

# Physical properties of celery juice

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Lau, A.K., March, A.C., Lo, K.V. and Cumming, D.B. 1992. **Physical properties of celery juice.** *Can. Agric. Eng.* **34**:105-110. Before undertaking a project to design a pilot-scale freeze concentration system to dewater celery juice, laboratory studies were carried out to determine the physical, thermal and rheological properties of celery juice at various total solids concentration and various temperatures. The properties measured were solids content, specific gravity, electrical conductivity, freezing point and melting characteristics, specific heat, thermal conductivity and dynamic viscosity. Regression analyses were carried out for correlating specific gravity, electrical conductivity and specific heat with total solids content. Freezing point depressions of celery juice were compared to similar materials. Values of thermal conductivity were reported for a range of solids concentrations. Rheological properties of celery juice were represented by a family of dynamic viscosity curves.

Avant la conception d'un système pilote de concentration par congélation, destiné à déshydrater le jus de céleri, des études de laboratoire ont été effectuées pour déterminer les propriétés physiques, thermiques et rhéologiques du jus en fonction de diverses concentrations de matières solides et de températures. Les propriétés mesurées étaient les suivantes: teneur en matières solides, densité, conductivité électrique, point de congélation, caractéristiques de fusion, chaleur massique, conductivité thermique et viscosité dynamique. Des analyses de régression ont également été effectuées pour corrélérer la densité, la conductivité électrique et la chaleur massique avec la teneur en matières solides. Puis on a comparé les abaissements du point de congélation du jus de céleri à d'autres matières similaires. Des valeurs de conductivité thermique ont été déterminées pour toute une série de concentrations de matières solides, et les propriétés rhéologiques du jus ont été représentées par des courbes de viscosité dynamique.

## INTRODUCTION

Large quantities of celery are harvested from mid-July to mid-November in the Fraser Valley area of British Columbia, Canada. The celery harvested is used only to supply the fresh produce market, and up to 50 percent of the crop is either left in the field or designated for post-harvest processing rejection. Celery juice can be extracted from this otherwise wasted material to be used as a component in commercial vegetable juices and soups, thus providing a further source of revenue. A frozen celery juice concentrate is needed to reduce the bulk to be transported to distant markets, for example, Ontario where the commercial juices and soups are blended and manufactured.

Three main groups of technically feasible processes used for the concentration of liquid food are evaporation, reverse osmosis and freeze concentration. For juices containing aroma volatiles, the concentration process has to be highly selective. Where the high osmotic pressure of fruit juices precludes their concentration to the required level of total solids, Medina and Garcia (1988) recommended that reverse osmosis be em-

ployed as a first stage process with other technologies like freeze concentration completing the concentration system. As a result of significant advances made in improving the freeze concentration process (Deshpande et al. 1984; Smith and Schwartzberg 1985; van Pelt and Swinkels 1986), freeze concentration is now readily adopted in the food industry for manufacturing high quality products.

As part of a project funded under the Energy Research and Development in Agriculture and Food program, the feasibility of freeze concentrating celery juice was studied. To adapt and test at pilot scale a freeze concentration system for dewatering celery juice, laboratory studies were required to delineate the physical properties of celery juice. In particular, for predicting freezing rate, crystal growth rate and refrigeration capacity, data on thermal properties and rheological properties were needed.

The dewatering capacity of a freeze concentration system is highly dependent upon the initial and final dissolved solids content which affect the liquid viscosity at the freezing point temperature. Solute concentration is also a major factor that affects the nucleation rate and growth of ice crystals in a mixed suspension. The specific gravity and thus the bulk density of celery juice is needed for the calculation of the pumping power requirements, as well as for the design of the ice/concentrate separation device in a freeze concentration system. Celery juice is rich in salts (Buttkus 1978) and is a good conductor. The electrical conductivity of celery juice determined as a function of dissolved solids concentration can provide a simple and effective means for monitoring the product concentration continuously. An output signal from the meter could be used in a control circuit to maintain product uniformity by controlling internal flow rates and heat-removal rates. The freezing point (FP), specific heat and thermal conductivity of solutions are needed parameters for the design of freezing and thawing processes. The critical diameter of a crystal varies directly with the FP of the solution during crystallization, and the FP of the concentrate has a significant impact on the power consumption of the process. Viscosity values aid in the sizing of pumps and aid in determining the applicability of a flooded wash column for the separation of ice from the concentrate/ice slurry.

