Effect of waxing on the quality of *limau madu* (*Citrus reticulate*) during storage at 25 and 10 °C

[Kesan pelilinan pada kualiti limau madu (*Citrus reticulate*) semasa penyimpanan pada suhu 25 dan 10 °C]

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Key words: waxing, physical, chemical and quality changes, shelf life, Citrus reticulate

Abstract

Changes in quality of limau madu (Citrus reticulate) treated with 1.0% and 1.5% Semperfresh wax were observed during storage at 25 °C with relative humidity 65–70% and at 10 °C with relative humidity 85–90%. Untreated fruits were used as control. Fruits stored at 25 °C were evaluated on alternate days whereas fruits stored at 10 °C were evaluated at weekly intervals. Samples were analysed to determine changes in physical and chemical attributes, and sensory evaluation. Overall results indicated that fruits stored at 25 °C were still acceptable until day 8 with treated fruits having a better quality and shiny skin. Higher weight loss was observed in control fruits (2.5-6.5%) as compared to the wax treated fruits (0.5-1.8%). However, no significant difference was shown between fruits treated with 1.0% and 1.5% wax treatment. Similar observation was noted in fruits stored at 10 °C for 4 weeks. Weight loss of wax treated fruits was lower (0.03-1.5%) as compared to the control fruits (0.5-2.5%). No significant difference in colour change as shown by the L and hue values between 1.0% and 1.5% wax treatment. Slight yellow colour formation was observed in the control fruits stored at 10 °C after 3 weeks of storage. However, no significant difference was observed in the the different wax treatment until the end of 4 weeks. The TSS values of the control fruits decreased significantly after 8 days of storage at 25 °C. However no significant difference was observed between 1.0% and 1.5% wax treatment. The TSS values of both treated and control fruits were quite stable (12.8-12.6%) during the 4 weeks of storage at 10 °C. Significantly lower pH values were observed in control fruits both during storage at 25 °C and 10 °C. Semperfresh treatment did not influence the taste, odour and overall acceptability of both fruits stored at 25 °C and 10 °C.

Introduction

Waxing can provide an additional protective coating for fresh products and can also give the same effect as modified atmosphere storage in modifying internal gas compositions (Park 1999). The application of wax as a protective coating on citrus fruits is not new. The Chinese applied wax coating to oranges and lemons in the 12th and 13th centuries (Park 1999). Citrus fruits, once harvested, are subject to desiccation and progressive deterioration which greatly affect flavour, aroma and appearance of the produce.

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Currently, waxing has become an important component in the storage and marketing of citrus fruits because of its ability to reduce moisture loss, delay softening and improve appearance. Postharvest research on citrus indicates that the application of wax does not produce offflavour while reducing moisture loss. The application of wax in apple also does not produce off-flavour and retard the ripening process (Ben-Yeoshoshua et al. 1993). Application of wax in banana helps to delay ripening and chlorophyll loss while in mango it inhibits the development of yellow peel colour and delays ripening changes (Krishnamurthy 1989).

Although a lot of work had been reported on the effect of waxing in extending the storage life of fruits, there is little information and quantitative data concerning the effect of wax on limau *madu* (*Citrus reticulate*). Locally grown *limau madu* has a typical green colour upon maturation and has already been accepted at the markets as an edible green citrus. However, chlorophyll degradation may occur with change in the environmental conditions, thus a tinge of yellow colour may scatter over the fruit skin of limau madu. Formation of full yellow colour is not normal unless the fruits are grown in the highlands. The overall appearance of fruits will be affected if the intensity of the yellow colour is not uniform.

Suitable postharvest treatment is needed to avoid the development of yellow colour during storage and market display so as to deliver consistent products to the markets. Application of calcium in the form of calcium chloride at a concentration of 1% and 2% on *limau madu* stored at 25 °C, RH 65–70% did not retard the yellow colour development. The normal ripening processes progressively continued with no marked difference in the physical and chemical changes between the control and calcium treated fruits. After 1 week storage, the yellow colour slowly developed which progressively extended to almost 50% after 14 days (Latifah et al. 2004a). The effectiveness of calcium treatments were reported on other fruits such as apples and pear (Yuen 1993). The use of stretch film wrapping appeared to give significant impact in controlling the weight loss and yellow colour development as observed in fruits stored at 25 °C and RH 65–70% (Latifah et al. 2003). Similar results were also observed when the fruits were stored at 10 °C at RH 85–90% for 4 weeks (Latifah et al. 2004b).

