

Some thermal properties of fresh and osmotically dehydrated Kiwifruit above and below the initial freezing temperature

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Abstract

Thermal properties of fresh and osmotically dehydrated Kiwifruit were experimentally measured. Enthalpy and heat capacity, in the range from $-40\text{ }^{\circ}\text{C}$ to $40\text{ }^{\circ}\text{C}$, and initial freezing temperature were determined by DSC. Density was measured by picnometry in the range between $-70\text{ }^{\circ}\text{C}$ and $30\text{ }^{\circ}\text{C}$. Prediction equations from the literature for enthalpy, heat capacity and density as a function of temperature, for different water contents, were fitted to experimental data. Regression equations relating soluble solids content and initial freezing temperature with water content were also obtained.

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1. Introduction

Osmotic dehydration (OD) of food materials, especially of fruits and vegetables, used prior to freezing leads to substantial energy savings. The osmotic dehydration step consists of a simple immersion of the foodstuff in a highly concentrated solution of sugar and/or salt close to room temperature. In most cases sugars, generally sucrose, glucose and different corn syrups are used as dehydrating agents for fruits (Agnelli et al., 2005). As a consequence of the concentration gradient, the water from the food moves into the solution and solutes from the solution move into the product. The water removal is carried out without phase change in the liquid phase, which enhances heat and mass transfer coefficients (Lenart and Lewicki, 1988). During subsequent freezing, the energy requirements decrease due to the lower water content of the product.

The interest in dehydration prior to freezing is also due to the nutritional and organoleptic properties of the final

products (Ponting et al., 1966). Osmotic dehydration, carried out at moderate temperatures, protects thermosensitive compounds such as flavors, pigments and vitamins (Ponting et al., 1966; Vial et al., 1991). Also, as it prevents food from getting in contact with air, oxidation reactions (Raoult-Wack, 1994) and loss of volatile compounds (Ponting, 1973) are limited.

Kiwifruit (*Actinidia deliciosa*) is a valuable fruit with characteristic taste and flavor and high vitamin contents; so numerous methods to extend its storage life through processing have been devised. In particular freezing of Kiwifruit, partially dehydrated in air or in sugar solutions (dehydrofreezing), has been studied in several published research papers and found to be a valuable processing alternative (Dalla Rosa et al., 1980; Forni et al., 1990; Torreggiani et al., 1987; Vial et al., 1991; Torreggiani et al., 1994; Bressa et al., 1997; Robbers et al., 1997; Spiazzi et al., 2000; Talens et al., 2001; Talens et al., 2003; Marani et al., 2007).

To define operating conditions, to calculate freezing times and to design the adequate process equipment for dehydrofreezing, a number of thermophysical properties

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Nomenclature

| | | | |
|----------|------------------------------|----------|---|
| A | constant in Eq. (5) | m_0 | mass of fresh sample (kg) |
| B | constant in Eq. (5) | m_s | mass of dried sample (kg) |
| C | constant in Eq. (5) | n | constant in Eq. (5) |
| C_p | heat capacity (kJ/(kg °C)) | SG | solid gain (%) |
| D | constant in Eq. (7) | SS | soluble solids content (%) |
| E | constant in Eq. (7) | T | temperature (°C) |
| F | constant in Eq. (9) | T_{cr} | initial freezing temperature (°C) |
| G | constant in Eq. (9) | WC | water content (dimensionless) |
| H | enthalpy (kJ/kg) | WL | water loss (%) |
| H_{cr} | enthalpy at T_{cr} (kJ/kg) | WR | weight reduction (%) |
| m_f | final mass of sample (kg) | ρ | density (kg/m ³) |
| m_i | initial mass of sample (kg) | ρ_u | density of unfrozen sample (kg/m ³) |

of the food to be treated are needed including initial freezing point, specific heat capacity and enthalpy, density and thermal conductivity (Agnelli et al., 2005). All of these properties depend on composition and temperature (Tocci et al., 1998).

