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QUALITY CHANGES IN FRESH DATE FRUITS (BARHI) DURING INDIVIDUAL QUICK FREEZING AND CONVENTIONAL SLOW FREEZING

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Fresh date fruits (*Barhi cultivar*) at Khalal stage of maturity were frozen by two different methods; individual quick freezing (IQF), and conventional slow freezing (CSF). The frozen dates were stored for 9 months at -20°C and -40°C, through conventional and individual quick freezing methods, respectively. Texture profile analysis parameters (hardness, elasticity, chewiness and resilience), color, and nutrition (enzymes and sugars) properties for fresh and frozen dates were measured after every three months of frozen storage. IQF technique showed better result in quality maintaining of fresh date fruits than CSF. Color and textural parameters of the frozen dates were affected by the freezing method and the frozen storage period. IQF method is superior in preserving the fresh *Barhi* fruits color values, textural parameters and nutritional aspects compared to CSF method.
Keywords: individual quick freezing, textural parameters, freezing method

INTRODUCTION

The date palm (*Phoenix dactylifera* L.) is a dominant tree in South West Asia and North Africa. Presently global production, consumption and industrial development of dates are constantly growing as date fruits are important source of energy, essential nutrients and possess some medicinal benefits (Al Farsi and Lee, 2008; Al-Abdoulhadi *et al*., 2011; Chandrasekaran and Bahkali, 2013; Naqvi *et al*., 2015). There is a necessity to utilize freezing and frozen storage for prolonging the shelf life of fresh date fruits, especially *Barhi* cultivar, which is favored and widely consumed at Khalal maturity stage where the fruit is sweet, crispy and yellow in color.

Color plays a fundamental part in the consumers' evaluation of the food quality. The major quality attribute that affects consumers' selection is the change in color (Zhang *et al*., 2004). Enzymatic oxidation of phenolic substances is the main reason for color changing. Ice crystals formed during freezing enhance enzymatic oxidation due to the destruction the cells and tissues of the product and therefore increased contact between phenolics, oxygen and enzymes (Ruenroengklin *et al*., 2008).

Textural parameters of frozen foods play an essential part in determining the acceptability of these products by consumers. Higher values of hardness, chewiness and resilience of the pulp indicate better quality products (Zhang *et al*., 2007; Krause *et al*., 2008; Kaushik *et al*., 2013). Physical modifications in frozen fruits include displacement of water, strains in cell size, mechanical injury and cracking. Several researchers have studied the effects of freezing on textural quality of fruits (Delgado and Rubiolo, 2005; Van Buggenhout, *et al*., 2006; Sousa, *et al*., 2007).

Date fruits, irrespective to the cultivar; contain more than 75% sugars on a dry-weight basis (Kanner *et al.*, 1978). Fruit sugars have a significant part in preserving fruit quality and determining its nutritional status (Akhatou and Recamales, 2013). Al-Mashhadi *et al*. (1993) studied the effects of freezing and frozen storage on the sugars of Khudry and Munifi date cultivars. They found that the fructose and glucose sugars increased while sucrose sugar decreased after 12 months of frozen storage at -18°C, -15°C and -10°C.

Freezing only helps to reduce the activity of enzymes (Maier *et al*., 1964; Whitaker, 1994; Marin and Cano, 1992). Invertase, Peroxidase (POD) and Polyphenol oxidase (PPO) are essential enzymes responsible for the quality deterioration in most of the frozen fruit (Whitaker, 1994). The activity of these enzymes decreases as the temperature decrease. However, the chemical reactions reduce more rapidly with the addition of salt during freezing process.

Mikki and Al-Taisan (1993) investigated the physicochemical variations of date fruits due to freezing at -18°C. The frozen storage period was six months during which thawed samples were analyzed periodically to determine physicochemical changes and carry out sensory evaluation to judge changes in texture, color, and taste attributes. The study concluded that frozen fruits have shown an increase in their moisture content, while reducing sugars, pH, and tannin contents decreased at the end of storage period.