The purpose of this paper is to report the results of tests undertaken to determine the following properties for celery juice: solids content, specific gravity, electrical conductivity, freezing point and melting characteristics, specific heat, thermal conductivity and dynamic viscosity. As juice concentration increases progressively during the freeze concentration process, these physical properties were evaluated at various total solids concentrations.

## MATERIALS AND METHODS

Methods adopted in this study for measuring physical characteristics, thermal properties and rheological properties of liquid food have been thoroughly reviewed by Mohsenin (1980, 1986).

Initially, the juice was extracted by screw press from celery stalks which were rejected from the fresh market. Clarified juice was produced by filtration with a Millipore 0.22  $\mu\text{m}$  mean pore diameter filter. Juice was frozen and held at  $-20^{\circ}\text{C}$ . One barrel each of unclarified and clarified celery juice was allowed to thaw completely and then was thoroughly mixed prior to and during sampling. The samples were stored at  $3^{\circ}\text{C}$  before testing.

For the purpose of substrate characterization, it was assumed that freeze-drying would provide samples that were of acceptably close quality to freeze-concentrated juice. Ten litres of single-strength juice was freeze-dried to a light brown powder. The powder form of the juice was biologically stable and allowed for easy sample preparation for lab analysis. A range of concentrations was prepared by adding powder in measured quantities to 100 ml volumetric flasks and filling to the line with room temperature distilled water.

All tests for physical properties used rehydrated juice samples unless indicated otherwise, and duplicate or triplicate samples were used in each test run for each physical property.

### Solids content

The determination of total solids (TS) concentration in mass percentage was carried out by drying a known mass of sample in an oven at  $105^{\circ}\text{C}$  for 24 hours. These dry samples were then placed in a muffle furnace at  $550^{\circ}\text{C}$  for 20 minutes to determine the percentage of volatile solids (VS). Suspended solids (SS) and volatile suspended solids (VSS) were determined using the same drying procedure outlined above from the suspended material filtered out of the samples with Whatman glass microfibre 934-AH filters in a Buchner funnel vacuum filtration assembly. The filters were conditioned in the furnace at  $600^{\circ}\text{C}$  for 15 minutes to eliminate any trace moisture or volatiles.

Initially, both clarified and unclarified juice samples from the original celery juice that had been frozen were used in the above-mentioned test procedure. Subsequent measurements of this parameter, during the evaluation of physical properties as a function of total solids content only, made use of rehydrated celery juice.

### Specific gravity

A 25 ml flask was weighed before and after filling to the line, in turn, with room temperature ( $22^{\circ}\text{C}$ ) distilled water and celery juice. A Mettler H6 digital analytical balance was used to determine all masses to the nearest 0.1 mg. Tests were performed in ascending order of concentration.

### Electrical conductivity

Electrical conductivity (EC) was determined by use of an EC meter (YSI Model 31) and probe. Unfiltered juice samples prepared from freeze-dried celery juice powder were tested at  $26^{\circ}\text{C}$ .

### Freezing point and melting characteristics

A refrigerated circulating bath was used to determine the

freezing point for each level of concentration. The apparatus was precooled to one of five temperatures between  $-10^{\circ}\text{C}$  and  $-18^{\circ}\text{C}$ . Samples were tested according to the following procedure: Carbon dioxide cartridges were rinsed with distilled water. Each cartridge, with an interior volume of about 10 ml, was filled with 7.00 ml of a given juice sample having a dissolved solids concentration between 5% and 30%. Since the suspended solids concentration was low, the dissolved solids (DS) and TS concentrations were essentially the same. The cartridges were then placed upright in a circular plastic holder and the thermocouple leads from all samples were attached to a datalogger (Digitek Model 1268). A Kaye model K140-4 Ice Point Reference was used as a temperature reference. The datalogger was activated as the samples were immersed in the bath. Readings were recorded at 15 s intervals until the temperature of the samples approached that of the bath, terminating the run. Runs were made both with and without agitation of the samples.

