



IMPACT OF SPRAYING SOME CHEMICAL SUBSTANCES ON PROTECTING VALENCIA ORANGE TREES FROM HEAT STRESS INJURIES

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Abstract

High temperatures avoid maturity and ripening processes of citrus fruit and lead to injuries as sunburn which reduces the grade and quality of Valencia orange. To overcome this problem, this experiment was carried out during two successive growing seasons (2017 and 2018) on Valencia orange trees grown in the National Research Centre farm for research and production in Al-Nubaria region, Al-Behira Governorate, Egypt using kaolin, calcium types (hydroxide, carbonate, silicate), magnesium carbonate. We observed that all sunburn treatments improved vegetative growth characteristics, and leaves macro and micro elements, and fruit physical and chemical characteristics of Valencia orange trees compared to control. Among all those treatments, there was a clear superiority of calcium silicate treatment which improved most of the previous characteristics. It was also clear that all calcium materials improved peel protein and calcium content which in turn led to decrease the percentage of sunburned fruits.

Keywords: Valencia orange, reflective materials, kaolin, calcium types (hydroxide, carbonate, silicate), magnesium carbonate, yield, fruit quality, Sunburn.

Introduction

Temperature is a major climatic factor that limits geographical distribution of plant species and is the most significant factor affecting antioxidant activity in vegetables and fruits (Réblová, 2012). Temperature sensitive crops include perennial crops such as almonds, grapes, berries, citrus and stone fruits (Lobell and Field, 2011). High temperatures during the summer in Egypt lead to a very high-water evaporation rate. Such, the reproductive phase is one of the most temperature-vulnerable stages, whereas fruits are known to mature normally at temperature as high as 35 °C, as well as higher temperatures block ripening processes. So, sunburn is a physiological disorder associated with high temperature and drought probably causes a water imbalance. It often happens when the fruits are very susceptible (Bella *et al.*, 2002) or as a result of the negative effect of summer petroleum spray oils in citrus (Tumminelli *et al.*, 2003), therefore the application of modern production technology played a key role to overcome that problem. To reduce sunburn problems, either using a specific height of trees (high amount of foliage canopy) which can be used to deal with high temperatures and to provide protection from heat stress, or calcium types and kaolin which are good alternatives with relative low cost, safe use, reduced particle size and water diffusion ability (Glenn *et al.*, 2003). Also, Glenn *et al.*, (2002) found that crimson Seedless grapevines treated with calcium carbonate or crystalline limestone were less prone to sunburn damage than untreated ones and this is due to reducing both fruit temperature and exposure to ultraviolet (UV) radiation. Reflective kaolin spray was found to decrease leaf temperature by increasing leaf reflectance and to reduce transpiration rate more than photosynthesis in many plant species grown at high solar radiation levels (Nakano and Uehara, 1996). Silicon, the second most abundant element in the earth's crust, however, various studies have demonstrated that Si application significantly increased plant growth (Alvarez and Datnoff, 2001). Silicon is also known to increase drought tolerance in plants by maintaining plant water balance, photosynthetic activity,

erectness of leaves and structure of xylem vessels under high transpiration rates (Hattori *et al.*, 2005). Spraying magnesium carbonate (Silicon) as anti-transpiration increased growth parameters and improved banana yield weight and fruit characteristics (Abd El-Kader *et al.*, 2006). The present study was outlined to study the effect of some chemical substances "anti-stress" as kaolin, calcium types (hydroxide, carbonate, silicate), magnesium carbonate on reducing transpiration and their effect on growth and nutrients status, yield and fruit quality of Valencia orange trees under sandy soil conditions.

Materials and Methods

Plant

Valencia orange (*Citrus sinensis* Osbeck) trees, grafted on volkamer lemon rootstock (*C. volkameriana*, L.), were about twelve years old, and in healthy and uniform condition, planted in a system of 3.5 x 5 meters, and grown on sandy soil in the National Research Centre farm for research and production in Al-Emam Malek village, Al-Nubaria region, Al-Behira Governorate, Egypt. This study was carried out during two successive growing seasons (2017 and 2018).