It seems that so far there are limited research works reported on the freezing of fresh dates. Thus this research work deals with the study of the utilization and comparison of two freezing methods *viz*. individual quick freezing and conventional deep freezing on the quality and stability of date fruits (*Barhi cultivar*) at Khalal stage.

MATERIALS AND METHODS

***Fresh dates*:** Fresh yellow dates (cv. *Barhi*) at Khalal stage of maturity were obtained from a commercial farm in Qassim, Saudi Arabia. Dates were sorted to discard the damaged fruits and immediately kept for less than 6 h in a cold store at 5°C.

***Physical properties, moisture content and water activity determination*:** The physical properties of fruits (length and diameter; surface area; volume; mass; density), water activity, moisture content, color, texture profile analysis (hardness, elasticity, chewiness and resilience), and nutrition (enzymes, sugars) properties for fresh fruits were evaluated for fresh fruit and for the frozen ones after thawing every three months. The moisture content of dates flesh was determined using AOAC procedures (AOAC, 2005) where the samples were dried at 70°C for 48 hours under a vacuum of 200 mm of mercury (Vacutherm model VT 6025, Heraeus Instrument, D-63450, Hanauer, Germany) (Hassan, 2005). Water activity of the dates flesh was measured at room temperature using Aqua-lab (Model CX-2T, readability 1 mg, Decagon Devices Inc., Washington) (Hassan, 2005).

***Color examination*:** The fruits color values were expressed by the parameters (L*, a*, b*): Where L* indicates (whiteness or brightness/darkness), a*(redness/greenness) and b^*(yellowness/blueness) measured using a color meter (Color Flex, Model No.45/0, Hunter Associates Laboratory Inc., VA, USA). In addition the color was expressed by the total color difference (ΔE), Chroma, hue angle and browning index (BI) as defined by the following equations (Maskan, 2001):

$$\Delta E = \sqrt{(L^*{}_0 - L^*)^2 + (a^*{}_0 - a^*)^2 + (b^*{}_0 - b^*)^2} \quad (1)$$

Where: L^*_0, $a^*_0$and b^*_0 are the color parameters of fresh fruits (before freezing).

$$Chroma = \left(a^{*2} + b^{*2}\right)^{0.5} \quad (2)$$

$$Hueangle = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (3)$$

$$BI = \frac{[100(x - 0.31)]}{0.17} \quad (4)$$

Where: $$x = \frac{(a^* + 1.75L^*)}{(5.645L^* + a^* - 3.012b^*)} \quad (5)$$

***Texture profile analysis (TPA)*:** The texture profile analysis parameters were measured using a texture analyzer (TA-HDi, Model HD3128, Stable Micro Systems, Surrey, England) (Hassan, 2005). Fruit samples were compressed with a rod velocity of 1.5 mm/s to a depth of 5 mm. The compression was done twice to give two complete texture profile curves. The force-time deformation curves were obtained in which the following parameters were obtained: Hardness (the maximum force required to compress the sample), Resilience (the ability of the sample to recover its original form after deformation), Chewiness (the work needed to be done to make a solid food swallow-able, which is numerically formulated by the product of gumminess and springiness). The data was processed using Texture Expert Exceed, version 2.05 (Stable Micro systems) (Hassan, 2005).

***Extraction and estimation of invertase enzymes*:** The method was used to extract the invertase enzyme by mixing a sample (15 g) with NaCl solution (4%) containing 1 g polyvinyl pyrotidone for two min. at a temperature of 2°C and then centrifuged at 2000 rpm for 30 min. at 5°C. The upper liquid layer containing the enzyme (supernatant) was collected and kept for enzyme assay. The previous steps were repeated again on the remaining solid material and then the superrantant was taken and added to the previous amount. This solution is soluble invertase. The insoluble invertase was obtained by water dialysis of the extraction residues at 2°C till all sugars were removed.