A differential scanning calorimeter DSC (Perkin Elmer Model DSC-2) along with a thermal analysis data station were used to further examine the melting characteristics of the prepared juice samples.

Melting curves were obtained to gain some insight into the phase change characteristics of the juice at various levels of concentration. After each test, the graphical results were analyzed to determine the melting point onset temperature, the specific energy gain and the temperature corresponding to the maximum melting rate for the sample.

### Specific heat

The DSC also provided a rapid and dynamic method to determine the specific heat of celery juice, as described by Otten et al. (1980). The sample and the sapphire standard were forced to simultaneously scan through a temperature range of  $-40$  to  $20^{\circ}\text{C}$  at a constant rate of  $10^{\circ}\text{C}/\text{min}$ . The difference in the power input between the two cells was a direct function of the mass and specific heat of the sample. A specific heat value was obtained from the DSC thermograms by dividing the heat input rate in MJ/s by the temperature scanning rate in  $^{\circ}\text{C}/\text{s}$  and the sample mass in mg to yield the appropriate units of  $\text{kJ}\cdot\text{kg}^{-1}\cdot^{\circ}\text{C}^{-1}$ . A comparison of the sample's thermogram (rate of energy input versus temperature) with that of the sapphire standard of known specific heat allowed calculation of the sample's specific heat.

### Thermal conductivity

The thermal conductivity at the freezing point for various concentrations of celery juice was calculated from the following empirical equation for fruit juices and sugar solutions (Heldman and Singh 1981). This equation has incorporated the effect of the thermal conductivity of water and of the solids component in the product.

$$k = [326.575 + 1.0412 \cdot FP - 0.00337 \cdot (FP)^2][0.796 + 0.009346 \cdot (100 - TS)] \cdot 10^{-3} \quad (1)$$

where:

- $k$  = thermal conductivity ( $\text{W}\cdot\text{m}^{-1}\cdot^{\circ}\text{C}^{-1}$ ),
- $FP$  = freezing point ( $^{\circ}\text{C}$ ), and
- $TS$  = total solids (%).

Once the specific heat and thermal conductivity are known, thermal diffusivity can be readily calculated.

### Viscosity

A Cannon-Fenske Routine type viscometer was used to estimate the viscosity of celery juice over a range of temperatures and dissolved solids concentrations. Experiments were carried out at temperatures down to the freezing point or its vicinity. The temperature control jacket was connected to a Lauda RCS-6D refrigerated circulating bath for very stable temperature control ( $\pm 0.02^\circ\text{C}$ ). A #100 Z13 tube with a recommended range of viscosities from 3 to  $15 \cdot 10^{-3}$  Pa·s was used for all runs. The efflux time (s) was measured to the nearest 0.1 s and the kinematic viscosity value ( $\text{m}^2/\text{s}$ ) was calculated by multiplying the efflux time by a temperature-compensated tube constant. The kinematic viscosity can easily be converted to dynamic viscosity (Pa·s) via the specific gravity.

## RESULTS AND DISCUSSION

### Solids content

The solids analysis for the unconcentrated juices is presented in Table I. The unclarified juice had a TS concentration of 3.5% compared with 2.6% for the clarified juice; the filtration of the whole, unconcentrated juice reduced the TS by 24% and the VS by 29% by removing 98% of the SS. The mass percentage solids in the clarified and unclarified juices were related according to the regression equation:

$$TS_c = 0.409 + 0.806 TS_u \quad (R^2 = 0.966) \quad (2)$$

where:

$TS_c$  = total solids in clarified juice (%), and

$TS_u$  = total solids in unclarified juice (%).

