

## **Cryogenic Freezing of Fresh Soft Dates**

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**Abstract.** Seven cultivars of widely consumed fresh soft dates, at two stages of maturity, i.e., Rutab (fully ripe) and Munassif (half ripe), have been cryogenically frozen using liquid nitrogen. The cultivars were: Sukkari, Nabt Sultan, Nabt Saif, Maneefi, Sillaj, Reziz, and Khalas, all purchased fresh from the local market during the 1989 date production season. The experimental time-temperature data of the fresh soft dates during the cryogenic freezing process were generated and compared with the conventional freezing time-temperature data. The initial freezing points of the date cultivars were in the range  $-16$  to  $-18^{\circ}\text{C}$ . Quality attributes of the thawed dates kept frozen at  $-50^{\circ}\text{C}$  for 12 months were compared with their fresh cultivars counterparts. Significant differences ( $P < 0.05$ ) were detected by sensory panelists, in color, texture, flavor, and overall acceptability of most cryogenically frozen cultivars at their Munassif stage of maturity. Panelists were not able to detect significant differences ( $P < 0.05$ ) while scoring for the overall acceptability of all cultivars at their Rutab stage of maturity, except Nabt Saif. Cryogenically frozen Nabt Saif cultivar at the Munassif stage of maturity was not significantly different ( $P < 0.05$ ) from its fresh one in color, texture, flavor, and overall acceptability.

### **Introduction**

Cryogenic freezing refers to very rapid freezing achieved by exposing food items, unpackaged or thinly packaged to an extremely cold freezant undergoing a change of state. Heat removal which is accomplished during a change of state is used to distinguish cryogenic freezing from liquid immersion freezing. The most common food grade cryogenic freezants are boiling nitrogen and boiling or subliming carbon dioxide [1,2]. In most instances the rapid freezing rate achieved by cryogenic freezing results in numerous small ice crystals which in turn has been claimed to lead to less rupturing of cell walls and subsequently a reduced drip loss of cellular fluids. It is generally accepted that the quality of cryogenically frozen food equals or exceeds that of the conventionally frozen foods [3].

Conventional freezing of fresh soft dates is commonly practised at home by consumers in Saudi Arabia to secure a continuous supply of fresh soft dates during off-season which extends for about nine months. Fresh soft dates produced in Indio,

California (USA), has been experimentally frozen as early as 1955 and made an excellent fresh frozen product which was successfully preserved for three years at  $-30^{\circ}\text{C}$  [4]. The quality of the food system following freezing process depends to a high extent on the variety chosen, growing conditions and stage of maturity at harvest [5,6].

Published investigations on freezing of fresh soft dates are scarce. However, a few investigations on cold storage, frozen storage, and freezing maturation of fresh soft dates are available [7,8].

The objectives of this study were to cryogenically freeze selected common cultivars of fresh soft dates to establish cryogenic time-temperature relationships, compare data with conventional freezing, determine initial freezing points, and to evaluate the sensory quality attributes of the cultivars following 12 months of frozen storage at  $-60^{\circ}\text{C}$ .

### Materials and Methods

#### Cryogenic freezing

Seven cultivars of selected fresh soft dates at two stages of maturity, i.e. Munassif and Rutab, were purchased from the local market during the 1989 date production season and used in this study. The cultivars were Sukkari, Nabt Sultan, Nabt Sarf, Maneem, Silaj, Reziz, and Khalas. The fresh cultivars were stored in a well designed, temperature-controlled cold storage room at  $5 \pm 1^{\circ}\text{C}$ , for a maximum of two days prior to the freezing process to assure minimal changes of quality attributes. The purchased fresh dates were packed in cartons containing about 15 kg of each cultivar. Munassif and Rutab stages of each cultivar were hand picked with careful selection of whole, uninjured, good quality dates.

The cryogenic freezing experiments were performed using an integrated liquid nitrogen cryogenic freezing system (Model 990 freezing chamber CMS 328, with LI-450, integrated with model 1010A Microcomputer programmable freezing controller, CRYOMED of a company in Michigan, USA). The system operates by the injection of liquid nitrogen, and a blower located at the side of the freezing chamber, circulates gaseous nitrogen to assure uniform cooling and freezing effects. A liquid nitrogen transfer hose and pressure relief set at 151.7 KPa (22 psig), connect to an electric solenoid valve to supply coolant to the freezing chamber on the command of the controller.

Unpackaged fresh soft dates were placed in stainless steel freezing racks inside the freezing chamber. In each cryogenic freezing experiment conducted, four perfo-

rated freezing racks were used to hold 1 kg per rack of each stage for each of the two different cultivars used per experiment. Temperatures of the dates and gaseous liquid nitrogen inside the freezing chamber were measured as a function of time during the cryogenic freezing process, using type T (Cu/Co), Teflon coated 24-gauge thermocouples (Omega Engineering, Inc., Stanford, CA, USA). The thermocouples tips were inserted inside the flesh of the fully and half ripe dates to a depth approximately equal to 3 mm through the surface and towards the interior of the date flesh. Three dates in each rack were selected for time-temperature measurements during the cryogenic freezing process. Two thermocouple wires were hung loosely inside the chamber for time-temperature measurements of the freezing medium.