Invertase activity in *Barhi* dates at Khalal stage and during frozen storage was measured by a method based on Kanner *et al*. (1978). The assay mixture included 0.5 Macetate buffer pH 4.5, 1.5 M sucrose and enzyme extract (1 mL) with total volume of 5 mL. This mixture was kept for 1 h at 37°C and I mL sample was withdrawn at 10 min interval. One unit of invertase was defined as the amount of enzyme, which hydrolyzed 0.5 µM sucrose per min under the above conditions.

***Extraction and estimation of peroxidase and polyphenol oxidase enzymes*:** Peroxidase and polyphenol oxidase enzymes were extracted and estimated according to the method used by Cano *et al*. (1995). Each enzyme was extracted by 0.2M sodium phosphate buffer and 1M sodium chloride at 5°C then filtered and centrifuged and the supernatant was collected. The amount of enzymes in the samples was measured using the polarization device (Polarimeter, Autopol IV Six Wavelength) manufactured by Rudolph Research, USA.

***Sugar analysis*:** Sugar analysis (fructose, glucose, and sucrose) of dates was determined by the AOAC standard procedure (AOAC, 2005) using high-performance liquid chromatography system (HPLC), LC-10 AD, Shimadzu Corporation, Kyoto, Japan.

***Freezing methods*:** Two methods were used in freezing the fresh date fruits, individual quick freezing and conventional slow freezing using traditional deep freezers.

***Individual Quick Freezing (IQF) method*:** Individual Quick Freezing using air as a medium to freeze (Advanced I.Q.F Spiral Freezer, Advanced Equipment Inc., Richmond, BC, Canada) was utilized to freeze the fresh dates. After the freezer reached steady state, fresh fruits were fed inside the freezer at a rate of 2 kg/min and the frozen fruits go out from the freezer after 34 minutes for its entry. The temperature of the fruits pulp (near the pit) and the surrounding air inside the freezer were measure every 5 seconds using type K thermocouples connected with data Loggers. The freezer was set at -43°C inside the freezer. The frozen fruits were collected at the exit point of the freezer and packaged in rigid polyethylene boxes (½ kg capacity) and directly stored in ultra-freezers at -40°C.

***Conventional Slow Freezing (CSF) using deep freezers method*:** A traditional deep freezer (Chest Freezer, Sanyo Elec. Co., Ora / Gun Japan) was used for the conventional freezing of fresh fruits. The fruits were filled in rigid polyethylene plastic boxes (½ kg capacity). Thermocouples were placed to measure the temperature of the fruits pulp (near the pit) and the temperature inside the freezer every 5 minutes. Freezers' temperature ranged between -18 to -24°C at steady state. However, it was assumed that the average freezer temperature is -20°C.

***Thawing processes*:** The frozen fruits were removed from the storage freezers and thawed by leaving them at room temperature for two hours.

***Statistical analysis*:** All needed analyses were performed using the IBM SPSS software package (IBM SPSS version 22). Experimental data were analyzed by means of analysis of variance (ANOVA) and mean separation was by Duncan's multiple range test at $P< 0.05$.

RESULTS AND DISCUSSION

Physical properties, moisture content and water activity*:** ***The data obtained on the physical properties of the fresh *Barhi* fruits are shown in Table 1. The average mass of the fruits of fresh *Barhi* fruits is 14.88 g. The average density values of the pulp of the fresh fruits, which is important for the calculation of temperature distribution within the fruit, were higher than the density of water as shown in Table 2.

Table 2 also shows the experimental data on the moisture content (wet basis) and water activity of the fresh *Barhi* fruits. The fresh *Barhi* fruits are characterized with high moisture content (68.6%) and water activity (0.91) values. This indicates that *Barhi* fruits were highly perishable.

***Color of fresh and frozen fruits*:** The experimental data on the basic color parameters (L*, a* and b*) of the *Barhi* fruits frozen by two methods, Individual Quick Freezing (IQF) and Conventional Slow Freezing (CSF) as a function of time are shown in Figure 1.