Foliar spray compounds

Foliar spray substances were used in each season in mid-June at a concentration (3 %) to reduce the impact of heat stress on growth, productivity, and quality.

- 1- kaolin, extra pure [$K_2Al_2Si_2O_8 \cdot H_2O$] (product from "SDFCL" Sd fine-Chem Limited 315- 317, T.V. Industrial Estate, 248, Worli Road, Mumbai- 30, India).
- 2- Slaked lime "Calcium hydroxide" [$Ca(OH)_2$] (obtained from a local market)
- 3- Calcium silicate [CaO_3Si] (product from Sigma-Aldrich Chemistry, USA).
- 4- Calcium carbonate [$CaCO_3$] (Produced by Al-Gomhoria pharmaceutical company in Egypt).
- 5- Magnesium carbonate [$MgCO_3$] (Produced by Al-Gomhoria pharmaceutical company in Egypt).

6- This was done comparing with the trees sprayed with water (control).

All spray solutions contained 0.1 % triton B as a wetting agent to avoid the surface tension and sprayed till run off. Each treatment received 20 L of the applied solution, in the morning hours of the day in mist form. A complete randomized block design was adopted in this experiment with six treatments, where each treatment contained three replicates with one tree each.

Other horticultural practices were similar for all trees as recommended from national campaign for improving citrus productivity in Egypt. Drip irrigation system was used to irrigate all trees.

Measurements

Growth and yields: in early September, leaf area was measured using the formula of $0.608 \text{ constant} \times (\text{maximum leaf length} \times \text{maximum leaf breadth})$ according to Shrestha and Balakrishnan (1985). Leaf chlorophyll content was determined as CCI (Chlorophyll Content Index) using Chlorophyll content Meter 003109 (CCM-200 plus Opti-Sciences). At commercial harvest in early April, yield as weight and number of fruits per tree was recorded. Canopy volume of trees was measured in early December which tree shape was considered as a one-half of a prolate spheroid (volume = $\frac{4}{6} \times \pi \times \text{height} \times \text{radius}^2$ "which $\pi = 22/7$ ") as described by Roose *et al.*, (1989). Cropping efficiency was calculated by dividing the fruit yield weight by the tree canopy according to Whitney *et al.* (1995).

Leaf mineral composition: leaf samples were collected in early September and were mature fully expanded from non-fruited non flushing spring cycle growth (5 months old)

Results and Discussion

Table 1: Mitigation of heat stress effects using some sunburn treatments on leaf area, chlorophyll content, tree canopy, yield/tree, and crop efficiency of Valencia orange trees during 2017/2018 seasons.

Treatments	Leaf area (cm ²)		Total chlorophyll (CCI)		Tree canopy (m ³)		Yield/tree (kg)		Crop efficiency (Kg/m ³)	
	1 st	2 nd	1 st	2 nd	1 st	2 nd	1 st	2 nd	1 st	2 nd
Kaolin	23.49 b	22.82 ab	108.35 a	61.33 b	81.17 a	82.33 ab	31.57 ab	48.13 a	0.39 b	0.58 c
Slaked lime "calcium hydroxide"	20.52 bc	22.60 ab	107.67 a	61.96 b	63.03 b	79.89 ab	38.55 a	45.82 a	0.61 a	0.59 c
Calcium silicate	21.25 bc	26.79 a	108.44 a	92.38 a	79.80 a	98.14 a	37.53 a	50.33 a	0.47 ab	0.52 c
Calcium carbonate	19.05 c	25.56 a	106.03 a	60.67 b	58.14 b	61.30 bc	26.82 ab	48.74 a	0.46 ab	0.81 ab
Magnesium carbonate	23.51 b	19.65 b	97.84 a	62.51 b	54.26 bc	56.29 c	27.58 ab	49.56 a	0.50 ab	0.88 a
Control (water spray)	27.62 a	24.70 a	78.40 a	81.87 ab	45.18 c	46.14 c	21.27 b	32.74 b	0.47 ab	0.71 b

Values followed by the same letter/s over each column didn't significantly differ at 5% level