All thermocouples leads were connected to a data acquisition system (HP 30555 personal computer based data acquisition system, Hewlett Packard, P.O. Box 10301, Palo Alto, USA) for automatic data collection, storage and analysis. The integrated freezing system is illustrated in Fig. 1. The properties of the cryogenic freezant, liquid nitrogen, are shown in Table 1. Each cryogenic freezing experiment was continued for about 12 min. Cryogenically frozen dates were instantly packed and sealed in polyethelene bags and immediately transferred to a temperature controlled ultra freezer set at  $-50^{\circ}\text{C}$  (GFL, Gesellschaft fur Lab ortechnik m.b.H, D 3006 Burgwedel, Germany).

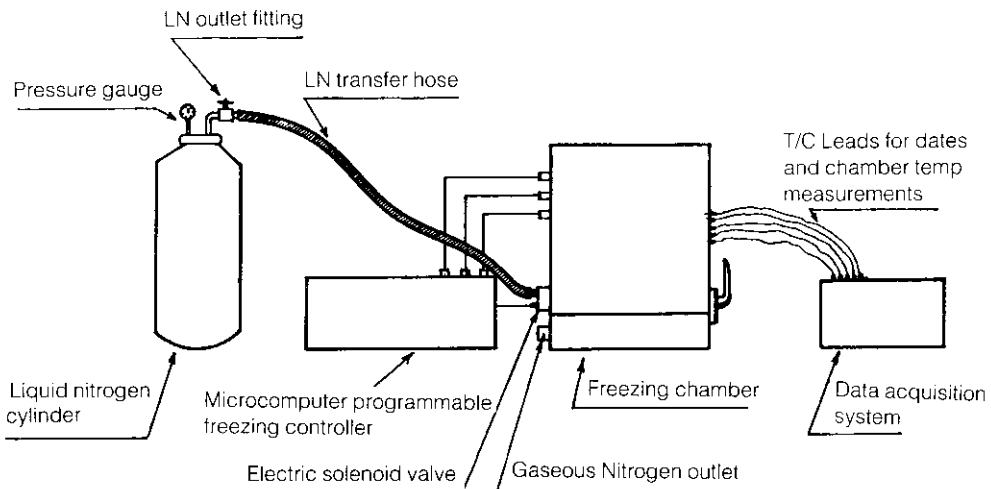


Fig. 1. The integrated cryogenic freezing system.

**Table 1. Properties of the cryogenic freezant, liquid nitrogen\*.**

Mwt	Boiling point at 1 atm (°C)	Latent heat at boiling point (KJ/Kg)	Specific heat, gas at 1 atm (KJ/Kg °C)	Usable heat** capacity (KJ/Kg)
28.016	-195.81	199.55	1.0244	374.45

\*From Fennema, 1975

\*\*Usable heat capacity = Latent heat + (Specific heat x  $\Delta T$ ).

$\Delta T = 28.88^{\circ}\text{C} - (\text{Temperature of boiling})$ .

### Conventional freezing

A pilot scale deep freezer (Model No. SCD-93), SCHAEFER Corporation, 174 Schaefer Drive, P.O. Box 100, Madison, Alabama 35758, U.S.A.) was used to simulate the slow freezing process as practised by consumers at home in Saudi Arabia. Polyethelene bags were used to package 1 kg samples of each of the seven cultivars at each of the two stages of maturity. Thermocouples were attached to two samples of dates in each package, as previously described in the cryogenic freezing section-, and two thermocouples were attached to the interior of the deep freezer. Time-temperature data were collected, stored, and analyzed using the data acquisition system.

### Sensory evaluation

A set of twenty semi trained panelists consisting of staff members and research and teaching assistants from the Food Science and Agricultural Engineering Departments, participated in the sensory tests. The tests continued for four consecutive days from 10:30 a.m. to 12 noon. Two cultivars per day were tested during the first three days, and one cultivar during the fourth day. Fresh soft dates of the same cultivars were purchased from the local market during the 1990 date production season to compare with those obtained during 1989 production season and kept frozen at  $-50^{\circ}\text{C}$  for 12 months. The cryogenically frozen dates were thawed at room temperature ( $25^{\circ}\text{C}$ ) for about 90 min prior to the beginning of each session. In each session, each panelist was supplied with fresh and thawed soft dates at their two stages of maturity. All tests were carried out at a diffused light room, and water was provided for mouth rinsing between samples. Each sample was coded by a random number, and panel scores were based on a nine point hedonic scale, with 9 representing like extremely and 1 representing dislike extremely [9]. The quality attributes tested were color, flavor, texture and overall acceptability.

### Statistical analysis

Data was analyzed using a statistical analysis software package [10] at the main frame computer system of King Saud University. General linear model (GLM) along with Duncan's Multiple Range Test (DMRT) were used to analyze data and to determine the significant differences among treatment means.

