# Physico-chemical changes during growth and development of grapefruits (*Citrus paradisi* Macf.). II. Chemical changes

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## ABSTRACT

The chemical changes during growth and development of seedy pink-fleshed 'Red Blush' grapefruits were evaluated to provide base-line information regarding the biochemistry of the developing fruit and to assist in determining harvest maturity of grapefruits. The fruits exhibited a typical non-climacteric pattern of respiration. Respiration rate decreased from 239.8 mg CO<sub>2</sub>/kg-hr (4 WAA) to 21.1 mg CO<sub>2</sub>/kg-hr (26 WAA). Total soluble solids (TSS) and total sugars progressively increased from 4 weeks after anthesis (WAA) up to 26 WAA at physiological maturity, and then remained constant. Reducing sugars, titratable acidity and ascorbic acid content progressively increased from 4 WAA, reaching a peak at 16, 10 and 8 WAA, respectively, and then declined to minimum values at physiological maturity. Grapefruits should be harvested at least at physiological maturity, where the fruit attains maximum size and weight, rind color develops, TSS and total sugars reach above 14% and 14 g/100g fresh weight, titratable acidity is reasonably low, ascorbic acid content reasonably high and the fruit is still firm.

#### **INTRODUCTION**

Grapefruit (*Citrus paradisi* Macf.) is an important citrus fruit which originated in the West Indies and then spread to many tropical and sub tropical countries (Salunkhe and Desai, 1984). During the last five decades, the demand of citrus had increased due to the improved economic conditions and health awareness of consumers, together with the advances in agricultural sciences and technology of by-products. Citrus fruit is fast becoming a stable food product in the daily diet of many people (Ting and Attaway, 1971).

Citrus fruits rank first in their contribution of vitamin C to human nutrition. They are also a rich source of calcium and potassium (Kader and Arpaia, 2002). The annual world production of grapefruits and pummelos (*Citrus grandis*) is 710,948 metric tons (FAO, 2010). China ranks first in citrus production, followed by Nigeria, India, and Columbia (FAO, 2010).

In Sudan, grapefruits are produced in Kassala, River Nile, Northern, Sinnar, South Kordofan, South and West Darfur States, with annual total production of 196,000 metric tons (HSA, 2014). Judging by palatability and external appearance, the quality of grapefruits grown in Sudan has been commended as superior to fruits grown in other leading citrus-producing areas (Robbie and Fisher, 1954).

Most citrus fruits have a period of rapid size increase, which varies in length according to the variety and environmental factors. During fruit development, the major changes in chemical constituents include those in fruit color (carotenoids), total nitrogen (amino acids, amines, peptides and proteins), carbohydrates, organic acids (citric, oxalic, malonic and succinic), flavonoids and

limonoids, lipids, volatile compounds, waxes, steroids, terpenoids, vitamins and inorganic mineral constituents (Salunkhe and Desai, 1984).

Citrus fruits are non-climacteric and their respiration and ethylene production rates are low (Kader and Arpaia, 2002). Their compositional changes are minimal and they do not undergo rapid chemical or physical changes after the fruit is detached from the tree. Citrus fruits contain no starch and they cannot be picked green for after harvest ripening. There is no post-harvest improvement in fruit quality (Wills *et al.*, 1998). These necessitate that the fruit should be picked at the optimum maturity.

Harvesting citrus at the proper level of maturity is essential for good quality produce. Overmaturity or under-maturity affects the quality adversely (Wills *et al.*, 1998). Many physical and chemical changes undergone by the developing fruit have been used as means of assessing the optimal picking date for immediate consumption or storage. None of these parameters are reliable individually for determining harvest maturity. It usually requires a combination of physical and chemical parameters, coupled with considerable experience (Salunkhe and Desai, 1984). Minimum maturity requirements of citrus fruits are based on juice content (lemon and lime), soluble solid content, titratable acidity and the ratio of the two (orange, grapefruit and mandarine) (Kader and Arpaia, 2002).