Experimental values and prediction equations for these properties can be found in literature for many fresh foods, covering the ranges of cooling and freezing temperatures (Rha, 1975; Mohsenin, 1980; Jowitt et al., 1983a,b; Sweat, 1985). Also some information is available for hot-air dried or freeze-dried and rehydrated fruits and vegetables (Sá and Sereno, 1994; Sá et al., 1998, 1999; Sereno et al., 1998; Kasapis et al., 2000; Telis and Sobral, 2001; Bai et al., 2001; Baroni et al., 2003). However, this information is not valid for osmotically dehydrated foods whose composition and structure is different from that of those hot-air dried, due to soluble solids uptake and less structural damage during the OD process. There is little specific information for OD fruits and vegetables. Only a few sets of data from the literature (Sá and Sereno, 1994; Sá et al., 1998, 1999; Silva et al., 1996; Sereno, 2000) or from previous works by this research group (Tocci et al., 1998; Tocci and Mascheroni, 1996, 1998, 2001) are available, and only for a reduced set of fruits and dehydrating solutions. In the specific case of Kiwifruit, only the two latter papers provide experimental data on some thermal properties of OD Kiwifruit.

Therefore, the aim of the objectives of this work were to:

- Measure the dehydration rate of Kiwifruit in sucrose solution and the relationship between water content and soluble solids in the partially dehydrated food.
- Measure specific heat capacity, enthalpy and density as a function of temperature for fresh and partially dehydrated Kiwifruit.
- Determine initial freezing temperature as a function of water content.
- Compare measured values with literature data and, if possible, develop general relationships.

2. Materials and methods

Kiwifruits (*A. deliciosa*) of the cultivar Hayward were purchased at a local market, choosing fruits with similar degree of ripeness. Fruits were washed, hand-peeled and superficially dried. Samples were prepared in the shape of cubes with 1 cm side. Ten 50 g cubic samples were prepared. Two of the samples were kept as fresh product and the remaining eight samples were dehydrated.

2.1. Osmotic dehydration

Dehydrations were performed by dipping each of the samples in an open mesh plastic net bag, into a 20 L stainless steel vessel filled with the OD solution. The vessel was provided with a coil through which circulates an ethylene glycol–water mixture to regulate the temperature of the osmotic solution. The equipment was fitted with a stirrer to ensure good mixing of the OD solution over the whole system (Agnelli et al., 2005).

The samples were submerged in the OD solution at 20 °C for different immersion times (0, 2, 6, 12 or 24 h). Duplicate tests were run in all cases. The osmotic concentration agent was a saturated aqueous solution of commercial grade sucrose (60.0% w/w).

Once the desired immersion time was reached, two samples were taken from the vessel, intermittently washed for 30 s with distilled water, blotted in absorbent paper and weighed.

The change of mass was measured and the weight reduction (WR), water content (WC) and soluble solids content (SS) were estimated as follows:

Weight reduction (WR) was calculated from the weight difference of the sample before and after the treatment:

$$WR = \frac{100(m_i - m_f)}{m_i} \quad (1)$$

Water content (WC) was determined by drying in a vacuum stove at 70 °C until a constant weight was reached:

$$WC = \left(\frac{m_0 - m_s}{m_0} \right) \quad (2)$$

Soluble solids content (SS) was measured in an Abbe refractometer (Bellinham + measured in °Brix).

In addition, water loss (WL) and solid gain (SG) were calculated according to a mass balance:

$$WL = \left[\left(1 - \frac{TS^0}{100} \right) - \left(1 - \frac{TS}{100} \right) \left(1 - \frac{WR}{100} \right) \right] \times 100 \quad (3)$$

$$SG = \left[\left(1 - \frac{WR}{100} \right) \frac{TS}{100} - \frac{TS^0}{100} \right] \times 100 \quad (4)$$

where TS^0 and TS are the initial and current values of the solid content (%).

2.2. DSC determinations

Samples (fresh or dehydrated) were mashed and homogenized and small samples (10–25 mg each) were taken and sealed in the specific sample holder (capsules) for DSC evaluation. Four samples were prepared for each dehydration time (two from each duplicate sample) and the results of these runs were averaged.

Tests were conducted on a Differential Scanning Calorimeter with automatic data recording (Tocci and Mascheroni, 1997). Samples were stabilized at –40 °C and heated from –40 °C to 40 °C at a rate of 2 °C min⁻¹ a low value that minimizes the temperature lags likely to occur in the event of a poor thermal contact of the sample-capsule-base system (Callanan and Sullivan, 1986). The lower bound of –40 °C was considered sufficient so as to cover the typical temperature range of industrial processes. Besides, below that temperature, the additional amount of water frozen is negligible. For frozen foods, measurements are always performed heating (thawing) the samples, because during freezing it is common to have subfreezing, which leads to erroneous values in measured properties.