Data illustrated in Table (1) reveal that all sunburn treatments improved vegetative growth characteristics of Valencia orange trees. The water spray (control) gave the highest leaf area in 2017 season followed by magnesium carbonate sunburn treatment, while, calcium silicate treatment was the superior in 2018 season, followed by calcium carbonate although the insignificance between treatments in the second season. Calcium silicate treatment

according to Jones and Embleton (1960), then washed, dried at 70 °C until a constant weight, ground and digested using an acid mixture consisting of nitric, perchloric and sulfuric acids in the ratio of 8:1:1 (v/v), respectively according to Chapman and Pratt (1978). Nitrogen was measured by semi-micro Kjeldahl method of Plummer (1978). Phosphorus was determined using a spectrophotometer at 882-OVV by the method outlined by Jackson (1973). Potassium, calcium and sodium were determined by a flame photometer "Jenway PFP7". Magnesium, iron, manganese, zinc and copper were determined using atomic absorption Spectrophotometer "Perkin Elmer 1100" (Cottanie *et al.*, 1982).

These measurements were performed in the Agricultural Services Unit and Laboratory Analysis of Research Project (Micronutrients and Other Plant Nutrition Problems in Egypt) in NRC.

Fruit quality: ten fruits were randomly sampled per each tree for determination of weight, diameter, peel thickness, then from the juice, total soluble solids percentage (TSS Brix %) was determined by Carl Zeiss hand refractometer; total acidity was measured as anhydrous citric acid % and vitamin C was expressed as mg ascorbic acid per 100 ml juice according to A.O.A.C. (1995).

Some peel mineral composition: in the dry peel tissue, protein was determined as nitrogen as above then multiplied by 6.25. Calcium was determined according to (Cottanie *et al.*, 1982).

Statistical analysis: The data obtained in each season were analyzed by ANOVA according to Snedecor and Cochran (1982). Means were separated by Duncan (1955) and multiple range test using a significance level of $P < 0.05$.

attained the highest leaves total chlorophyll in the two seasons despite the lack of differences among treatments in 2017 season. About tree canopy, kaolin and calcium silicate were the most efficient treatments in 2017 and 2018 seasons, followed by slaked lime "calcium hydroxide", while, the water spray (control) gave the lowest tree canopy in the two seasons. All sunburn spraying materials improved trees yield compared to control in both seasons. Regarding crop

efficiency, slaked lime "calcium hydroxide" was the superior in the first season although the lack of variance between treatments in the first season, while magnesium carbonate treatment was the highest in the second season, followed by calcium carbonate. These results could be due to the effect of those materials on increasing vegetative growth, photosynthesis rate. This led to an overall positive effect on total production per tree. So, it is therefore recognized that the increments happened in vegetative growth of orange (leaf total chlorophyll content, leaf area) treated with anti-transpirations such as kaolin was possibly due to two aspects. First was the protection of tissues from climatic condition, and second was the increase of water potential at a time when the plant growth was more dependent on water status than on photosynthesis. Under hot conditions, when untreated trees shut down photosynthesis, trees treated with those materials continue to photosynthesis at high levels because the trees remain cooler and their stomata remain open. This can lead to higher levels of carbon fixation that in turn may result in

benefits such as high tree vigor and fruit number. Calcium carbonate or crystalline limestone is marketed as a liquid that is mixed with water and sprayed onto crop foliage and fruit to form a thin crystalline layer that reflects some sunlight (Crusciol *et al.*, 2009). Using anti transpiration agent improves water use efficiency by decreasing leaf transpiration rate, reducing leaf water loss and improving assimilation area of plant in sandy soil (Nehad, 2015). Silicate accumulates in the leaf, forming a doubled layer and this accumulation promotes a reduction in transpiration and decreases water loss (Freitas *et al.*, 2011). Silica plays an imperative function, which can provide a greater leaf area towards the light, thereby increases the efficiency of plant photosynthesis (Quanzhi and Erming, 1998). This result is attributed to silicon which enhances the state of water in plant cells, therefore, making the plant easier to process photosynthesis (Crusciol *et al.*, 2009). This increment of the yield can be discussed with the stimulation of silicate for the vegetative growth of Valencia orange, as shown in Table 1.

Table 2 a: Mitigation of heat stress effects using some sunburn treatments on some minerals content of Valencia orange leaf during 2017 and 2018 seasons.