### Results and Discussion

The pattern of temperature changes that fresh soft dates undergo during any freezing process is considered as one of the most critical factors associated with adequate design or selection of suitable freezing systems to ensure optimum product quality. The experimental time-temperature data during the cryogenic freezing process of the seven fresh soft date cultivars at their two stages of maturity are illustrated in Figs. 2-5. In all the cryogenic freezing experiments conducted, the freezing chamber temperature dropped from 20°C to -120°C in about 5.5 min. At the same time the slowest freezing date cultivar reached at least -20°C at the point of the thermocouple junction within its interior. From the freezing curves of Figs. 2-5, ranges

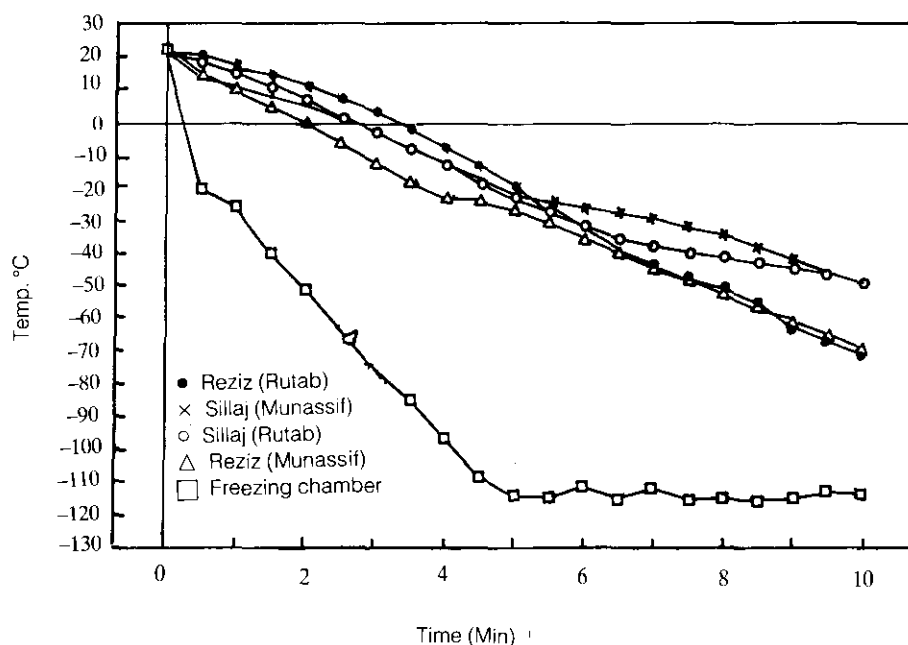


Fig. 2. Cryogenic freezing curves for the cultivars Reziz and Sillaj at their two stages of maturity.

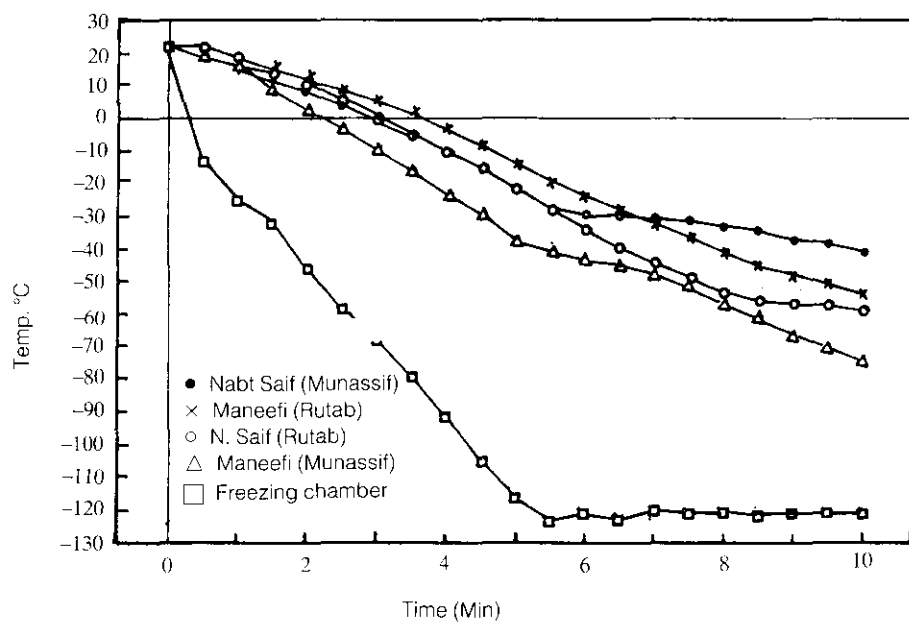


Fig. 3. Cryogenic freezing curves for the cultivars Nabt Saif and Maneefi at their two stages of maturity.

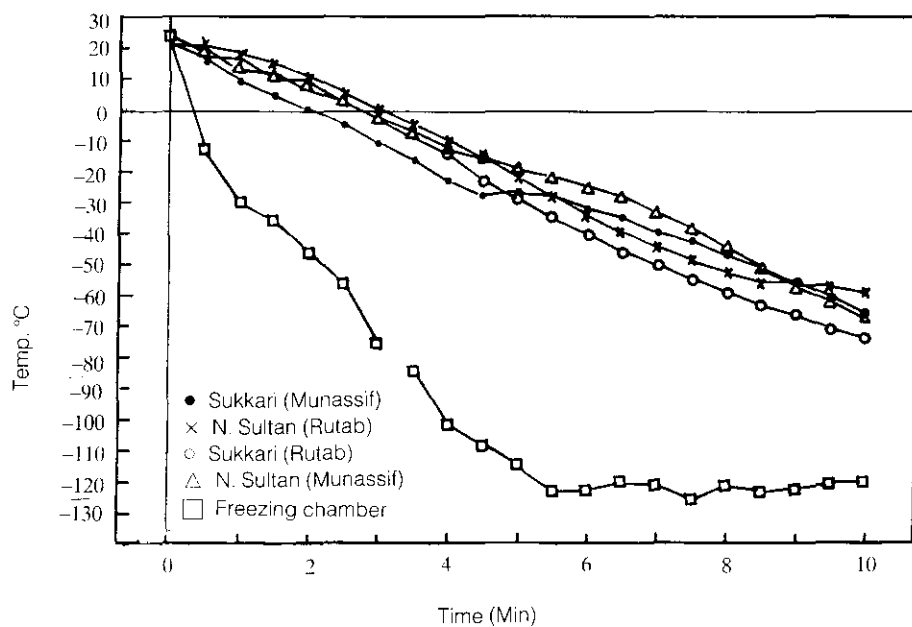


Fig. 4. Cryogenic freezing curves for the cultivars Sukkari and Nabt Sultan at their two stages of maturity.