Sufficient data are not available on many commercial cultivars to fix maturity standards for harvesting, on the basis of fruit growth and physical and chemical parameters (Abu-Goukh *et al.*, 2005). Previous studies on physical changes during growth and development of grapefruits indicated that the fruits followed a typical sigmoid curve. Fruit fresh weight, volume, length, diameter, peel thickness, pulp diameter, juice content, and peel color progressively increased from 4 weeks after anthesis (WAA) up to 26 WAA at physiological maturity and then remained constant. Peel/pulp ratio and fruit flesh firmness steadily decreased with advancement in growth and development, reaching minimum values at physiological maturity (Almahi and Abu-Goukh, 2016).

This study was carried out to evaluate the chemical changes during growth and development of seedy pink-fleshed 'Red Blush' grapefruits and to provide base-line information regarding the biochemistry of the developing fruit to assist in determining harvest maturity of grapefruits.

## **Experimental material**

#### **MATERIALS AND METHODS**

Grapefruits of seedy pink-fleshed 'Red Blush' cultivar were selected for this study. Ten trees were selected in a private orchard in Al-Ezeirgab area, Khartoum North (15°43' N, 32°33'E) during 2014/2015 season. At the time of flowering (April), the newly open flowers were tagged and fruit samples were harvested at different stages of growth and development. The first sample was picked four weeks after anthesis, and sampling continued every two weeks up to the physiological maturity (12 samples). Sixty fruits of uniform size per sample were picked and arranged in a completely randomized design with 4 replicates. The fruits were washed and air dried.

## **Parameters studied**

Chemical changes were determined on 15 fruits picked at the designated stage. Respiration rate was determined using the total absorption method (Mohamed-Nour and Abu-Goukh, 2010), and respiration rate was expressed in mg  $CO_2/kg$ -hr. Total soluble solids (TSS) were determined on the fruit juice extracted by pressing the fruit pulp in a garlic press using a Kruss hand refractometer (Model HRN-32). Two readings were taken from opposite sides of each fruit and mean values were calculated and corrected according to the refractometer chart.

Thirty grams of pulp of the fruit were minced and homogenized in 100 ml of distilled water for one minute in an electric mixer (Molinex model No. 241) and centrifuged at 6000 rpm for 10 minutes in a Gallenkamp portable centrifuge (CF-400). The volume of the supernatant, which constituted the pulp extract, was determined. Total sugars were determined in pulp extract according to the anthrone method of Yemm and Willis (1954). Reducing sugars were measured according to the technique described by Somogyi (1952). Total and reducing sugars were expressed in grams per 100 grams fresh weight. Titratable acidity was determined in the pulp extracts according to the method described by Ranganna (1979) and was expressed in percent citric acid.

Thirty grams of pulp from the fruit were homogenized in 100 ml of oxalic acid for one minute in an electric mixer (Molinex model No. 241) and centrifuged at 6000 rpm for 10 minutes in a Gallenkamp potable centrifuge (CF-400). The volume of the supernatant was topped to 250 ml oxalic acid. Ascorbic acid content was determined using 2,6-dichlorophenol-indophenol titration method of Ruck (1963) and was expressed in mg/100g fresh weight.

#### Statistical analysis

Analysis of variance, followed by Fisher's protected LSD test with a significance level of  $P \le 0.05$  were performed on the data (Gomez and Gomez, 1984).

# **RESULTS AND DISCUSSION**

## **Respiration rate**

The respiration curve exhibited a typical non-climacteric pattern. The respiration rate significantly decreased with advancement in growth and development from 239.8 mg CO<sub>2</sub>/kg-hr (4 WAA) to 21.1 mg CO<sub>2</sub>/kg-hr (26 WAA). (Fig.1). Fruits harvested near horticultural maturity showed gradual decline in the rate of respiration and produce no ethylene, and grapefruits are classified as non-climacteric fruits (Kader and Arpaia, 2002). Similar results were reported with pink-fleshed 'Foster' grapefruits (Abu-Goukh and Elshiekh, 2008). Aharoni (1968) studied the pattern of respiration of different cultivars of grapefruits and oranges picked at different stages of growth and development. The low rate of respiration and the unobservable climacterics are associated with the slow rate of chemical reaction that occurs in these fruits during growth and development.