The mean values of the basic color parameters L*, a* and b* of the fresh *Barhi* fruits were 68.35, 10.87 and 48.44, respectively. These values show that *Barhi* fresh fruits at Khalal stage of maturity are characterized with their bright yellow color. From Figure 1 it is clear that the L*, a* and b* values had changed for frozen fruits with the period of frozen storage which extended for nine months. The L*values of the frozen fruits decreased at the end of the frozen storage to 50 and 43.7 in IQF and CSF, respectively. Whereas, a* values increased during the same period to 14.8 and 15.4 in IQF and CSF, respectively. The b*values followed the same behavior of L* and decreased to 38.2 and 33.9 in IQF and CSF, respectively. These results indicate that there were notable changes in the values of the basic color parameters of the fresh *Barhi* fruits after freezing and frozen storage in both freezing

Table 1. Average value of physical properties of the fresh *Barhi* fruits at Khalal stage of maturity.

	Mass (g)	Volume (cm^3)	Density ($g\ cm^{-3}$)	Area (cm^2)	Length (mm)	Larger diameter (mm)	diameter neck fruit (mm)	diameter tip fruit (mm)
Average ($\overline{X}$)	14.88	14.54	1.029	27.95	34.26	27.51	21.74	19.17
SE	0.338	0.379	0.006	0.551	0.258	0.254	0.168	0.168
SD	2.619	2.933	0.045	3.015	1.996	1.964	1.305	1.301
No of samples	60	60	60	30	60	60	60	60

SE = Standard error, SD = Standard deviation

Table 2. Mean values of pulp density, moisture content and pulp water activity for the fresh *Barhi* fruits at Khalal stage of maturity.

	Thickness of pulp (mm)	Density of pulp (cm^3)	Moisture content (w.b.) %	Water activity
Average ($\overline{X}$)	8.062	1.182	68.631	0.911
SE	0.467	0.018	1.724	0.022
SD	1.402	0.055	5.172	0.066
No. of samples	9	9	9	9

SE = Standard error, SD = Standard deviation methods. Nevertheless, IQF method is superior in preserving the fresh Barhi fruits basic color parameters compared to CSF method. This can be attributed to that freezing always causes physical and chemical changes in food due to ice formation (Reid, 1996). At slow freezing rates, such as in CSF, big ice crystals are formed mainly in the extracellular space of fruits leading to higher pigment diffusion. In contrast, quick freezing rates in case of IQF is associated with the formation of tiny ice crystals in the intracellular space thus leading to less pigment diffusion (Van Buggenhout *et al*., 2009).

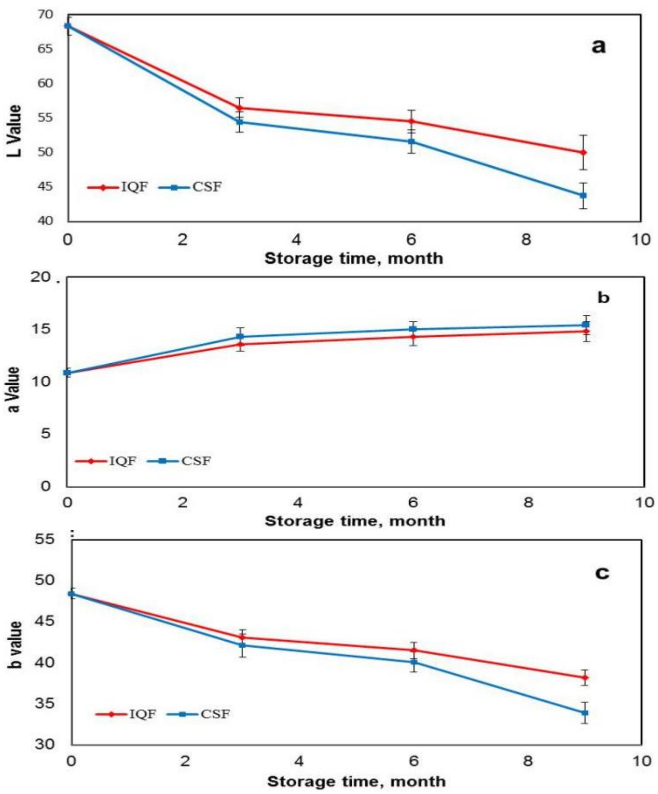

Figure 1. Changes of basic color parameters values of *Barhi* fruits frozen by IQF and CSF at different storage times. (a) L*, (b) a* and (c) b*. (Mean values ± standard deviation of ten replicate measurements are shown)**.**