**Table I. Results of solids analysis for unconcentrated celery juice**

	Total solids (%)	Volatile solids (%)	Suspended solids (%)	Volatile suspended solids (%)
Clarified	2.62 $\pm$ 0.07	1.70 $\pm$ 0.04	0.0098 $\pm$ 0.0005	trace amt
Unclarified	3.47 $\pm$ 0.03	2.39 $\pm$ 0.05	0.44 $\pm$ 0.01	0.42 $\pm$ 0.01

### Specific gravity

Experimental results of specific gravity were analyzed by means of a linear regression of specific gravity on total solids content, which returned the equations:

$$SG_c = 1.000 + 0.006 TS_c \quad (R^2 = 0.984) \quad (3)$$

$$SG_u = 0.997 + 0.005 TS_u \quad (R^2 = 0.971) \quad (4)$$

where:

$SG_c$  = specific gravity of clarified juice, and

$SG_u$  = specific gravity of unclarified juice.

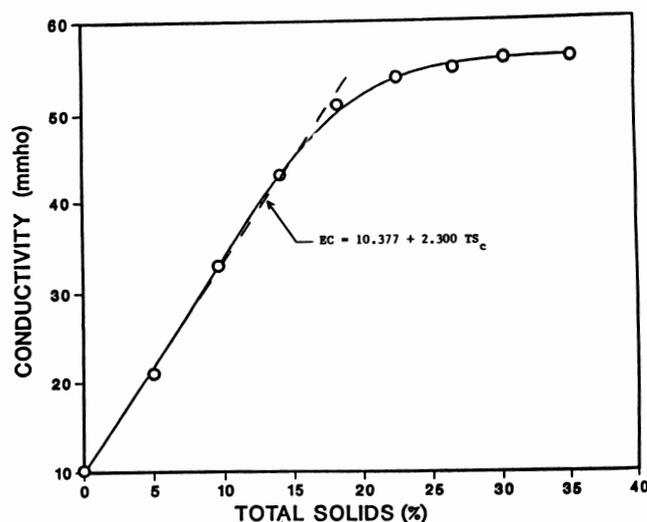
### Electrical conductivity

The electrical conductivity readings, as demonstrated in Fig. 1, are quite sensitive to changes in the level of total solids content

below 18%, and show a nearly linear response for EC vs TS. The straight line portion may be mathematically described by

$$EC = 10.377 + 2.300 TS_c \quad (R^2 = 0.982) \quad (5)$$

where  $EC$  = electrical conductivity (mmho).



**Fig. 1. Electrical conductivity at a temperature of 26°C.**

Since 18% TS represents a concentration factor of seven, while the most likely final concentration for the juice in a freeze concentration process is four (10.5% TS), this appears to be a very useful method for determining solids concentration and providing control loop feedback signals during operation. Temperature compensated probes allow readings taken at temperature from as low as  $-5^\circ\text{C}$  to as high as  $100^\circ\text{C}$  to be converted to standard readings at  $26^\circ\text{C}$ .

### Freezing point and melting characteristics

Based on the transient temperature measurements, the freezing point (FP) temperatures were taken to be the highest ones achieved, as obtained from the crystallization exotherms following supercooling. The degree of supercooling generally decreased as the TS concentration increased and as the FP of the solutions approached the bath temperature. Crystallization exotherms which were rounded in shape with no isothermal portion, may have been peaking at some temperature below the actual FP of the solution. Therefore, the refrigerated circulating bath method yielded at best a close approximation of the actual FP, which could only be determined precisely by allowing steady-state conditions to prevail after each temperature increment or decrement. The latter procedure is exceedingly time-consuming, since at each operation the proportions of ice and solution must approach equilibrium. However, the FP temperatures obtained from these tests are adequate for the design and start-up of a freeze concentration system having a TS monitor and control loop with which to fine-tune the bulk temperature to produce the desired concentration.