Parameters Seasons Treatments	N (g/100g)		P (g/100g)		K (g/100g)		Ca (g/100g)		Mg (g/100g)	
	1 st	2 nd	1 st	2 nd	1 st	2 nd	1 st	2 nd	1 st	2 nd
Kaolin	2.47 b	2.48 a	0.400 b	0.450 ab	1.510 b	1.380 ab	5.50 ab	5.80 ab	0.460 b	0.420 b
Slaked lime "calcium hydroxide"	2.52 ab	2.50 a	0.400 b	0.380 abc	1.550 b	1.440 ab	5.30 b	5.65 b	0.440 b	0.420 b
Calcium silicate	2.55 a	2.54 a	0.500 a	0.420 abc	1.830 a	1.00 b	5.95 ab	5.15 b	0.410 b	0.510 a
Calcium carbonate	2.36 c	2.45 a	0.460 a	0.500 a	1.530 b	1.400 ab	6.27 a	6.35 a	0.450 b	0.360 c
Magnesium carbonate	2.38 c	2.32 ab	0.300 c	0.340 bc	1.850 a	1.770 a	5.35 b	5.60 b	0.527 a	0.510 a
Control (water spray)	2.08 d	2.133 b	0.300 c	0.320 c	1.760 a	1.423 ab	6.00 ab	5.15 b	0.450 b	0.480 a

Values followed by the same letter/s over each column didn't significantly differ at 5% level

Adequate ranges for citrus leaf were: 2.4 - 3.5 (N), 0.15 - 0.3 (P), 1.2 - 2.0 (K), 3 - 7 (Ca), 0.25 - 0.7 (Mg). (Werner, 1992).

All sunburn treatments increased leaves macro elements content compared to control (Table 2a). Calcium silicate gave the highest leaf nitrogen content in both seasons, followed by slaked lime "calcium hydroxide" compared to control although the lack of differences between the sprayed sunburn materials in the second season. Concerning leaves phosphorus content, calcium silicate and calcium carbonate were the uppermost in 2017 and 2018 seasons compared to control, followed by kaolin and slaked lime "calcium hydroxide" in the second season. Spraying the trees of Valencia orange with magnesium carbonate achieved the highest values of leaf potassium content in the two seasons, while calcium carbonate gave the highest leaf calcium content in 2017 and 2018 seasons. Spraying the trees with magnesium carbonate gave the highest values of leaves magnesium content in both seasons, followed by calcium

silicate and control in the second season. Under subtropical conditions like Egypt, using anti transpirations may reduce the plant transpiration rate; consequently, the amount of used water improved the water use efficiency (Singh *et al.*, 1999). Silicon is not considered as an essential element for plant growth, in addition, its role in plant biology is not completely understood. By losing water from shoots, silicic acid is concentrated, then converts to silica gel ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$) and consequently increases plant's resistance to stress (Epstein, 1999). In this concern Abd El-Kader *et al.* (2006) could not detect any differences between anti-transpiration material treatments on N, P and K content of banana plant leaves. While Abou-Baker *et al.* (2011) reported that magnesium silicate solution gave the highest values of N % and P % in contrast to silicon which gave the highest K % values in bean plant tissue.

Table 2b: Mitigation of heat stress effects using some sunburn treatments on some minerals content of Valencia orange leaf during 2017 and 2018 seasons.

Parameters Seasons Treatments	Fe (ppm)		Mn (ppm)		Zn (ppm)		Cu (ppm)		Na (g/100g)	
	1 st	2 nd	1 st	2 nd	1 st	2 nd	1 st	2 nd	1 st	2 nd
Kaolin	139.0 bc	138.0 bc	25.80 ab	31.80 a	10.00 b	13.00 a	5.70 b	4.90 c	0.240 ab	0.270 a
Slaked lime "calcium hydroxide"	160.0 ab	110.0 c	30.50 ab	29.80 ab	16.00 a	11.00 bc	4.17 c	4.90 c	0.150 b	0.160 b
Calcium silicate	116.0 d	167.0 ab	23.70 b	22.80 c	13.00 ab	12.00 ab	5.40 bc	4.70 c	0.280 ab	0.290 a
Calcium carbonate	170.0 a	190.0 a	27.50 ab	29.50 ab	10.00 b	10.00 c	5.50 b	7.00 bc	0.260 ab	0.170 b
Magnesium carbonate	174.0 a	199.0 a	32.10 a	29.80 ab	12.00 ab	11.00 bc	4.50 bc	8.00 b	0.310 ab	0.180 b
Control (water spray)	120.0 cd	114.0 c	27.80 ab	26.50 bc	11.00 b	12.00 ab	8.00 a	11.00 a	0.380 a	0.210 b