The data on the color derivative parameters i.e. color difference (ΔE), Chroma, hue angle and browning index (BI) of the fruits frozen by IQF and CSF methods are plotted and depicted in Figure 2. The color derivative parameters values of the fruits frozen by IQF method varied depending on the period of the frozen storage. The results of the CSF method indicates a large decrease in the values of L* and b* after 9 months of frozen storage. This has led to a significant decrease in Chroma and the hue angle values and an increase in the color difference and browning index compared to the individual quick freezing. This may be due to reduced tissue damage resulting in lowered pigment diffusion (Delgado and Rubiolo, 2005). These results were similar to those observed by other researchers (Duanet *et al*., 2007; Ruenroengklin *et al*., 2008; Neog and Saikia, 2010).

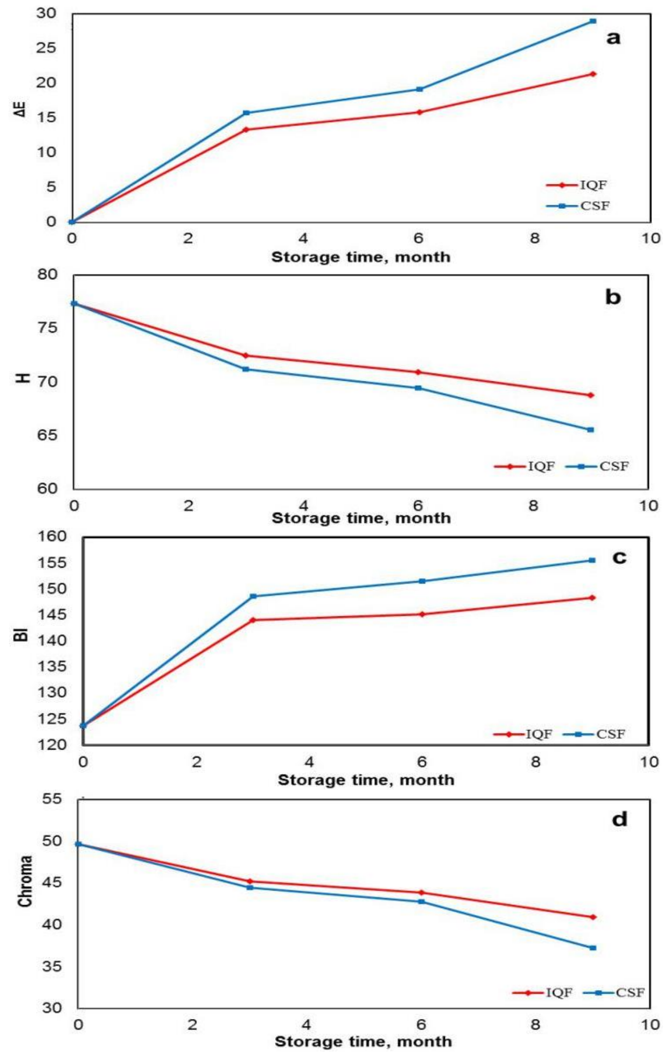

Figure 2. Changes of color derivative parameters of *Barhi* fruits frozen by IQF and CSF at different storage times. (a) Color deference (ΔE), (b) Chroma, (c) Hue angle and (d) BI.

***Textural parameters*:** The textural profile analysis (TPA) parameters for the fresh fruits and frozen fruits (after thawing) during 9 months of frozen storage are shown in Figure 3. The results indicate that fresh *Barhi* fruits are distinguished with their higher mechanical properties. The results also depict that fresh *Barhi* fruit is firm as revealed by its high average hardness value (119.48 N).