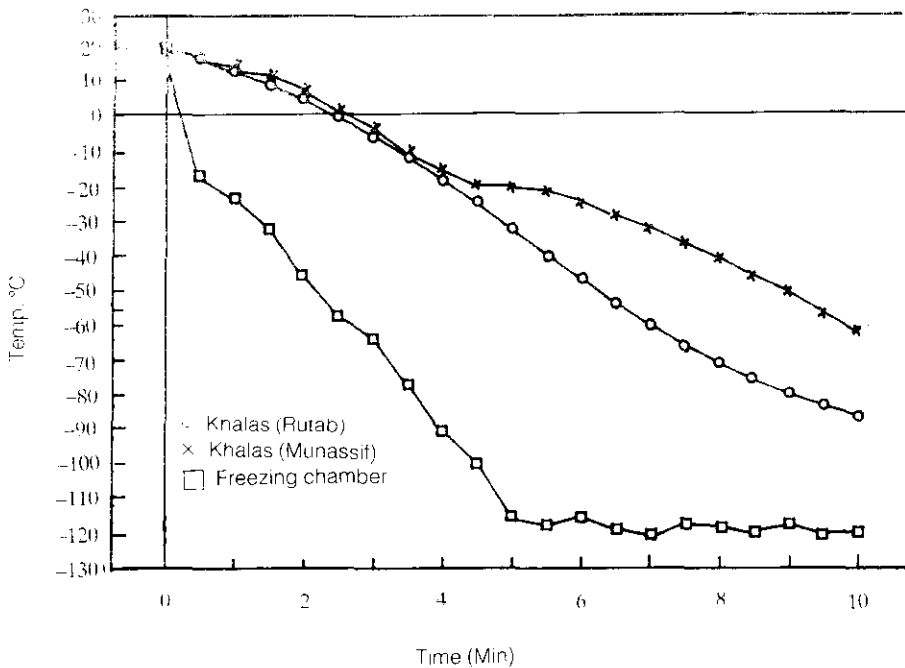


Fig. 5. Cryogenic freezing curves for the cultivar Khalas at its two stages of maturity.

of times needed for the seven date cultivars to drop to temperatures equal to  $-30^{\circ}\text{C}$ ,  $-40^{\circ}\text{C}$ , and  $-50^{\circ}\text{C}$  are 5-7 min., 5.5-9 min., and 6-10 min., respectively. Since heat removal is extremely rapid, pattern differences between cooling and freezing stages are not distinct within the time-temperature curves. Due to the higher water content of the Munassif stage as compared to Rutab, it was expected that fresh soft dates at the Munassif stage will freeze faster. However, a significant variation was not readily apparent, except for the cultivar Khalas, where unexpectedly its Rutab stage of maturity was consistently lower in temperature as compared to the Munassif stage. Despite the fairly consistent patterns of the time-temperature curves for all the cultivars at their two stages of maturity, the major uncertainty in the temperature measurements was the position of the thermocouple junction inside the interior flesh of dates.

The cryogenic frozen dates were hard solid, yellow or yellowish-brown in color, with some volume contraction observed in some of the frozen samples. In most cases, the color changed to yellow, especially for the Munassif stage of maturity, and distinction between the fully ripe part (brown) and the half ripe part (yellow) was difficult. However, during the process of thawing for the Munassif stage, the frozen

date slowly retained its original fresh color. The same observation was noticed for the Rutab stage of all date cultivars. Time-temperature curves for the conventional freezing runs of the seven fresh soft date cultivars at their two stages of maturity are shown in Figs. 6-9. The temperature of the deep freezer fluctuated between  $-20$  to  $-29^{\circ}\text{C}$  throughout the experiment. Freezing time of dates to reach  $-20^{\circ}\text{C}$  ranged between 4 and 5.5 hr.

Experimental attempts were carried out to determine the initial freezing point (or freezing point depression) for all date cultivars at their two stages of maturity. Neither direct cryogenic freezing of fresh soft dates, nor using date homogenates in cryotubes with and without added water was successful in distinctly determining the initial freezing point from freezing curves. However, part of the traditional freezing data (slow freezing) were analyzed graphically to determine the distinct initial freezing point region, and results of three cultivars, Khalas, Sukkari, and Reziz are presented in Figs. 10, 11 and 12. These curves suggest that the initial freezing point is within the range  $-16$  to  $-18^{\circ}\text{C}$ . However, a more detailed study is needed.

