. Abdel-Aziz et al. (2000) indicated same results for weight loss of juice in controlled temperature and humidity conditions. A decline in juice weight was attributed to transpiration rate and dehydration during storage period.

**Physiological analysis:** Statistical analysis of physiological parameters showed that treatments, storage and their interaction significantly affected loss in weight, chilling injury and ethylene production. Minimum loss in weight was recorded (0.64%) in T₆, whereas maximum weight loss was observed in T₀ (2.46%). The weight loss among treated samples ranged from 1.12% to 3.25%. However, with the storage period, T₀ exhibited minimum loss in weight as compared to rest of the treatments. Lowest value of ethylene was observed in fruits of T₀ (0.039 µL·kg⁻¹·h⁻¹) as compared to untreated fruits which shows maximum ethylene production (0.096 µL·kg⁻¹·h⁻¹).

Before storage kinnow mandarin produced 0.028 µL·kg⁻¹·h⁻¹ ethylene, whereas after 42 days it was 0.114 µL·kg⁻¹·h⁻¹ as shown in Figure 3. Higher index of chilling injury (3.59%) was noted in fruits of T₀, whereas T₆ showed lower chilling injury (0.61%) as depicted in Figure 4. The fruits which were analyzed after 42 days storage showed higher index of chilling injury (3.63%) than the fruits being analyzed after 35, 28, 21, 14 and 7 days during storage interval with chilling injury indexes 2.88, 2.38, 2.08, 1.35 and 1.28%, respectively. Fruit weight is an important parameter for fruit quality. According to Silvia et al. (2009), Valencia oranges treated with HPMC-lipid emulsion along with sodium benzoate stored for 60 days at 5°C significantly reduced the weight loss, which indicated the effectiveness of these coatings as a moisture barrier as shown in Figure 5. At the end of this storage period, weight losses on all samples were around 3.9-4.4%. Weight loss in orange fruit during ripening is due to both transpiration and respiration. Transpiration is associated with water vapor pressure of the surrounding atmosphere and the fruit surface. Weight loss contributed by respiration is a result of carbon atoms, in the form of carbon dioxide molecules, leaving the fruits. Ethylene production also increased the ripening of fruits which ultimately decrease the shelf life of fruits. Generally, different types of wax materials especially cellulose based wax was helpful for the reduction in rate of respiration and ethylene production in kinnow during storage period because ethylene production in different wax coated fruits was found 90% lower than uncoated ones. Most physiological and biochemical changes involved during ripening of fruits are softening, change of color from green to yellow, loss of acidity, conversion of starch to sugars, formation of cuticular waxes, synthesis of aromatic compounds, changes in flavor, color and texture of fruits (Kays and Paull, 2004).

Different factors like type of cultivar, storage conditions and postharvest treatments may affect the development of chilling injury in oranges and mandarins. Similarly, Perez et al. (2002) concluded that development of chilling injury in the Fortune mandarins took place when stored for specific duration (> 60 days) at below 8°C temperature. According to some scientists, chilling injury was slight in initial 30 days of experimentation while, severe chilling injury developed after 5 weeks of storage.

**Biochemical analysis:** During storage data related to biochemical parameters such as total soluble solids, titratable acidity and ascorbic acid contents showed significant results for storage and treatments as depicted in Table 1 & 2. T₀ fruits showed minimum TSS (9.62°Brix), whereas maximum TSS were observed in T₀ (11.34°Brix) during entire duration. As the storage time increased TSS also increased, maximum TSS (11.80°Brix) were observed after 35 days of interval. High titerable acidity was observed in fruits of T₆ (0.29%) as compared to all other treatments. Fruits of controlled treatment showed minimum acidity (0.22%). After 42 days of storage, acidity level decreased from 0.34 to 0.15%. Maximum vitamin C (39.82 mg/100g) was recorded in fruits of T₀ as compared to controlled treatment which showed minimum (22.17mg/100g). Vitamin C decreased with the passage of time, in initial day maximum vitamin C was 40.27 (mg/100g) and after 42 days of storage, it was 37.84 (mg/100g).