The success of waxing fruits depends mainly on the coating types that is appropriate for specific produce. If coating is too thick, detrimental effects can be expected due to the amount of internal oxygen concentration below a desirable level. These will restrict the respiration rate of the fruits and allow the accumulation of carbon dioxide concentration above a critical tolerable level which causes fruit injury as reported by Smith et al. (1987) on apples (cv. Cox's orange Pippin) coated with sucrose fatty acid ester.

The purpose of this study was to observe the effect of waxing on *limau madu* during storage at 25 °C and 10 °C. Temperature of 25 °C is chosen to simulate the home environment whereas temperature of 10 °C to simulate the retail environment at the hypermarkets. Physical and chemical changes were monitored to relate with quality changes, shelf life and acceptability of the fruit.

Material and methods

Limau madu (Citrus reticulate) used in the study were harvested at commercial maturity from a private farm at Alor Limbat, Kuala Terengganu. *Limau madu* was considered commercially mature when the upper part of the fruit skin is soft with glossy appearance and easy to peel off (Ahmad Tarmizi and Pauziah 2005). Upon arrival at MARDI's laboratory in Serdang, Selangor, fruits were sorted and washed to remove the extraneous matter from the field. Only fruits having full green colour were used as samples. Fruits were treated with 1.0% and 1.5% wax, whereas untreated fruits were used as control. Fruits were packed in 24 corrugated fibreboard (CFB) boxes with net weight of 4 kg/box. Each CFB box contained 20 fruits.

Semperfresh is an edible wax emulsion composed of sucrose esters of fatty acids, sodium carboxymethyl cellulose and monodiglycerides of fatty acids. The wax was in liquid concentrate form (500 g/litre) and was diluted with distill water to obtain the desired concentration (1.0% and 1.5%). The diluted solution was stirred for 30–40 min occasionally to ensure homogenous solution was obtained. Waxing was applied by dipping the *limau madu* into the solution for 5 min.

The fruits were then stored for 8 days at 25 °C (RH 65–70%) and 4 weeks at 10 °C (RH 85–90%). Fruits stored at 25 °C were removed on alternate days while those stored at 10 °C were removed on weekly basis. At each observation date, three CFB boxes containing 60 fruits were evaluated.

Weight loss

The weight loss of *limau madu* was measured before and after storage. Weight loss was expressed as percentage. Ten fruits from each box/each treatment were specifically coded and used for weight loss measurement throughout the study. The weight loss recorded was based on the average of 30 fruits.

Skin colour

The skin colour of *limau madu* was evaluated using the colour meter Minolta Chroma meter (Model 300, Osaka, Japan) by measuring the values of L* (lightness), a* (greenness) and b* (yellowness). Hue angle, an indicator of ripeness is expressed as b/a. Ten fruits from each box/each treatment were specifically coded and used for colour measurement throughout the study. The skin colour recorded was based on the average of 30 fruits.

Chemical analysis

Limau madu was analysed for total soluble solids by using a refractometer (Model Atago Digital DBX-5). The pH value was determined using Orion digital pH meter (Model SA520). Ascorbic acid content was measured by titrating with 2,6 dichlorophenolindophenol (Ranganna 1977). For chemical analysis, the *limau madu* juice was obtained by squeezing the flesh in waring blender with three fruits representing a replicate. Chemical analysis was conducted in the three replicates.

Sensory evaluation

The sensory evaluations were carried out by a panel of 10 fixed panellists. The panel was requested to evaluate the fruit for colour, texture, taste, odour and overall acceptability using a 7-point hedonic scale (1 = very unacceptable, 2 = unacceptable, 3 = moderately unacceptable, 4 = neither good nor bad, 5 = moderately good, 6 = good and 7 = very good).

Statistical analysis

A complete randomized design was used for the experimental set up with two treatments (concentration of the waxing treatments, 1.0% and 1.5%) and three boxes for three replicates. Data were analysed statistically using analysis of variance (ANOVA) (Steel and Torrie 1980) and the differences among the means from three replicates were determined for significance at p < 0.05using Duncan multiple range test using the Statistical Analysis System (SAS Inst. 1990).