Values followed by the same letter/s over each column didn't significantly differ at 5% level

Adequate ranges for citrus leaf were: 35 - 135 (Fe), 19 - 50 (Zn), 19 - 100 (Mn), 5 - 15 (Cu) (Wutscher and Smith, 1994).

It's clear from the data in Table 2b that all sunburn treatments increased leaf micro elements content compared to control except the copper content in which the control (water spray) was the superior. In connection with leaf iron content, there was a clear superiority of both magnesium carbonate and calcium carbonate treatments in this regard in both seasons. Magnesium carbonate treatment gave the highest value of leaf manganese content in the first season,

whereas, kaolin treatment was the highest in the second season. Concerning leaves zinc content, slaked lime "calcium hydroxide" showed the highest value in the first season, while kaolin was the highest in 2018 season. Although the low distinction between the treatments in the first season, kaolin and calcium silicate treatments gave the highest values of leaves sodium content in the second season.

Table 3: Mitigation of heat stress effects using some sunburn treatments on yield components and physical fruit characteristics of Valencia orange trees during 2017/2018 seasons.

Treatments	Sunburned fruit %		No. of fruit /tree		Fruit weight (gm)		Fruit diameter (cm)		Peel thickness (cm)	
	1 st	2 nd	1 st	2 nd	1 st	2 nd	1 st	2 nd	1 st	2 nd
Kaolin	7.09 bc	5.33 b	170.00 a	185.00 a	189.72 bc	259.72 c	7.07 ab	7.87 bc	0.43 a	0.57 b
Slaked lime "calcium hydroxide"	7.28 b	5.33 b	168.33 a	178.00 ab	230.00 a	257.77 c	7.41 a	7.72 bc	0.49 a	0.57 b
Calcium silicate	5.57 c	2.67 c	168.33 a	188.00 a	225.27 a	268.05 bc	7.36 a	8.06 abc	0.42 a	0.58 b
Calcium carbonate	6.26 bc	3.67 bc	154.33 a	164.00 ab	173.89 bc	297.22 ab	6.63 b	8.21 ab	0.47 a	0.51 c
Magnesium carbonate	6.72 bc	4.00 bc	143.33 a	154.00 bc	193.89 b	321.11 a	6.95 ab	8.51 a	0.51 a	0.51 c
Control (water spray)	18.66 a	16.00 a	130.00 a	135.00 c	166.94 c	242.77 c	6.66 b	7.57 c	0.53 a	0.60 a

Values followed by the same letter/s over each column didn't significantly differ at 5% level

It's discerned from data in Table 3 that all sunburn treatments enhanced fruit physical characteristics compared to control. Calcium silicate treatment decreased sunburned fruits in both seasons, followed in ascending order by calcium carbonate and magnesium carbonate, while water spray (control) gave the highest sunburned fruits in the two seasons. Calcium silicate and kaolin treatments gave the highest number of fruits per tree in the second season, while, no big differences between treatments in the first season despite the supremacy of kaolin, slaked lime "calcium hydroxide" and calcium silicate treatments respectively. Slaked lime "calcium hydroxide" and calcium silicate treatments gave the highest fruit weight and fruit diameter in the first season respectively, while magnesium carbonate

treatment gave the highest fruit weight and fruit diameter in the second season. Calcium carbonate and magnesium carbonate attained the lowest peel thickness in 2018 season, whereas, no obvious differences among treatments in 2017 season. The results were in agreements with those obtained by Abd El-Naby *et al.* (2005) who showed that using slaked lime on mandarin was more preferred for its economic cost and led to obtain fruits with low sunburn disorders and good quality favorable to consumers. The results showed that slaked lime and sun shield sprays resulted in obvious decrease of sunburn disorders percentage compared with water spray (control) by about 51.98 - 67.87 and 45.74 - 72.84 % for the two seasons, respectively.