Above mentioned results depicted that increase in TSS was due to the increase in rate of respiration and hydrolysis of starch in cold chamber. Thakur et al. (2002) also found similar results in kinnow mandarin during cold storage after 40 days. This effect was opposed by Babalar et al. (2007) who concluded a rapid increased in TSS in strawberry fruits. A correlation of inversely proportional was found between TSS and TA during storage. TA was higher in those fruits which are coated with emulsion of CMC (1.1%) along with soybean and sodium oleate stored for 27 days at 25°C and 75% relative humidity with storage time (Hasan, 2004). According to some other observations, decrease in titerable acidity may be due to decrease in oxygen transfer rate from the external medium to fruit, as the CMC amount in the emulsions increased. When oxygen transfer rate decreased, the glucose was subjected to anaerobic fermentation and produced alcohol, which decreased titerable acidity. Coated fruit significantly maintained vitamin C contents due to its binding layer which plays an important role to prevent the oxidation process in fruit. Vitamin C decreased with storage time when stored at 4°C as reported by Keditsu et al. (2003). The decrease in ascorbic acid level was related with a reduced capability.
of preventing oxidative damage. During storage period, higher level of vitamin C was observed in fruits coated with HPMC+ moringa extract than in control samples, similar to that was reported by Dang et al. (2010), who found that the content of vitamin C of coated fruits was higher than that of the control. Moreover, Randhawa et al. 2014 analyzed various organic acids from Pakistani citrus species and observed the effect of refrigeration storage on their deterioration. They measured degradation percentage of different organic acids and depicted that vitamin C was highest (51.43±2.06 mg/100ml) in kinnow and decreased from 11 to 15 % during entire storage. Hence different factors responsible for oxidation of ascorbic acid include exposure to oxygen, metals, light, heat, and alkaline pH (Chitravathi et al. 2014).

![Figure 1](image1.png)

**Figure 1.** Effect of Ca and Mg salts along with cellulose based coating on Firmness (Nm²) under controlled storage

![Figure 2](image2.png)

**Figure 2.** Effect of Ca and Mg salts along with cellulose based coating on Juice weight (%) under controlled storage.
Figure 3. Effect of Ca and Mg salts along with cellulose based coating on Ethylene gas production (μL•kg⁻¹•h⁻¹) under controlled storage.

Figure 4. Effect of Ca and Mg salts along with cellulose based coating on Chilling injury (%) under controlled storage.

Figure 5. Effect of Ca and Mg salts along with cellulose based coating on % Loss of Weight (%) under controlled storage.
Table 1. Effect of Ca and Mg salts along with cellulose based coating on biochemical parameters in different days of storage.

<table>
<thead>
<tr>
<th>Storage (Days)</th>
<th>Total soluble solids (Brix)</th>
<th>Titerable acidity (%)</th>
<th>Vitamin C (mg/100g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>9.607&lt;sup&gt;G&lt;/sup&gt;</td>
<td>0.345&lt;sup&gt;A&lt;/sup&gt;</td>
<td>40.271&lt;sup&gt;A&lt;/sup&gt;</td>
</tr>
<tr>
<td>07</td>
<td>9.995&lt;sup&gt;F&lt;/sup&gt;</td>
<td>0.316&lt;sup&gt;B&lt;/sup&gt;</td>
<td>39.733&lt;sup&gt;B&lt;/sup&gt;</td>
</tr>
<tr>
<td>14</td>
<td>10.427&lt;sup&gt;E&lt;/sup&gt;</td>
<td>0.282&lt;sup&gt;C&lt;/sup&gt;</td>
<td>39.338&lt;sup&gt;BC&lt;/sup&gt;</td>
</tr>
<tr>
<td>21</td>
<td>10.871&lt;sup&gt;D&lt;/sup&gt;</td>
<td>0.252&lt;sup&gt;D&lt;/sup&gt;</td>
<td>38.995&lt;sup&gt;CD&lt;/sup&gt;</td>
</tr>
<tr>
<td>28</td>
<td>11.331&lt;sup&gt;C&lt;/sup&gt;</td>
<td>0.224&lt;sup&gt;E&lt;/sup&gt;</td>
<td>38.614&lt;sup&gt;DE&lt;/sup&gt;</td>
</tr>
<tr>
<td>35</td>
<td>11.809&lt;sup&gt;A&lt;/sup&gt;</td>
<td>0.195&lt;sup&gt;F&lt;/sup&gt;</td>
<td>38.195&lt;sup&gt;EF&lt;/sup&gt;</td>
</tr>
<tr>
<td>42</td>
<td>11.693&lt;sup&gt;B&lt;/sup&gt;</td>
<td>0.159&lt;sup&gt;G&lt;/sup&gt;</td>
<td>37.848&lt;sup&gt;F&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