A typical temperature-time curve for 25% TS celery juice is shown in Fig. 2. Following the onset of crystallization at  $-14^\circ\text{C}$ , the released heat of crystallization caused the temperature to rise to the FP of  $-8^\circ\text{C}$ . Figure 3 depicts the FP temperatures at various TS levels. For comparison purpose, the FP curves for coffee extract, wine and blackcurrant juice (Thijssen 1974) are shown in the same graph. The rapid low-

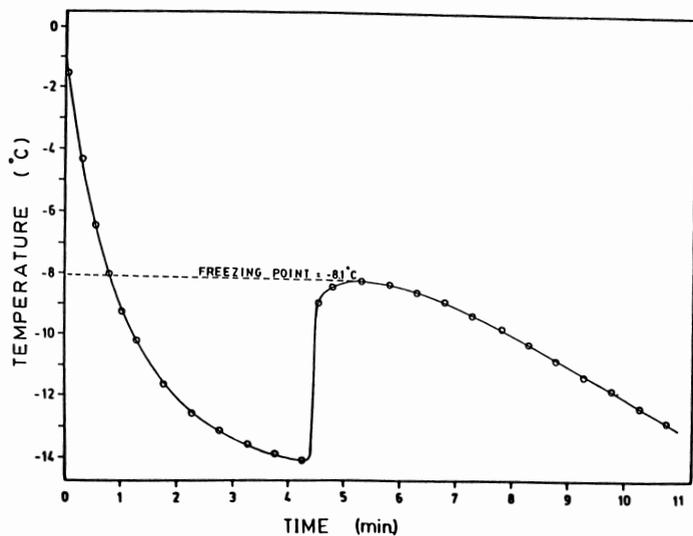


Fig. 2. Typical freezing curve of celery juice at 25% total solids.

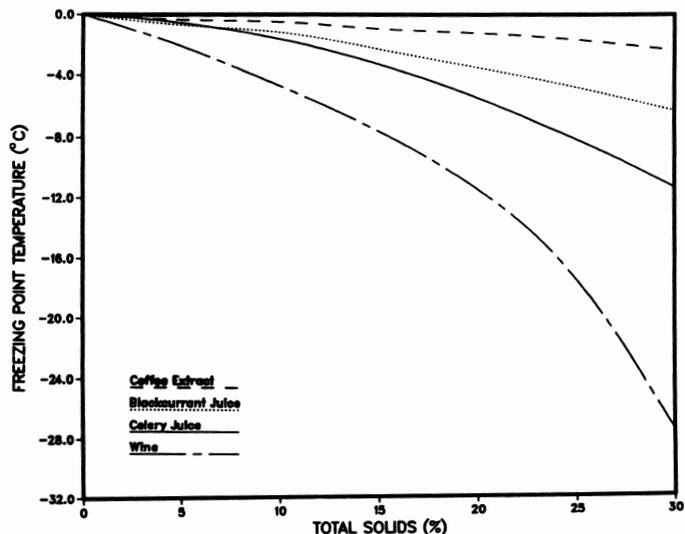


Fig. 3. Freezing point depression as a function of total solids content (celery juice: present study; other materials: Thijssen 1974).

ering of the FP for wine is due mainly to the increasing concentration of alcohol.

Figures 4(a), (b) and (c) illustrate the melting curves and contrast the maximum melting points, peak shapes and areas for 10%, 25% and 40% TS, respectively. The shape of the peaks shown are very similar, while the peaks are shifted to the left for increasing solids content, demonstrating the FP depression due to the higher TS concentration. The melting peak is seen to cover a wide temperature range rather than occurring at a fixed temperature as is the case for an operating freeze concentration system. The differential scanning calorimeter results are based, however, not on the melting of pure ice crystals in various TS concentrations, but on the melting of a mixture of ice crystals and solution.