Enthalpy was calculated by integrating the experimental data of heat capacity (C_p) vs temperature, taking –40 °C as the datum ($H_{-40} = 0$).

Initial freezing temperature T_{cr} was determined as the position of the thawing peak in the experimental heat capacity vs temperature plot (see Fig. 5), as described in Tocci et al. (1998) and Sereno (2000), among other references. This is probably the less accurate of the measured properties, because this point cannot be determined with good precision even when the DSC was calibrated against pure materials of known melting points (Tocci et al., 1998).

Enthalpy variation between –40 °C and T_{cr} (H_{cr}) was calculated for each sample. It can be considered as a characteristic value for the total enthalpy change during freezing or thawing and also appears as a parameter in some prediction equations, such as those used in this work.

2.3. Determination of densities

Densities were determined for three of the samples using 50 mL picnometers with toluene as the picnometric liquid. Cubes of fresh or partially dehydrated Kiwifruit were weighed and filled into the picnometers whose volumes were completed with toluene. Then, samples were stabilized to the desired temperature in laboratory freezers. The liquid level was topped up with thermo-equilibrated toluene and the picnometers were weighed in an electronic balance. Sample temperature was determined using a copper-constantan thermocouple and a data-acquisition system. Tests were performed in duplicate for each dehydration time.

2.4. Prediction equations

2.4.1. Enthalpy and specific heat

Most equations from the literature for the prediction of enthalpy and heat capacity in the temperature range below T_{cr} are based in the theoretical Clausius–Clapeyron relationship (Rha, 1975; Mohsenin, 1980; Jowitt et al., 1983a). To be able to deal with experimental data, some empirical versions are used because neither the average molecular weight nor the exact composition of solutes are known, and this relationship implies ideal behavior of solutes, which is not true for dehydrated foods. Therefore, the prediction equations used were similar to those proposed by Schwartzberg (1976) and adapted by Succar and Hayakawa (1983):

$$H = A(T + 40) + B/(n - 1)(1/(-T)^{n-1} - 1/40^{n-1}) + C \quad (5)$$

$$C_p = A + B/(-T)^n \quad \text{for } T < T_{cr} \quad (6)$$

and

$$H = D + E(T - T_{cr}) \quad (7)$$

$$C_p = E \quad \text{for } T \geq T_{cr} \quad (8)$$

where H = enthalpy (kJ/kg); C_p = heat capacity (kJ/(kg °C)); T = temperature (°C); T_{cr} = initial freezing temperature (°C); A , B , C , D , E and n are fitted constants. For high water content foods such as fresh fruits, vegetables and meats, the values of n and C should be very close to 2 and 0, respectively (Succar and Hayakawa, 1983). Dehydrated foods are expected to have lower values of n and positive values of C , due to the deviations of the behavior of the food respect to that of ideal solutions (Succar and Hayakawa, 1983). Constant D is the enthalpy at T_{cr} (H_{cr}).

2.4.2. Density

Succar and Hayakawa (1983) also propose an equation for the prediction of food density below T_{cr} . This equation is

$$\rho = F + G(T_{cr} - T) + (\rho_u - F)T_{cr}/T \quad (9)$$

Here ρ_u is the density of the unfrozen food, which is considered constant and is obtained by linear regression of

the experimental data of ρ for $T > T_{cr}$. The values of constants F and G are determined simultaneously through nonlinear parameter estimation. Eqs. (5)–(9) were used to fit the experimental data.

3. Results and discussion

3.1. Experimental measurements

3.1.1. Water content and total soluble solids

Average values of water content, WC, and soluble solids, SS (°Brix), are presented in Table 1. Fig. 1 gives the variation of WC and SS with time during OD. The shape of these plots is typical for this type of process, although composition may vary more or less rapidly with time as a function of different factors (particularly sample shape and size and concentration, temperature and stirring of solution). It is important to note that equilibrium concentrations are not reached even for the higher dehydration time tested (24 h). During industrial osmotic dehydration, whose duration tends to be shorter, prediction of intermediate values is important.