Table 2. Effect of Ca and Mg salts along with cellulose based coating on biochemical parameters in different treatments during storage.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Total soluble solids (Brix)</th>
<th>Titerable acidity (%)</th>
<th>Vitamin C (mg/100g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T&lt;sub&gt;0&lt;/sub&gt;</td>
<td>11.34&lt;sup&gt;A&lt;/sup&gt;</td>
<td>0.222&lt;sup&gt;E&lt;/sup&gt;</td>
<td>22.17&lt;sup&gt;E&lt;/sup&gt;</td>
</tr>
<tr>
<td>T&lt;sub&gt;1&lt;/sub&gt;</td>
<td>10.65&lt;sup&gt;E&lt;/sup&gt;</td>
<td>0.230&lt;sup&gt;E&lt;/sup&gt;</td>
<td>25.79&lt;sup&gt;CD&lt;/sup&gt;</td>
</tr>
<tr>
<td>T&lt;sub&gt;2&lt;/sub&gt;</td>
<td>11.18&lt;sup&gt;B&lt;/sup&gt;</td>
<td>0.285&lt;sup&gt;B&lt;/sup&gt;</td>
<td>26.67&lt;sup&gt;D&lt;/sup&gt;</td>
</tr>
<tr>
<td>T&lt;sub&gt;3&lt;/sub&gt;</td>
<td>10.99&lt;sup&gt;CD&lt;/sup&gt;</td>
<td>0.263&lt;sup&gt;C&lt;/sup&gt;</td>
<td>27.29&lt;sup&gt;B&lt;/sup&gt;</td>
</tr>
<tr>
<td>T&lt;sub&gt;4&lt;/sub&gt;</td>
<td>11.04&lt;sup&gt;E&lt;/sup&gt;</td>
<td>0.229&lt;sup&gt;E&lt;/sup&gt;</td>
<td>28.57&lt;sup&gt;BC&lt;/sup&gt;</td>
</tr>
<tr>
<td>T&lt;sub&gt;5&lt;/sub&gt;</td>
<td>10.91&lt;sup&gt;D&lt;/sup&gt;</td>
<td>0.250&lt;sup&gt;D&lt;/sup&gt;</td>
<td>27.10&lt;sup&gt;BD&lt;/sup&gt;</td>
</tr>
<tr>
<td>T&lt;sub&gt;6&lt;/sub&gt;</td>
<td>9.62&lt;sup&gt;F&lt;/sup&gt;</td>
<td>0.295&lt;sup&gt;A&lt;/sup&gt;</td>
<td>39.829&lt;sup&gt;A&lt;/sup&gt;</td>
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</table>

Conclusions: This study revealed the comparative analysis on the effects of cellulose based coating with salts concentration under controlled storage condition on the quality and the shelf-life of Citrus reticulata blanco. Results depicted that the combination, where 5% cellulose coating along with 1% calcium and magnesium salts were used, found best for maintaining the quality and extending the shelf-life of kinnow over other treatments or control. Hence, coated samples exhibited the least physiological loss in weight, juice % and less chilling injury with higher tiritable acidity, ascorbic acid and lower total soluble solids. Moreover, fruit firmness was also maintained inhibiting ethylene production. Keeping in view the results, it can be concluded that the treatment T<sub>6</sub> was the most effective in maintaining the quality characteristics and extending the shelf-life of kinnow.

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