Some irregular patterns are observed in Figs. 4(a) and (b) when temperature is at approximately  $-12^{\circ}\text{C}$ . The slight flattening of the slope indicates that a smaller amount of heat was required to be added for a constant rate of temperature in-

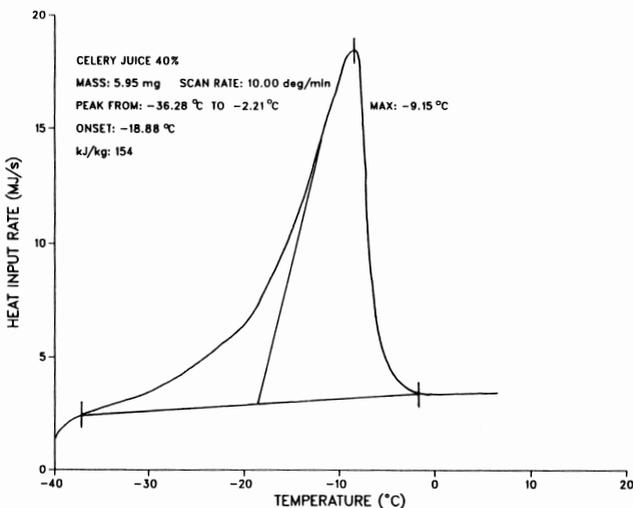
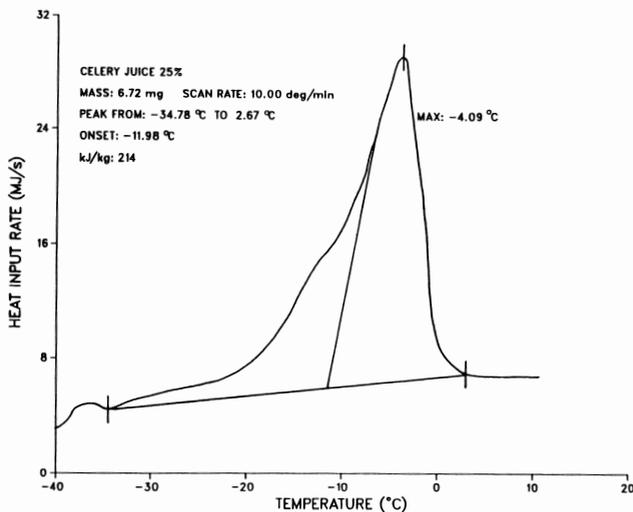
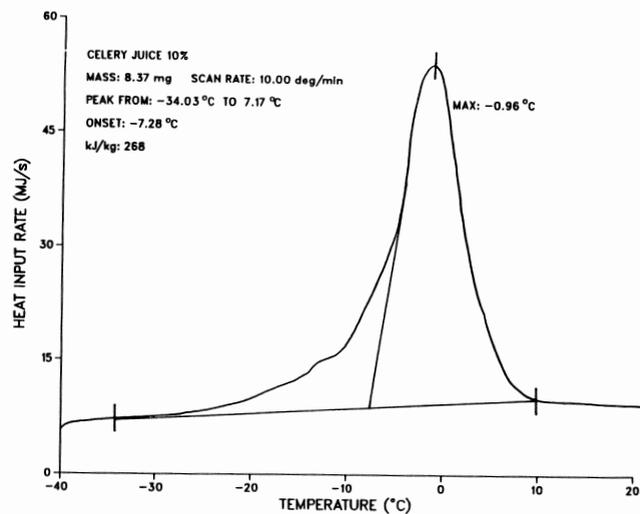


Fig. 4. Differential scanning calorimeter (DSC-2) melting curves (a) solids content = 10% (b) solids content = 25% (c) solids content = 40%.

crease, which can be explained by assuming the occurrence of a 'glass transition phenomenon' at this temperature. The samples were quickly cooled to  $-40^{\circ}\text{C}$  at  $360^{\circ}\text{C}/\text{min}$  prior to each calorimetry run, thus the solution would freeze before any