Variation of food thermophysical properties with composition is a function not only of WC, but also of SS and the type of soluble solids, fundamentally their molecular weight. This is because the concentration of soluble solids influence the value of T_{cr} and the ice content for any temperature below T_{cr} and, in turn, ice content determines heat

capacity, enthalpy and density. An accurate prediction method for food properties of OD foods should take into account both concentrations (WC and SS). However, SS could be related to WC. To check whether a general relation between WC and SS of fresh and OD Kiwifruit exists we looked at all the available experimental data of WC and SS of Kiwifruit, both fresh or OD in sucrose solutions at near ambient temperatures (20–30 °C), as high temperatures of osmotic dehydration could induce changes in structure that could lead to different relations between WC and SS. These data are summarized in Table 2 and plotted in Fig. 2.

Fig. 2 shows a clear linear correlation between SS and WC, with a high correlation coefficient. This relation is

$$SS = -89.79(WC) + 87.68, \quad R^2 = 0.9637 \quad (10)$$

Therefore, it can be assumed that the characterization of composition by only WC or SS will suffice to specify thermal properties of fresh and OD Kiwifruit.

In addition, Floury et al. (2006) predict that there is a linear relation between SG and WL provided that cell membrane permeability remains constant during dehydration. Fig. 3 shows an attempt to verify this hypothesis using our data (from this work and from Marani et al., 2007) and those taken from literature (Panagiotou et al., 1998;

Table 1
Experimental results of osmotic dehydration of Kiwifruit samples

| Sample | Dehydration time (h) | WC | SS (°Brix) | H_{cr} (kJ/kg) | T_{cr} (°C) |
|--------|----------------------|-------|------------|------------------|---------------|
| S1 | 0 | 0.840 | 12.5 | 236.0 | -1.50 |
| S2 | 2 | 0.710 | 17.5 | 186.1 | -2.18 |
| S3 | 6 | 0.676 | 26.0 | 175.9 | -3.41 |
| S4 | 12 | 0.581 | 36.0 | 142.0 | -6.08 |
| S5 | 24 | 0.506 | 44.0 | NA | -10.10 |

Table 2
Experimental conditions and literature references for experimental data for osmotic dehydration of Kiwifruit in sucrose solutions

| Reference | Type of sample | Concentration of the solution (%) | Temperature of the solution (°C) |
|---------------------------|------------------------------|-----------------------------------|----------------------------------|
| Torreggiani et al. (1999) | Slice, 1 cm thick | 70 | 25 |
| Talens et al. (2001) | Disk, $d = 4$ cm; $h = 1$ cm | 35, 45, 55, 65 | 30 |
| Marani et al. (2007) | Disk, $d = 4$ cm; $h = 1$ cm | 69 | 30 |
| This work | Cube, 1 cm side | 60 | 20 |

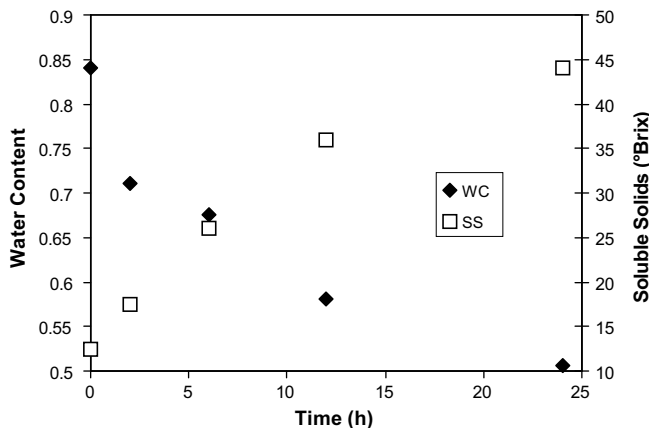


Fig. 1. Variation of WC and SS with time for dehydration of Kiwifruit cubes in saturated aqueous solution of sucrose at 20 °C.

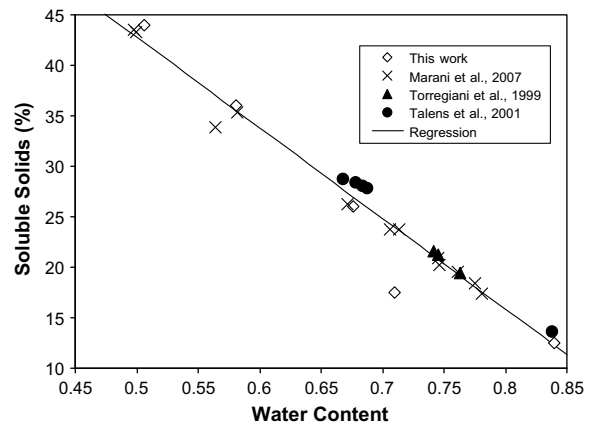


Fig. 2. Experimental data and linear regression equation of SS vs WC for fresh and OD Kiwifruit.