The textural profile analysis (TPA) parameters of the frozen fruits were highly influenced by freezing method and frozen storage time. In general, all tested TPA parameters values of the fruits frozen were lower compared to the fresh ones. There are high changes of texture during the first three months of storage. However, the changes tend to be decrease during the remaining period of storage.

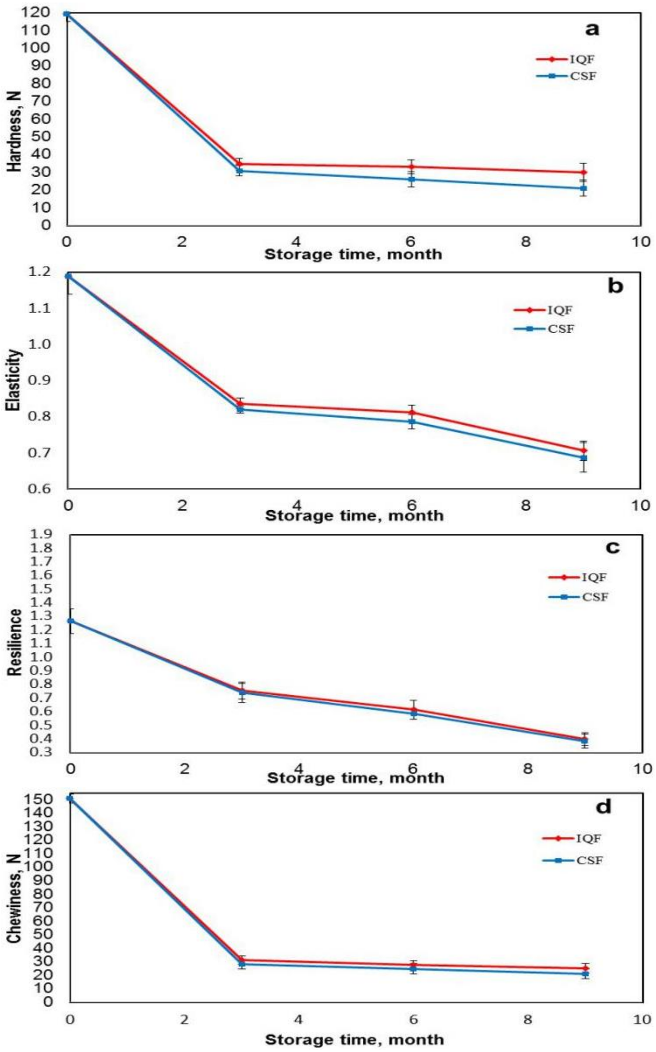

Figure 3. Effects of freezing methods in the TPA parameters of *Barhi* fruits at different storage times. (a) Hardness, (b) Elasticity, (c) Resilience and (d) Chewiness. (Mean values ± standard deviation of ten replicate measurements are shown).

As displayed in Figure 3a the reduction in hardness values of the fruits during the first three months of storage is 71% for IQF method, while it is 74.17% for CSF method. It is also noted that the hardness of the fruits frozen with IQF method decrease by 13.17% during storage period of 3 to 9 months, while the decline was much larger in the case of CSF method where it decease by 32%. The reduction in the elasticity values for the fruits frozen by both IQF and CSF methods is illustrated in Figure 3b. It is clear that the decline is at higher rate in CSF compared to IQF method. The resilience property of the frozen fruits decreased during frozen storage for both freezing methods. Figure 3c displays that the resilience of fresh fruits decreased by 40.27% for IQF and 41.69% for CSF method during the three first months of storage. The chewiness values of fresh fruits decreased during the same period of storage by 79.27% for IQF and 81.39 for CSF as given in Figure 3d. In addition, the elasticity of fresh fruits decreased during the three first months of storage by 29.55% for IQF and 32% for CSF as shown in Figure 3b.