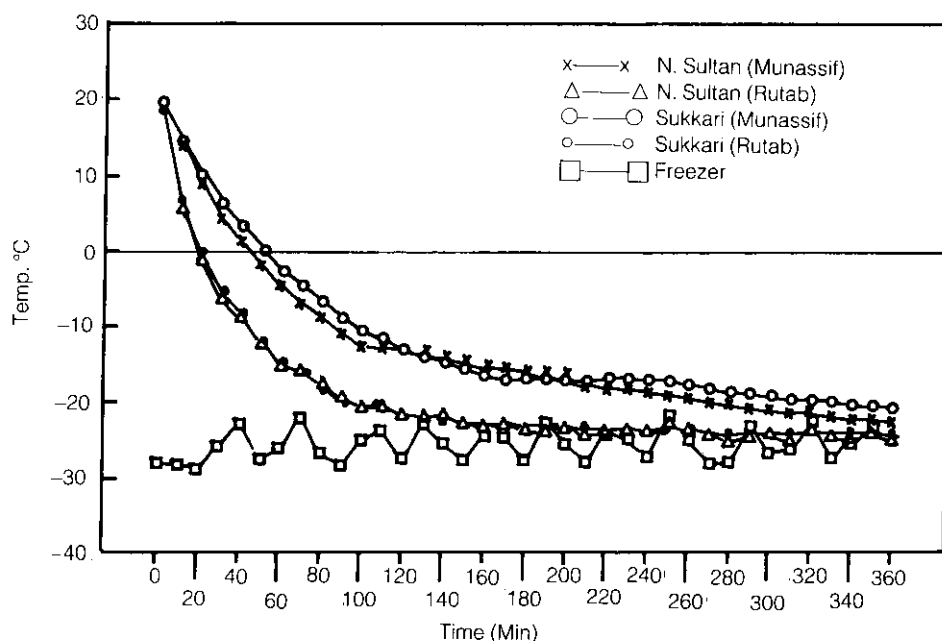


Fig. 6. Conventional freezing curves for the cultivars Nabt Sultan and Sukkari at their two stages of maturity.



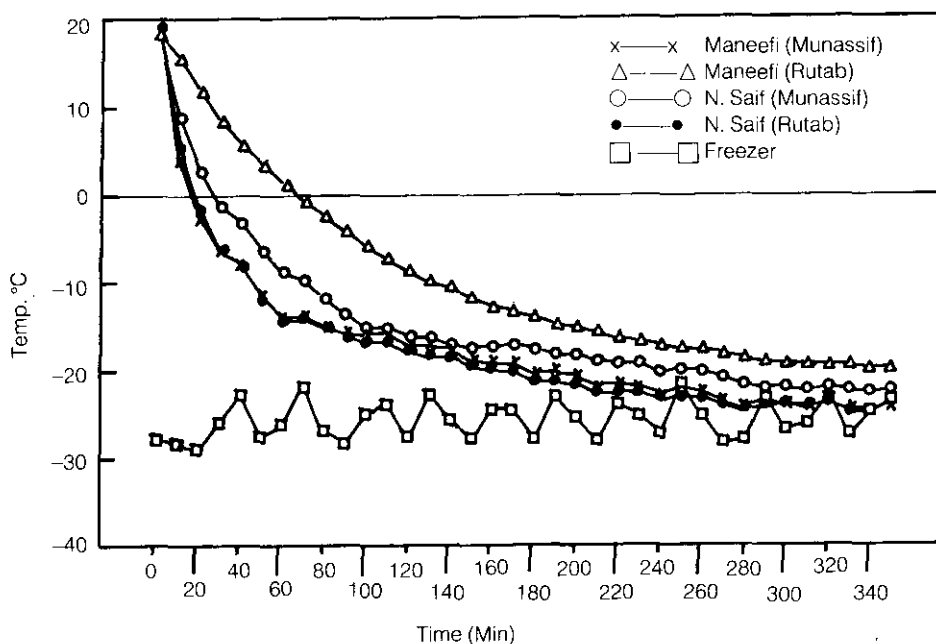


Fig. 7. Conventional freezing curves for the cultivars Maneefi and Nabt Saif at their two stages of maturity.

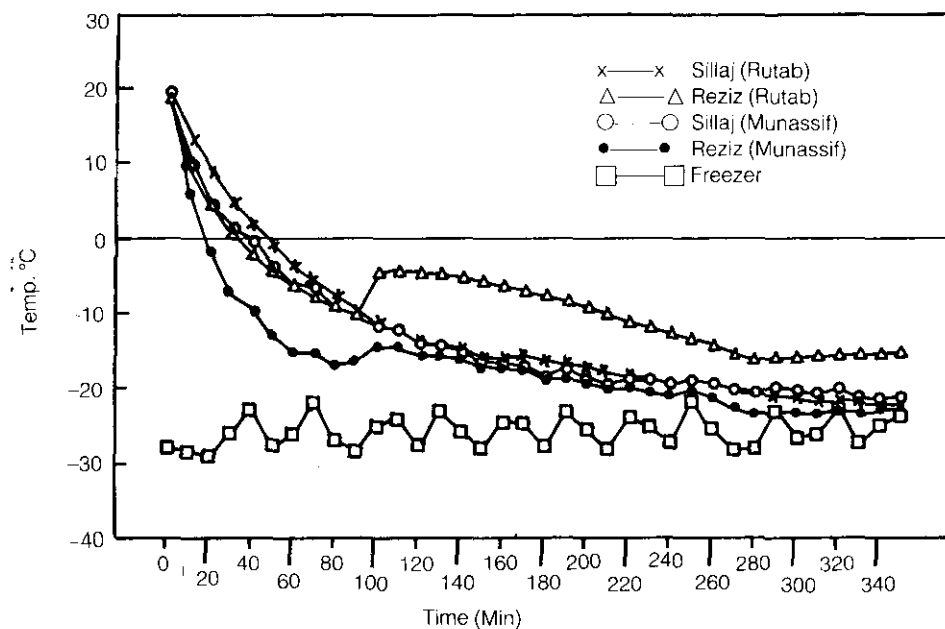


Fig. 8. Conventional freezing curves for the cultivars Sillaj and Reziz at their two stages of maturity.