Results and discussion

The ability of the wax treatment, to form a film barrier to water vapour transmission, retain the surface moisture and alleviate water stress, appears to be the dominant factor in improving appearance and in reducing the weight loss of *limau madu*. These were well demonstrated especially in *limau madu* stored at 25 °C. Wax treated fruits had significantly (p > 0.05) lowered

weight loss (0.5-1.8%) as observed throughout the 8 days storage. No significant difference between 1.0% and 1.5% wax treatment (*Figure 1*). The weight loss of the control fruits increased significantly with duration of storage [2.2% (day 2), 4.5% (day 6) and 6.4% (day 8)].

Limau madu treated with wax treatment (1.0% and 1.5%) also exhibited significantly lower weight loss compared to the control fruits throughout the 4 weeks storage at 10 °C (*Figure 1*). The loss of weight progressively increased with duration of storage both in the control and treated fruits with marked increase in the control fruits from 0.3% during the first week to 2.5% after 4 weeks storage. Weight loss of the treated fruits was very low (0.03%) during week 1 and week 2 and increased to 1.2% after 4 weeks of storage (*Figure 1*).

Lower weight loss of both treated and control fruits at lower temperature

(10 °C) compared to the *limau madu* stored at 25 °C was presumably due to the effect of temperature on water vapour pressure potential as been reported on cherries (Yaman and Bayoindurh 2002).

Weight loss significantly affect the overall fruit appearance of the control fruits stored at 25 °C as sign of shriveling was noticeable on the skin after day 6 onwards. The loss of weight also resulted in loss of turgidity, accelerated ripening, and exacerbated fungus infection as reported by Sastry (1985). These symptoms were observed on the control fruits on day 8 onwards as fungus infection was seen in patches on the skin.

External green colour of *limau madu* decreased with prolonged storage as observed both at 25 °C and 10 °C. The decrease was indicated with the reduction in the value of the hue angle as shown in *Table 1*. The hue angle decreased to almost



Figure 1. The weight loss of **limau madu** with 1.0% and 1.5% wax treatment stored at 25 °C and 10 °C. Evaluation was conducted on day 2, 4, 6 and 8 for samples stored at 25 °C and week 1, 2, 3 and 4 for samples stored at 10 °C. Bars with the same letters within time are not significantly different at p < 0.05

85% at the end of 8 days storage at 25 °C. The hue angle decreased with prolonged storage due to the development of yellow patches on fruits with increase of storage time. These yellow patches of yellow probably had also influenced the decreasing trend in the L* value indicating of darkness of the skin colour of *limau madu (Table 1)*. However, no significant difference between the control and treated fruits. The result was similar to that reported by Han et al. (2004) when strawberries were treated with chitosan-based edible coating. Changes in the skin colour of *limau* madu stored at 10 °C were not so obvious with no significant difference both in the L* and hue angle values in the control and wax treated fruits during 4 weeks of storage. The L* value decreased only 2% while hue angle was 34%, which probably had influenced the formation of tinge yellow as seen after 3 weeks onwards both in the control and treated fruits. Thus, wax treatment did not contribute to colour changes of *limau madu* during low temperature storage as there was no significant changes in the L and hue values with duration of storage (*Table 1*).

Table 1. Changes in the skin colour (L and hue values) of *limau madu* during storage at 25 °C and 10 °C

	Treatment	L* value	Hue value
Storage at 25 °C			
Day 0	Control	46.67a	16.43a
	1% wax	46.78a	16.43a
	1.5% Wax	46.48a	16.43a
Day 4	Control	43.27b	11.31b
	1% Wax	45.22a	12.70b
	1.5% Wax	45.43a	11.80b
Day 8	Control	40.22c	4.25c
	1% Wax	43.78b	5.21c
	1.5% Wax	42.18b	5.34c
Storage at 10 °C			
Day 0	Control	47.50a	16.43a
	1.0% Wax	47.36a	16.43a
	1.5% Wax	47.43a	16.43a
Week 1	Control	47.21a	14.38a
	1.0% Wax	47.55a	15.05a
	1.5% Wax	47.22a	15.32a
Week 2	Control	46.32a	12.23b
	1.0% Wax	46.46a	11.76b
	1.5% Wax	46.57a	10.37b
Week 3	Control	45.99a	10.18b
	1.0% Wax	46.35a	10.57b
	1.5% Wax	46.56a	11.32b
Week 4	Control	45.67a	10.17b
	1.0% Wax	46.78a	11.39b
	1.5% Wax	46.48a	10.83b