The above mentioned results showed that IQF method is preferable as compared to CSF method since deterioration in texture changes is less for the first method and closer to natural texture of *Barhi* fresh fruits. This is probably due to decrease of the injurious effects of crystallization and recrystallization on the microstructure of *Barhi* tissues during such quick freezing, frozen storage and thawing using IQF method. The significant role of the freezing rate in maintaining the texture of frozen foods was also reported by other studies (Sanz *et al*., 1999; Sun and Li, 2003; Zhang *et al*., 2004; Delgado and Rubiolo, 2005; Van Buggenhout *et al*., 2006; Sousa *et al*., 2007).

Effects of freezing methods on the enzymatic activity of Barhi fruits**:** Invertase enzyme activity was not detected in Brahi fruits (Khalal stage). This may be due to the temperature at which the dialysis process of the extract was done. Another possibility is that the invertase enzyme exists at minute quantities in the cultivar under study to the extent that it was out of the detection limits.

The enzymatic activity of both polyphenols oxidase (PPO) and peroxidase (POD) in *Barhi* fruits throughout frozen storage period (9 months) is shown in Figure 4.

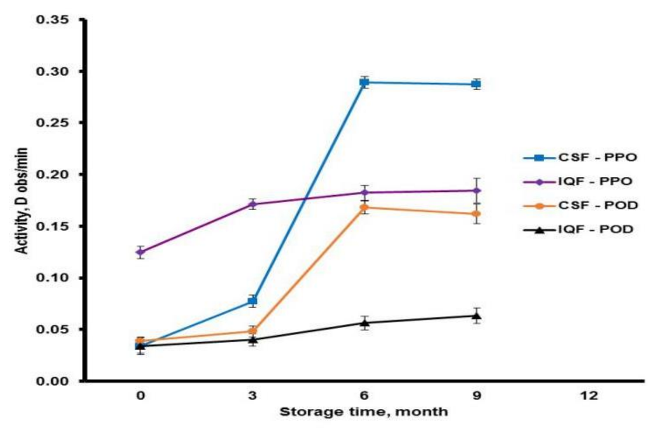

Figure 4. Effects of freezing methods in the enzymes activity of *Barhi* fruits at different storage times. (Mean values ± standard deviation of ten replicate measurements are shown).

It is evident from this figure there is an increase in the activity of both enzymes in the two freezing methods, but the increase in CSF is greater. This may be due to the enzymatic activity in the fruits as a result of no use of blanching treatment. Moreover, it is noted that the activity of both enzymes increased at a slower rate in the first three months. Then the activity increased dramatically during the next three months, while it was almost constant during the last three months. As for IQF method the activity of the two enzymes increased, but at very small rates during the frozen storage for a period of nine months. These enzyme activities might be the cause of deterioration of fruit texture and color during storage (Maier *et al*., 1964; Whitaker, 1994; Marin and Cano, 1992). Moreover, the enzyme activities are more active when freezing with CSF compared to IQF which might indicate the slower activities of IQF that led to retention of fruit properties more than that of CSF.

***Effects of freezing methods on Barhi fruits' sugars*:** Figure 5 displays the effects of IQF, CSF methods and frozen storage period on the fresh *Barhi* fruits sugars. From the figure it is clear that glucose and fructose proportions increased during the first three months of frozen storage for both methods; however, the increase in case of IQF was at slower and regular rate. Then glucose and fructose sugars decreased until the end of the frozen storage period for both methods. The increase in the reducing sugars (glucose and fructose) during the first period of frozen storage could be due to easier extraction after cell-wall rupture caused by ice crystal formation. This increase was also observed by Al-Mashhadi *et al.* (1993) for date fruits and is in agreement with previously reported studies in mango cultivars (Marin *et al*., 1992).