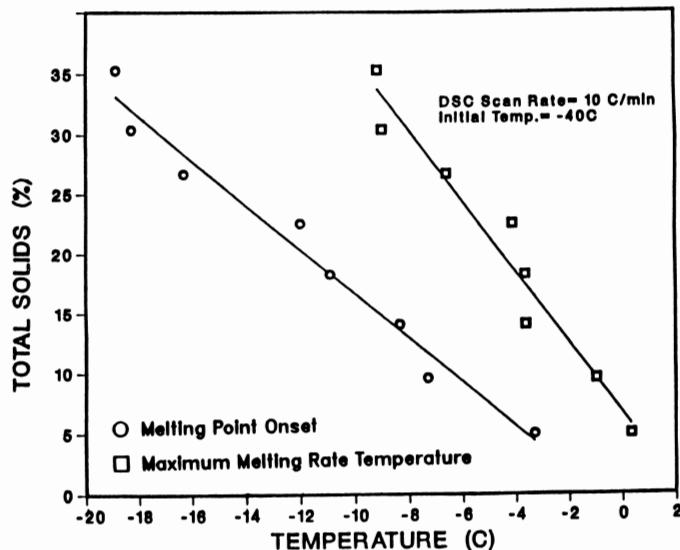


Fig. 5. Melting characteristics as derived from the melting curves.

significant crystal growth could take place. Upon warming, as the temperature reaches  $-12^{\circ}\text{C}$ , the solution has melted to the point where free water molecules have enough mobility to change from a random, 'glassy' state to a lower energy crystalline state. The heat of crystallization thus released momentarily reduces the rate of heat input. It shall be noted that this postulated glass transition is not likely a factor of concern in a freeze concentration process, since the FP is always approached from the high side.

The melting point onsets and the temperature corresponding to the maximum melting rate of the sample as derived from the melting curves are illustrated in Fig. 5. That the best-fit lines for the two variables diverge as percent TS increases, indicates the lowering of the FP and the simultaneous broadening of the melting peak with increasing percent TS. This phenomenon explains the difficulty encountered in establishing the FP of an increasingly impure solvent by the exotherm-plateau method as previously discussed.

### Specific heat

Specific heat of celery juice is plotted in Fig. 6 as a function of percent TS. Mean specific heat values for each TS content were calculated from test results with temperature ranging from  $-40^{\circ}\text{C}$  to  $20^{\circ}\text{C}$ . The experimental runs included measurement taken at 0% TS and an average value of  $4.21 \pm 0.17 \text{ kJ}\cdot\text{kg}^{-1}\cdot^{\circ}\text{C}^{-1}$  was obtained for water, which compares very well with the reported value of  $4.18 \text{ kJ}\cdot\text{kg}^{-1}\cdot^{\circ}\text{C}^{-1}$  (Kreith and Black 1980). The following linear equation was fitted to the experimental data:

$$c_p = 4.22 - 0.03 TS_c \quad (R^2 = 0.958) \quad (6)$$

where  $c_p$  = specific heat ( $\text{kJ}\cdot\text{kg}^{-1}\cdot^{\circ}\text{C}^{-1}$ ).

### Thermal conductivity

Table II shows the thermal conductivity for a range of TS content of celery juice at the corresponding temperature of its freezing point as interpolated from Fig. 3.

As TS changes from 0% to 18%, thermal conductivity only decreases by 11% from  $0.565$  to  $0.502 \text{ W}\cdot\text{m}^{-1}\cdot^{\circ}\text{C}^{-1}$ . The thermal conductivity was not sensitive to the standard deviation of

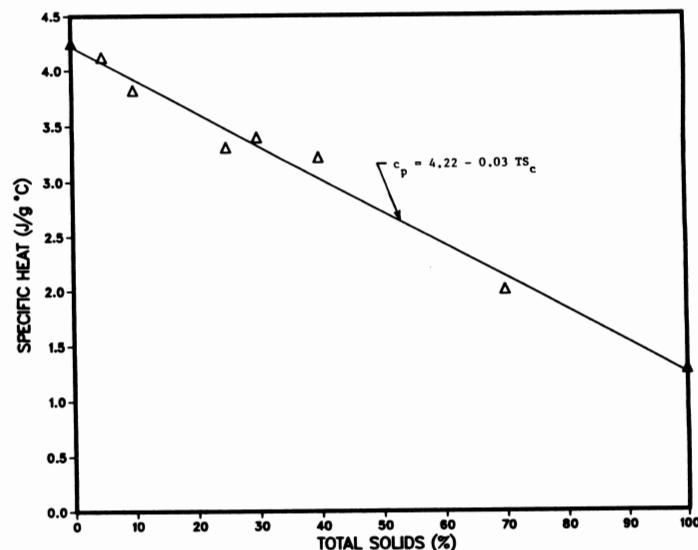


Fig. 6. Mean specific heat of celery juice as a function of total solids.

the measured freezing point at each solids content.