#### **Total soluble solids**

Total soluble solids (TSS) progressively increased from 10.4 % (4 WAA) to 14.3% (26 WAA) with an increase of 37.5% (Fig. 2). Abu-Goukh and Elshiekh (2008) found that maximum TSS in 'Foster' grapefruits was 11.5%. Ladaniya and Mahalle (2011) reported that TSS in 'Mosambi' oranges increased slowly, with no significant differences, between 180 and 210 days after fruit set. At 220 days, there was a significant increase in TSS to 11.7%. As the fruit matured, TSS increased to 12.8% at 240 days after fruit set. Nearly 75% to 85% of TSS in orange juice is sugars (Ladaniya, 2008).



Fig. 2: Changes in total soluble solids (TSS) during

## Total and reducing sugars

Changes in total sugars during growth and development of grapefruit followed the same pattern of TSS (Fig. 3). Total sugars significantly increased during growth and development, from 7.0 g/100g (4 WAA) to 14.1 g/100g (26 WAA). Ahmad *et al.* (1992) reported that total sugar content in grapefruits steadily increased during October to early December and became constant from mid December to January. Non- reducing sugars followed the same pattern of total sugars, reaching their maximum value on late January. Garray *et al.* (2002) reported that total sugars had increased in four 'Valencia' orange cultivars between the fourth and the fifth months of fruit development and then started to decrease thereafter. Towards the time of harvest (at the eighth month), the four cultivars showed a decline in total sugar content.



Fig. 3: Changes in total sugars during growth and development of "Red Blush" grapefruits.

Reducing sugars increased gradually from 1.3 g/100g (4 WAA), reaching a peak of 2.4 g/100g (16 WAA) and then declined to a value of 1.5 g/100g (26 WAA) (Fig. 4). Similar amounts of reducing sugars (1.53 g/100g) were obtained in the fruit 8 WAA and 26 WAA. This suggests that reducing sugars produced during growth and development of grapefruits are devoted merely to run the respiration process to provide energy requirements for the morphological, anatomical and physiological changes that take place during the exponential growth phases (8-22 WAA) and maturation phase (22-26 WAA) (Almahi and Abu-Goukh, 2016).



Fig. 4: Changes in reducing sugars during growth and development of "Red Blush" grapefruits.

#### Titratable acidity

Titratable acidity progressively increased from 0.36% at 4 WAA, reaching a peak of 0.46% at 10 WAA and then gradually decreased to 0.17%, at physiological maturity (26 WAA) (Fig. 5). In grapefruits and oranges, the free acid per fruit increased in early growth and then became approximately constant (Ting and Attaway, 1971). The decrease in titratable acidity was considered to be due to dilution, as the fruit increased in size and in juice content. The decrease in the concentration of acid with the gradual increase in total sugars during development, results in an increase in the ratio of total soluble solids to acid, which is the basis for determining the legal maturity of the fruits as well as their palatability (Ting and Attaway, 1971). A sharp decline in titratable acidity was reported during fruit maturation in 'Mosambi' oranges (Ladaniya and Mahalle, 2011) and 'Blood Red' oranges (Khokar and Sharma, 1984) and during storage of grapefruits (Abu-Goukh and Elshiekh, 2008). The loss of acidity during fruit maturation and storage appears to result, at least in part, to the use of acids as a respiratory substrate *via* the Kreb's tricarboxylic acid cycle (Buser and Mantile, 1977).



Fig. 5: Changes in titratable acidity during growth and development of "Red Blush" grapefruits.

#### Ascorbic acid content

The ascorbic acid content followed a similar pattern of acidity. It significantly increased from 59.7 mg/100g at 4 WAA, reaching a peak of 78.7 mg/100g at 8 WAA and then gradually decreased, reaching a minimum value of 38.9 mg/100g at physiological maturity (26 WAA) (Fig. 6). Ascorbic acid content was higher in immature oranges and grapefruits, and as fruit matured and increased in size, the concentration generally decreased (Ting and Attaway, 1971). Ladaniya and Mahalle (2011) reported that ascorbic acid content declined in 'Mosambi' oranges as the fruit matured, but the drop was not significant after 210 days. When harvested at the same time, the green colored oranges were lower in ascorbic acid content than the orange-colored fruits (Ting and Attaway, 1971).