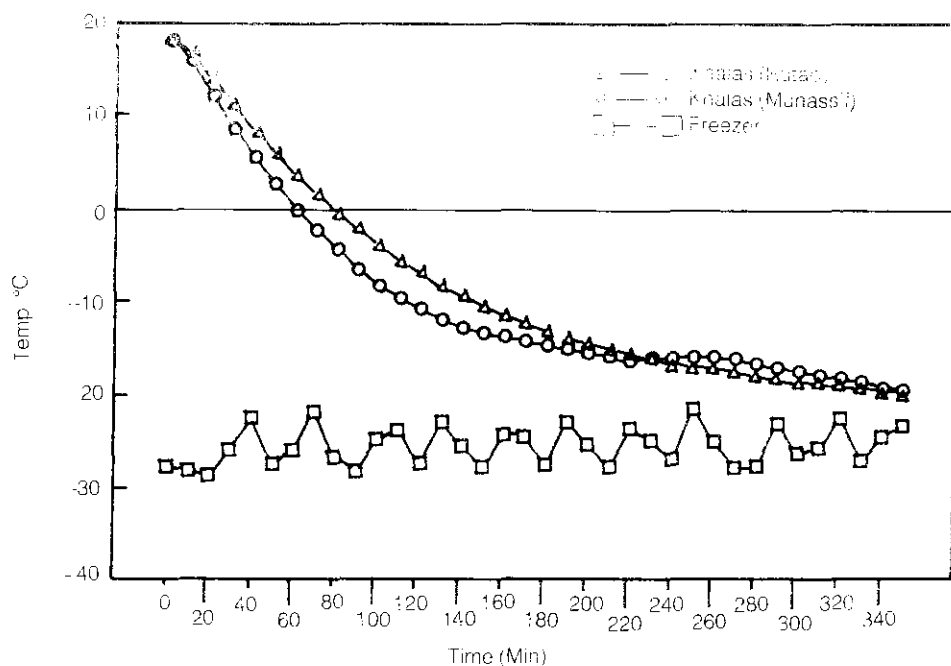


Fig. 9. Conventional freezing curves for the cultivar Khalas at its two stages of maturity.

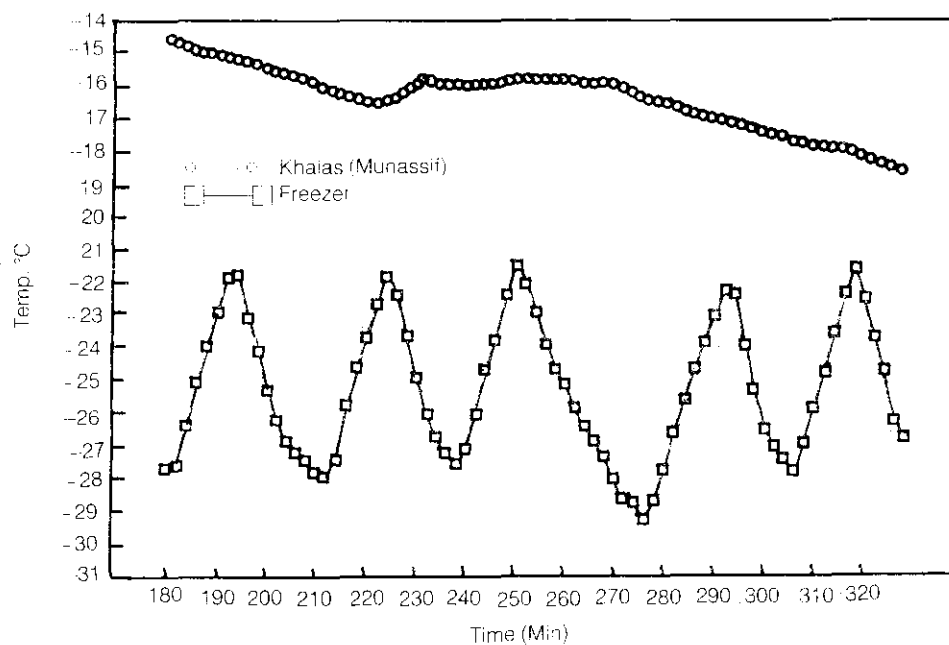


Fig. 10. The initial freezing point region for the cultivar Khalas at its munassif stage of maturity.