The results of specific heat and thermal conductivity demonstrated that their values were not significantly different from those of water, for total solids content of celery juice up to 18% (7-fold concentration factor).

### Viscosity

Results from the Cannon-Fenske Routine Viscometer are presented in Fig. 7 in the form of a family of dynamic (absolute) viscosity curves for celery juice. These results augmented Kuprianoff's (1966) results for fruit juices. For the range of temperatures investigated, at 10.5% and 18% TS levels (that is, 4-fold and 7-fold concentration factors), the viscosity of celery juice is about 40% and 85%, respectively, greater than that of water ( $1.8 \cdot 10^{-3} \text{ Pa}\cdot\text{s}$  at  $0^{\circ}\text{C}$ ).

Table II. Thermal conductivity of celery juice at its freezing point

Solids content (%)	Freezing point ( $^{\circ}\text{C}$ )	Thermal conductivity ( $\text{W}\cdot\text{m}^{-1}\cdot^{\circ}\text{C}^{-1}$ )
0	$0.0 \pm 0.05$	0.565
5	$-1.0 \pm 0.24$	0.549
10	$-2.2 \pm 0.26$	0.531
15	$-3.6 \pm 0.33$	0.513
20	$-5.7 \pm 0.68$	0.495
25	$-8.2 \pm 0.97$	0.476
30	$-9.1 \pm 1.80$	0.459

## CONCLUSIONS

Tests on physical characteristics, thermal properties and rheological properties were carried out for a range of total solids concentration and at various temperatures. Specific gravity, electrical conductivity and viscosity were found to increase with an increase in solids content of the juice samples, while freezing point temperature and specific heat vary adversely with solids content. Linear equations were fitted to experimen-

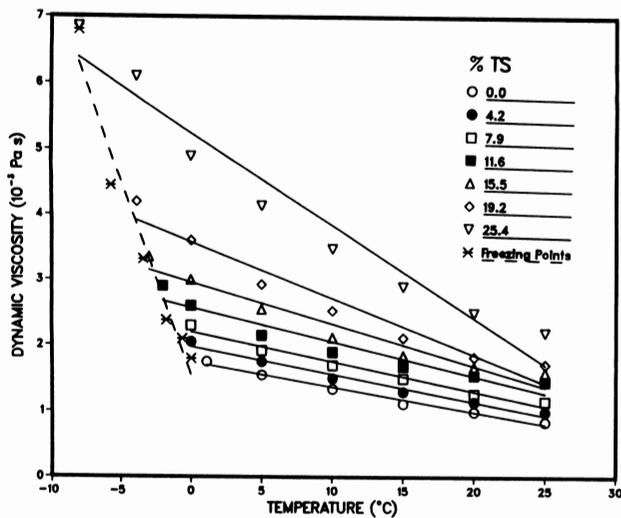


Fig. 7. Dynamic viscosity vs. temperature.

tal data, for correlating specific gravity, electrical conductivity and specific heat with total solids content.

Although celery juice had a larger freezing point depression compared to coffee extract and blackcurrant juice, its low solids concentration and viscosity still renders it suitable for the freeze concentration process. A maximum viscosity of  $200 \cdot 10^{-3}$  Pa·s and total solids concentration of 45-55% for the concentrated product had been recommended by Thijssen and van Oyen (1977). Increased viscosity can make pumping the concentrate and washing the ice very difficult. The low total solids content of the juice also implied that the thermal properties can be assumed to be the same as for water for design purposes.

#### ACKNOWLEDGEMENTS

The authors thank Neil Jackson for his technical assistance, and East Chilliwack Agricultural Co-op for their cooperation in supplying the juice samples. Funding provided by Agriculture Canada through ERDAF contract no. 04SB.01916-3-EP19 is also gratefully acknowledged.

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