Each value is the mean value from 20 fruits to *limau madu* Means with the same letters within column are not significantly different at 5% level



Figure 2. Changes in the TSS pH values of **limau madu** treated with 1.0% and 1.5% wax treatment stored at 25 °C and 10 °C. Evaluation was conducted on day 2, 4, 6 and 8 for samples stored at 25 °C and week 1, 2, 3 and 4 for samples stored at 10 °C. Bars with the same letters (abc) within time are significantly different at p < 0.05

The TSS value of the control fruits stored at 25 °C increased on day 2 (12.7 °Brix), and later decreased significantly (p < 0.005) until the end of 8 days of storage with 12.3 °Brix (Figure 2). However, the TSS values of the treated fruits (1.0% and 1.5% wax) were not significantly different throughout the 8 days of storage. The decreasing trend in the TSS value of the control fruits was probably related to the active biochemical changes and the use of sugar content for energy metabolism. There was no significant difference in the TSS value between the control and treated fruits with duration of storage in the fruits stored at 10 °C even though a slight increase was observed with prolonged storage (Figure 2).

Wax treatment had contributed to significantly higher pH value as shown in *limau madu* stored at 25 °C and 10 °C. However, no significant difference was noted between 1.0% and 1.5% (*Figure 2*). Inconsistent trend in the pH value was noticeable in fruits stored at 25 °C, whereas at 10 °C the value increased with duration of storage (*Figure 2*).

Changes in the skin colour of limau madu was not a significant signal for limau madu to have a prime taste as observed in the control fruits during storage at 25 °C. Pronounced yellow colour development in the control fruits did not influence the fruit taste as no significant difference was shown with duration of storage (Table 2). Significant changes was observed in colour and texture of the control fruits after day 4 onwards, however, no significant changes was observed on other sensory attributes throughout the 8 days of storage (Table 2). Similar results were also observed on the texture, taste, odour and overall acceptability of limau madu stored at 10 °C (Table 2).

Conclusion

Waxing improves the overall appearance of *limau madu* as it imparts a shiny look. Fruits retained its turgidity as the weight loss was reduced compared to the control fruits. Beneficial effects of wax treatment in reducing the weight loss of *limau madu* was clearly seen during storage at 25 °C. The weight loss of wax treated fruits was significantly reduced compared to the control fruits. The use of 1.0% wax treatment was sufficient to give positive effect in controlling weight loss as no significant difference was shown between 1.0% and 1.5%. Wax at 1.0% concentration was also sufficient in maintaining the green colour of limau madu stored at 25 °C. Pronounced yellow colour development was observed on the control fruits after day 6 onwards. However, at 10 °C the green colour was quite stable in both the control and treated fruits indicating that the temperature of 10 °C was suitable for controlling the chlorophyll degradation.

Significant contribution of wax treatment to the TSS value was shown only in fruits stored at 25 °C as higher value was maintained after day 4 onwards. Significantly lower pH was observed in the control fruits both during storage at 25 °C and 10 °C. Changes in the physical and chemical components of *limau madu* at 25 °C and 10 °C did not influence overall acceptability of the fruits. However, better appearance of wax treated fruits probably contribute to a significant impact in marketing strategy of *limau madu*.