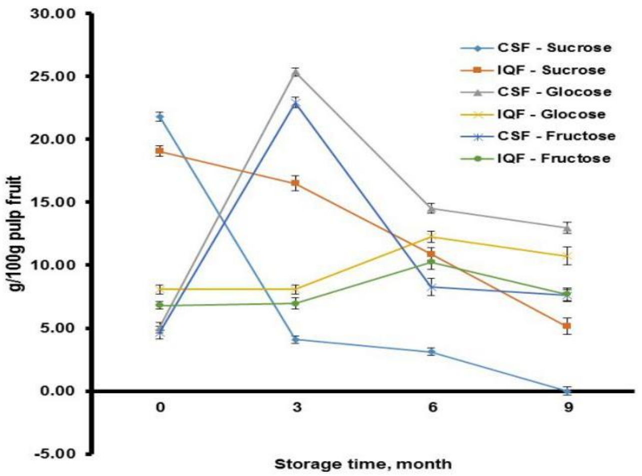

Figure 5. Effects of freezing methods on *Barhi* fruits' sugars at different storage times. (Mean values ± standard deviation of ten replicate measurements are shown).

The sucrose percentage in the frozen fruits decreased gradually till the end of the frozen storage period for both methods. The increase in the reducing sugars (fructose and glucose) perceived during the first period of frozen storage for both studied freezing methods. The reduction in the reducing sugars that was detected during the last period of storage was formerly identified by Mikki and Al-Taisan (1993).

From Figure 5 it is evident that the changes in sugars were much lower in case of fruits frozen by IQF method and stored at -40°C compared to those frozen by CSF method and stored at -20°C. This might be due to the higher damage of the cell-wall in case of CSF caused by bigger ice crystals (Marin *et al*., 1992).

***Fruits freezing curves*:** Freezing curves of fresh *Barhi* fruits during IQF and CSF are shown in Figure 6 and 7, respectively. The curves represent the change of the fruit center and its surrounding air temperature with time. From the figures of the freezing curves it can be noted that temperature distribution curves showed a high variation of freezing times of the two examined systems. The time to reach the fruit center for IQF was 60 minute whereas it was 1800 minute for CSF.

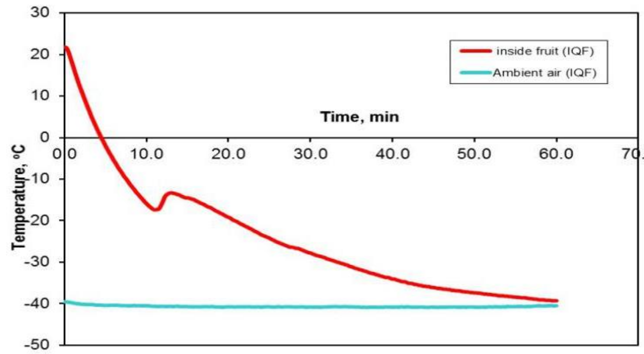

Figure 6. Temperature change with time during IQF of *Barhi* fruits.

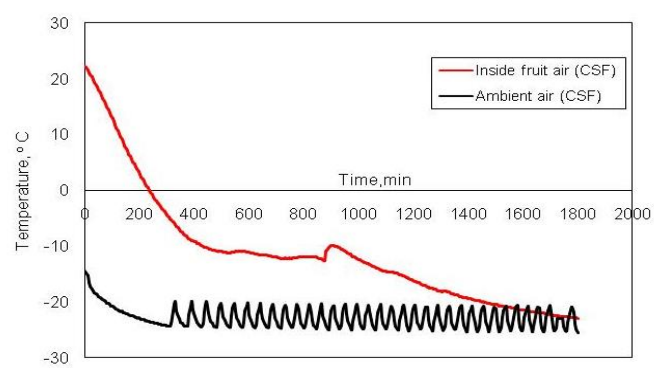

Figure 7. Temperature change with time during CSF of *Barhi* fruits.

***Conclusions*:** Freezing and frozen storage showed a clear effect on basic color values (L*, a* and b*) and their derivative parameters of the *barh*i fruits. The textural properties of the frozen fruits were greatly influenced; in general, textural properties decreased with storage time and were dependent on freezing method. Analysis of basic sugars (fructose, glucose, and sucrose) in the fruits showed a sharp increase in fructose and glucose along with decreases in sucrose for fruits until the end of the frozen storage period. Enzymatic activities led to more deterioration of fruits quality with storage time especially with CSF. IQF is superior in preserving the fruit fresh quality compared to CSF due to the fast freezing.

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