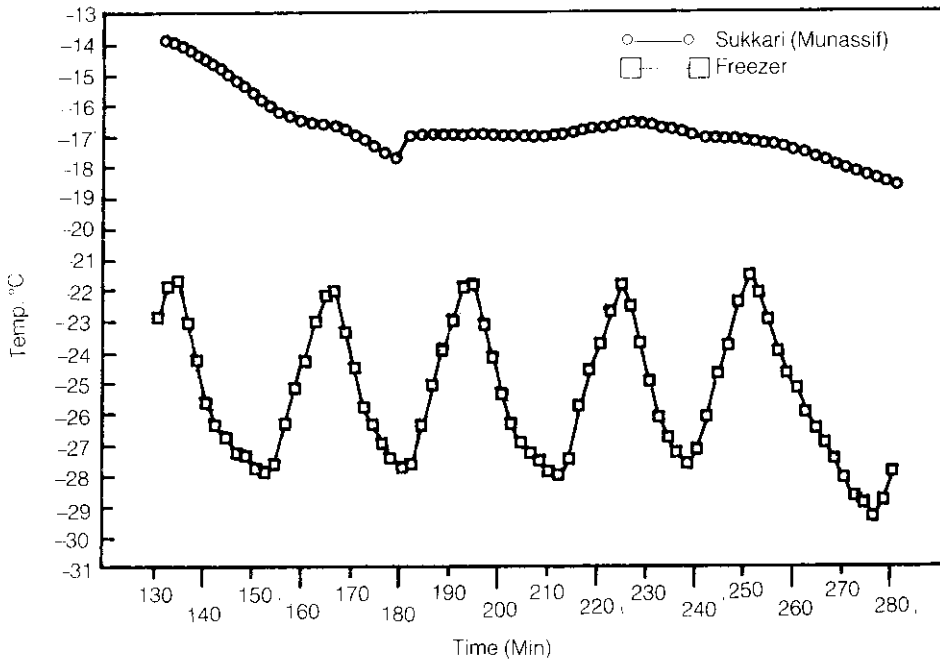


Fig. 11. The initial freezing point region for the cultivar Sukkari at its munassif stage of maturity.

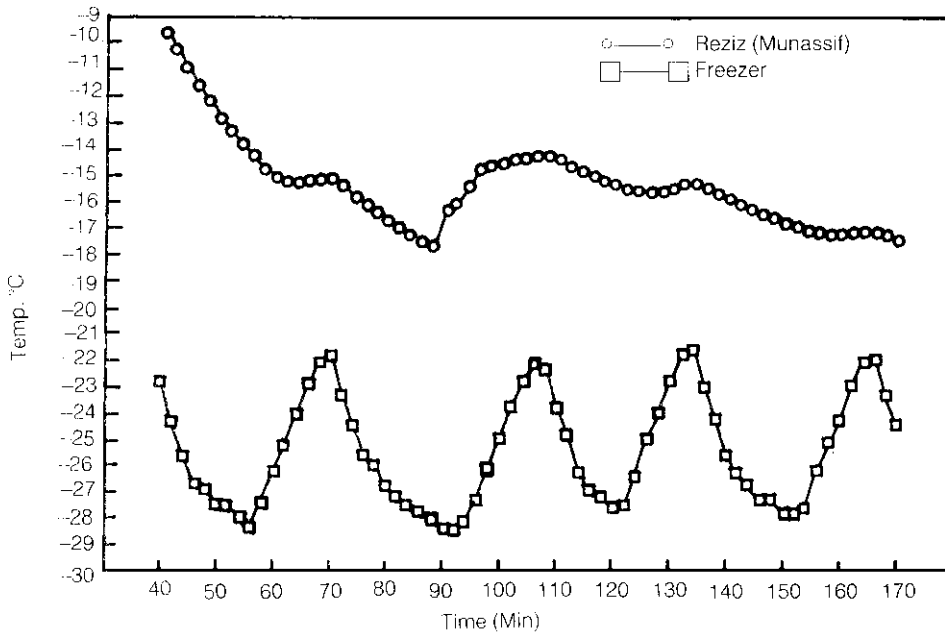


Fig. 12. The initial freezing point region for the cultivar Reziz at its munassif stage of maturity.

### Sensory evaluation

The major objective of the sensory evaluation analysis was to statistically determine whether there were significant differences ( $P < 0.05$ ) between fresh and cryogenically frozen soft dates stored at  $-50^{\circ}\text{C}$  for 12 months, of the seven studied cultivars at each of their two stages of maturity. Means of scores of quality attributes for the seven date cultivars at their two stages of maturity and their two levels of treatments are presented in Table 2. Fresh dates scored higher for all quality attributes.

**Table 2.** Means of sensory panel ratings for quality attributes of the seven cultivars of fresh soft dates (FRE) and thawed cryogenically frozen soft dates stored for 12 months at  $-50^{\circ}\text{C}$  (CRY), at their two stages of maturity, Munassif (MNF) and Rutab (RTB).