Table 4: Mitigation of heat stress effects using some sunburn treatments on chemical characteristics in juice and peel of Valencia orange fruit during 2017/2018 seasons.

Treatments	Total soluble solids (%)		Titratable acidity (%)		Ascorbic acid (mg/100 ml juice)		Peel protein %		Peel Ca (g/100g)	
	1 st	2 nd	1 st	2 nd	1 st	2 nd	1 st	2 nd	1 st	2 nd
Kaolin	11.61 b	12.77 ab	1.13 b	2.50 bc	44.35 a	46.80 a	2.19 b	2.00 ab	0.46 b	0.54 c
Slaked lime "calcium hydroxide"	10.78 c	12.77 ab	1.90 a	2.57 b	32.85 a	46.00 a	3.06 a	2.44 a	0.60 a	0.71 b
Calcium silicate	10.51 c	12.40 bc	1.85 a	2.13 d	40.96 a	43.60 a	2.62 ab	2.31 a	0.45 b	0.53 c
Calcium carbonate	12.53 a	12.47 bc	1.91 a	2.03 d	44.77 a	42.60 a	2.18 b	2.00 ab	0.63 a	0.84 a
Magnesium carbonate	12.95 a	11.97 c	2.21 a	2.20 cd	35.97 a	43.00 a	3.06 a	2.50 a	0.48 b	0.52 c
Control (water spray)	11.87 b	13.30 a	2.08 a	2.90 a	48.40 a	43.80 a	2.08 b	1.50 b	0.48 b	0.50 c

Values followed by the same letter/s over each column didn't significantly differ at 5% level

It's clear from the aforementioned data in Table 4 that spraying Valencia orange trees with some sunburn treatments enhanced fruit chemical characteristics. Magnesium carbonate and calcium carbonate treatments enhanced fruit total soluble solids percentage in the first season, while, water spray (control), kaolin and slaked lime "calcium hydroxide" were the highest in the second season. The lowest values of titratable acidity were obtained by kaolin treatment in 2017 season, and calcium carbonate and calcium silicate in 2018 season respectively. No significant differences were shown among treatments concerning fruits ascorbic acid content in both seasons, nevertheless, water spray (control) treatment showed the highest value in 2017 season, while, kaolin treatment was the highest in 2018 season. Regarding peel protein content, slaked lime "calcium hydroxide" and magnesium carbonate treatments showed up the highest content in both seasons, followed by calcium silicate. Calcium carbonate treatment improved peel calcium content in 2017 and 2018 seasons compared to other treatments. Also, application of sprayed materials led to improving fruit properties [weight, dimensions, peel thickness]; juice quality [high total soluble solid "TSS", low titratable acidity "TA", high TSS : TA ratio and high V.C] (Abd El-Naby *et al.*, 2005), while the quality of fruits was affected by sunburn (Racskó *et al.*, 2005). Similar trend was reported by Jian-guang *et al.* (2003) on apple, who found that protein levels increased due to the increase of the treatment's duration which in turn increased the resistance to sunburn. Sanyal *et al.* (1990) found that litchi fruits containing lower Ca content in the peel was affected by sunburn disorders.

Conclusion

According to the previous results, to eliminate sunburn disorder which reduces the grade and quality of Valencia orange fruits, we observed that all sunburn treatments improved vegetative growth characteristics, and leaves macro and micro elements, and fruit physical and chemical characteristics of Valencia orange trees compared to control. Among all those treatments, there was a clear superiority of calcium silicate treatment which improved most of the previous characteristics. It was also clear that all calcium materials improved peel protein and calcium content which in turn led to decrease the percentage of sunburned fruits.

Acknowledgment

We wish to express our deep appreciation and gratitude to the National Research Centre in Egypt for the financial support to this study through the project of "Mitigation of heat stress on some horticultural crops by using melatonin and agricultural treatments".

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