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Storage	Treatment	Colour	Texture	Taste	Odour	Overall acceptability
Storage at 25 °C						
Day 0	Control	6.7a	6.8a	6.8a	6.5a	6.7a
	1%	6.7a	6.8a	6.8a	6.5a	6.7a
	1.5%	6.7a	6.8a	6.8a	6.5a	6.7a
Day 2	Control	6.7a	6.7a	6.8a	6.4a	6.6a
	1%	6.7a	6.8a	6.8a	6.4a	6.7a
	1.5%	6.7a	6.8a	6.8a	6.4a	6.7a
Day 4	Control	6. 4b	6.4b	6.8a	6.4a	6.5a
	1%	6.7a	6.7a	6.8a	6.4a	6.6a
	1.5%	6.7a	6.7a	6.8a	6.4a	6.6a
Day 6	Control	6.2c	6.4b	6.8a	6.4a	6.5a
	1%	6.6a	6.6a	6.8a	6.4a	6.6a
	1.5%	6.6a	6.6a	6.8a	6.4a	6.7a
Day 8	Control	6.2c	6.4b	6.8a	6.4a	6.5a
	1%	6.6a	6.6a	6.8a	6.4a	6.6a
	1.5%	6.6a	6.6a	6.8a	6.4a	6.7a
Storage at 10 °C						
Day 0	Control	6.7a	6.8a	6.8a	6.5a	6.5a
	1%	6.7a	6.8a	6.8a	6.5a	6.5a
	1.5%	6.7a	6.8a	6.8a	6.5a	6.5a
Week 1	Control	6.6a	6.6a	6.8a	6.4a	6.5a
	1%	6.7a	6.8a	6.8a	6.4a	6.7a
	1.5%	6.7a	6.8a	6.8a	6.4a	6.7a
Week 2	Control	6.6a	6.6a	6.8a	6.4a	6.5a
	1%	6.7a	6.8a	6.8a	6.4a	6.7a
	1.5%	6.7a	6.8a	6.8a	6.4a	6.7a
Week 3	Control	6.6a	6.6a	6.6a	6.4a	6.5a
	1%	6.7a	6.8a	6.8a	6.4a	6.7a
	1.5%	6.7a	6.8a	6.8a	6.4a	6.7a
Week 4	Control	6.6a	6.6a	6.6a	6.4a	6.5a
	1%	6.7a	6.8a	6.8a	6.4a	6.7a
	1.5%	6.7a	6.8a	6.8a	6.4a	6.7a

Table 2. Changes in the sensory characteristics of *limau madu* with different wax treatments during storage at 25 °C and 10 °C

The sensory evaluations were carried out by a panel of 10 fixed panellists. The panel was requested to evaluate the fruit for various attributes using the 7-point hedonic scale (1 = Very unacceptable, 2 = Unacceptable, 3 = Moderately unacceptable, 4 = Neither good nor bad, 5 = Moderately good, 6 = Good and 7 = Very good). Means with the same letters within column are not significantly different at 5% level

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Abstrak

Perubahan kualiti limau madu (Citrus reticulate) yang diberi perlakuan pelilinan menggunakan Semperfresh 1.0% dan 1.5% dikaji semasa penyimpanan pada 25 °C dengan kelembapan relatif 65-70%, dan pada 10 °C dengan kelembapan relatif 85-90%. Buah tanpa rawatan dijadikan sebagai buah kawalan. Penilaian kualiti dilakukan setiap dua hari sekali bagi buah yang disimpan pada 25 °C dan setiap minggu bagi buah yang disimpan pada 10 °C. Sampel dianalisis untuk menilai perubahan fizikal, kimia dan penerimaan rasa. Keputusan menyeluruh menunjukkan buah yang disimpan pada 25 °C boleh diterima hingga hari kelapan dengan buah yang diberi rawatan pelilinan mempamerkan kualiti buah yang lebih baik dan buah kelihatan lebih berkilat. Buah kawalan mengalami kehilangan berat yang tinggi (2.5-6.5%) berbanding dengan buah yang diberi rawatan pelilinan (0.5-1.8%). Bagaimanapun tiada perbezaan ditunjukkan antara 1.0% dengan 1.5% rawatan pelilinan. Pemerhatian yang serupa dikesan pada buah yang disimpan pada 10 °C selama 4 minggu. Kehilangan berat yang rendah berlaku pada buah yang diberi rawatan pelilinan (0.03-1.5%) berbanding dengan sampel kawalan (0.5-2.5%). Tiada perbezaan yang ketara pada warna buah seperti yang ditunjukkan oleh nilai L and hue antara 1.0% dengan 1.5% rawatan pelilinan. Sedikit pembentukan warna kuning berlaku pada buah yang disimpan pada 10 °C selepas 3 minggu penyimpanan. Bagaimanapun tiada perbezaan yang ketara pada buah yang diberi rawatan pelilinan sehingga akhir 4 minggu penyimpanan. Penurunan yang ketara berlaku bagi nilai TSS buah kawalan selepas 6 hari penyimpanan pada 25 °C. Bagaimanapun tiada perbezaan ketara antara buah yang diberi pelilinan 1.0% dengan 1.5%. Nilai TSS bagi buah kawalan dan buah rawatan pelilinan agak stabil (12.8-12.6) semasa 4 minggu penyimpanan pada 10 °C. Nilai pH buah kawalan adalah rendah semasa penyimpanan pada 25 °C dan 10 °C. Rawatan pelilinan tidak dapat mempengaruhi rasa, bau dan penerimaan keseluruhan bagi buah yang disimpan pada 25 °C dan 10 °C.