#### CONCLUSION

Grapefruits should be harvested at least at physiological maturity, where the fruit attains maximum size and weight, rind color develops, TSS reaches >14%, total sugars above 14 g/100g fresh weight, titratable acidity is reasonably low, ascorbic acid content is reasonably high and the fruit is still firm.



Fig. 6: Changes in ascorbic acid content during growth and development of "Red Blush" grapefruits.

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# . التغيرات الكيميائية II التغيرات الفيزيائية والكيميائية أثناء نمو وتطور ثمار القريب فروت. أبوبكر علي أبوجوخ وعلى أزهرى محمد الماحى الخلاصة

قومت التغيرات الفيزيائية أثناء النمو والتطور لثمار القريب فروت أحمر اللب ذي البذور من الصنف "رد بلش"، وذلك لتوفير المعلومات الأساسية المتعلقة بكيمياء الثمار أثناء تطورها لتساعد في تحديد مرحلة اكتمال النمو لثمار القريب فروت. تبع معدل تنفس الثمار نمط التنفس غير الكلايماكتيرى، حيث انخفض من 239.8 ملجم ثاني أكسيد الكربون/كجم- ساعة (4 أسابيع بعد تفتح الأزهار) إلى 21.1 ملجم ثاني أوكسيد الكربون/كجم- ساعة (26 اسبوعاً بعد تفتح الأزهار) إلى 21.1 ملجم ثاني أوكسيد الكربون/كجم- ساعة (26 اسبوعاً بعد تفتح الأزهار). زادت نسبة المواد (4 أسابيع بعد تفتح الأزهار) إلى 21.1 ملجم ثاني أوكسيد الكربون/كجم- ساعة (26 اسبوعاً بعد تفتح الأزهار). زادت نسبة المواد الصلبة الكلية الذائبة والسكريات الكلية زيادة مطردة في الثمار من 4 أسابيع بعد تفتح الأزهار إلى 26 السبوعاً بعد تفتح الأزهار، عند مرحلة الكلية الذائبة والسكريات الكلية زيادة مطردة في الثمار من 4 أسابيع بعد تفتح الأزهار إلى 26 اسبوعاً بعد تفتح الأزهار، عند مرحلة الكلية الذائبة والسكريات الكلية زيادة مطردة في الثمار من 4 أسابيع بعد تفتح الأزهار إلى 26 اسبوعاً بعد تفتح الأزهار، عند مرحلة الكمور بيك زيادة مطردة بعد 4 أسابيع من تفتح الأزهار عدى وصلت إلى قمة بعد 16 و10 و8 أسابيع بعد تفتح الأزهار، وحامض الأسكوربيك زيادة مطردة بعد 4 أسابيع من تفتح الأزهار حتى وصلت إلى قمة بعد 16 و10 و8 أسابيع بعد تفتح الأزهار، وحامض الأسكوربيك زيادة مطردة بعد 4 أسابيع من تفتح الأزهار حتى وصلت إلى قمة بعد 16 و10 و8 أسابيع بعد تفتح الأزهار، في التوالي، ثم انخفضت بعد ذلك إلى أدنى مستوياتها عند مرحلة اكتمال النمو الفسيولوجي. ذلك يجب حصاد ثمار القريب فروت عند مرحلة اكتمال النمو الفسيولوجي. على الأزهار حتى وصلت إلى قمة بعد 16 و10 و8 أسابيع بعد تفتح الأزهار، وحامض الأسكور بيك زيادة مطردة بعد 4 أسابيع من تفتح الأزهار حتى وصلت إلى قمة بعد 16 و10 و8 أسابيع بعد تفتح الأزهار، في قاد مرحلة الأزهار حتى وصلت إلى قمة بعد 16 و10 و8 أسابيع بعد تفتح الأزهار، فروت عد مرحلة اكتمال النمو الفسيولوجي. على الأزهار حتى وصلت إلى قمار القير، فراك يعب حصاد ثمار القرب، فروت عند مرحلة اكتمال النمو الفسيولي مربع قمان الأزمار من 10 مار مرا 10 مار ما 10 مار و10 ما والفر ما 10 ماليي ما 10 ماليسية الغليم ما 10 مال مالي مالي ماليي ماليي المارم