Cultivar	Stage of maturity	Treatment	Color	Texture	Flavor	Overall acceptability
Sukkari	MNF	FRE	7.95 a	8.05 a	7.75 a	7.80 a
		CRY	6.95 b	6.75 b	6.75 b	6.90 b
	RTB	FRE	7.85 c	7.60 c	7.70 c	7.60 c
		CRY	7.30 c	7.05 c	6.75 d	6.85 c
Nabt Sultan	MNF	FRE	8.25 a	8.05 a	8.10 a	8.20 a
		CRY	7.15 b	7.40 b	6.80 b	7.05 b
	RTB	FRE	7.50 c	7.20 c	7.70 c	7.55 c
		CRY	7.35 c	6.95 c	7.15 c	7.10 c
Nabt Saif	MNF	FRE	7.95 a	7.75 a	8.15 a	8.00 a
		CRY	6.85 a	6.70 a	6.95 a	7.05 a
	RTB	FRE	7.40 c	6.75 c	7.45 c	7.35 c
		CRY	6.00 a	5.90 c	6.20 a	6.10 a
Maneefi	MNF	FRE	8.40 a	8.05 a	8.30 a	8.25 a
		CRY	6.95 b	7.05 b	7.50 b	7.25 b
	RTB	FRE	7.50 c	7.55 c	7.70 c	7.70 c
		CRY	6.85 c	6.70 c	6.65 a	7.05 c
Sillaj	MNF	FRE	8.45 a	8.20 a	8.00 a	8.10 a
		CRY	6.85 b	7.15 b	6.95 b	6.95 b
	RTB	FRE	7.45 c	7.10 c	7.30 c	7.30 c
		CRY	7.10 c	7.00 c	7.20 c	7.05 c
Reziz	MNF	FRE	7.50 a	7.95 a	7.65 a	7.85 a
		CRY	6.20 b	6.75 b	6.50 b	6.55 b
	RTB	FRE	7.40 c	7.25 c	7.65 c	7.50 c
		CRY	7.00 c	6.35 c	6.95 c	6.80 c
Khalas	MNF	FRE	7.60 a	7.35 a	7.45 a	7.35 a
		CRY	6.90 a	6.40 b	6.90 a	6.70 b
	RTB	FRE	7.70 c	6.90 c	7.15 c	7.25 c
		CRY	6.45 a	6.25 c	6.45 c	6.60 c

– a, b, c, d Means bearing different letters within each column for each stage of maturity of each cultivar are significantly different at  $P < 0.05$ .

– Scoring scale; Like extremely = 9, Dislike extremely = 1.

butes of all date cultivars at their two stages of maturity. Duncan's Multiple Range Test (DMRT) was carried out to determine the statistical difference in quality attributes between the fresh and the cryogenically frozen.

*Color.* Cryogenic freezing and frozen storage had a significant effect on the color of the date cultivars Sukkari, Nabt Sultan, Sillaj, Maneefi, and Reziz at the Munassif stage of maturity. However, no significant difference was detected for Khalas and Nabt Saif. At the Rutab stage there was no significant difference in color at the two treatment levels for Sukkari, Nabt Sultan, Sillaj, Maneefi and Reziz. Nabt Saif and Khalas showed a significant difference in color due to cryogenic freezing and frozen storage. It could be concluded that color is more stable at the Rutab stage as compared to the Munassif stage of maturity with respect to cryogenic freezing followed by frozen storage at  $-50^{\circ}\text{C}$  for 12 months.

*Texture.* Means of scores for texture at the Munassif stage of maturity were significantly different at the two treatment levels for all studied date cultivars except Nabt Saif. No significant difference was detected for texture at the Rutab stage for all the seven cultivars.

*Flavor.* Significant differences in flavor were detected at the Munassif stage of maturity for all cultivars except Nabt Saif and Khalas. At the Rutab stage of maturity there was no significant difference in flavor between the fresh and the cryogenically frozen Nabt Sultan, Sillaj, Khalas and Reziz. However, there were significant differences in flavor for Sukkari, Nabt Saif, and Maneefi.

*Overall acceptability.* Cryogenic freezing and frozen storage significantly affected the overall acceptability at the Munassif stage of maturity of all studied cultivars except Nabt Saif. Panelists were not able to detect significant differences on scoring for the overall acceptability of all cultivars at their Rutab stage of maturity, except Nabt Saif. This clearly suggests a conclusion that cryogenic freezing followed by frozen storage, at Rutab stage for the fresh soft date cultivars Sukkari, nabt Sultan, Reziz, Maneefi, Sillaj and Khallas, does not affect their overall acceptability as compared to their fresh ones. The same conclusion applies to Nabt Saif at its Munassif stage of maturity.

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## التجميد الفائق للرطب الطازج

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ملخص البحث: تم تجميد سبعة أصناف من التمور شائعة الاستهلاك في طور الرطب كامل النضج والمنصف باستخدام النيتروجين السائل، والأصناف التي تم تجميدها هي السكري وبت سلطان وبت سيف والمنيفي والسلج والرزيز والخلاص، ولقد تم شراؤها طازجة خلال الموسم الإنتاجي ١٩٨٩م.

ولقد قورنت النتائج التجريبية لتغير درجة الحرارة مع الزمن أثناء عملية التجميد الفائق مع نظيرتها أثناء عملية التجميد التقليدي، ولقد أوضحت النتائج التجريبية أن نقطة التجمد الابتدائية لأصناف الرطب التي تم تجميدها تقع في الحدود -١٦ إلى -١٨°م.

وبمقارنة نتائج التقويم الحسي لأصناف الرطب التي تم تجميدها وتخزينها وتخزيناً مجمداً عند -٥٠°م لمدة ١٢ شهراً مع نظيرتها الطازجة، وجد أن هناك فروقاً مؤثرة إحصائياً بالنسبة للون والقوام والنكهة والقبول العام لمعظم أصناف الرطب المجمدة في طور المنصف، ولم يتمكن المقيمون من اكتشاف أي فروق مؤثرة إحصائياً بالنسبة للقبول العام لكل الأصناف في طور الرطب كامل النضج عدا بت سيف.

ولم تظهر في نتائج التقويم الحسي لنصف بت سيف المجمد في طور المنصف أي فروق إحصائية مؤثرة للونه أو قوامه أو نكهته أو قبوله العام مقارنة بنظيره الطازج.