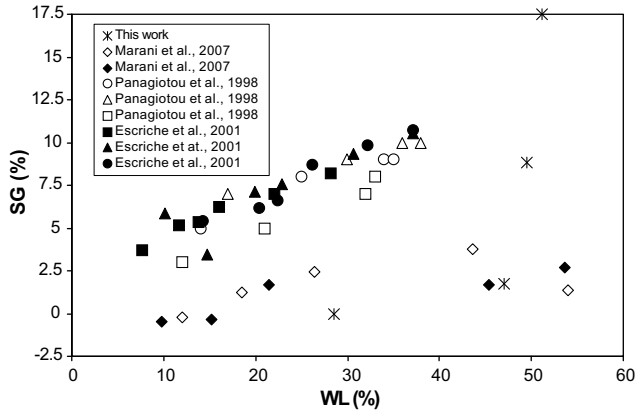


Fig. 3. Experimental data of SG vs WL for OD Kiwifruit in sucrose solution.

Escriche et al., 2001) for Kiwifruit dehydration in sucrose solutions under different conditions. For each set of data, SG has a roughly linear variation with WL up to values of WL of about 35, which is consistent with the hypothesis of Flourey et al. (2006). At higher WL values the relationship between SG and WL is less certain, probably due to changes in cell membrane structure which varies its permeability to solutes (Flourey et al., 2006). Nevertheless, the slope of the linear portion depends on food size and shape and working conditions, so it is specific to each data set and experimental results cannot be generalised to other conditions.

3.1.2. Enthalpy, heat capacity and initial freezing temperature

Measured values of H and C_p are presented in Figs. 4 and 5 for samples S1–S4 (data for sample S5 were inadvertently lost). For the same temperature, H lowers as WC decreases and simultaneously the initial freezing point T_{cr} lowers. Both results are consistent with the theory and are related to the decrease in WC and increase in SS.

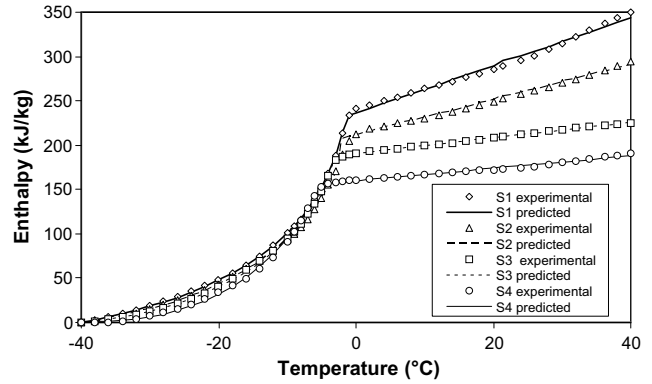


Fig. 4. Comparison of experimental values of enthalpy for Kiwifruit, against those predicted by Eqs. (5) and (7) (symbols: experimental; lines: predicted).

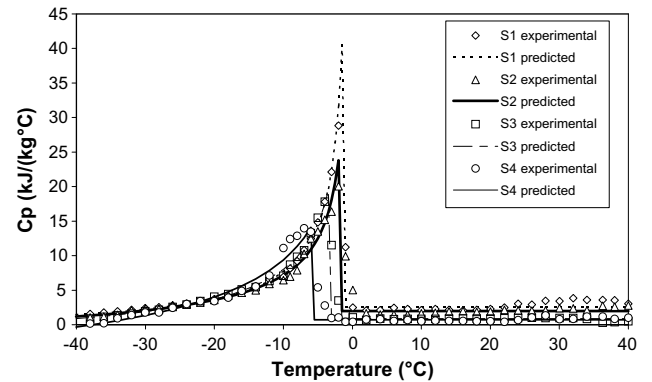


Fig. 5. Comparison of experimental values of enthalpy for Kiwifruit, against those predicted by Eqs. (6) and (8) (symbols: experimental; lines: predicted).

From these data the values of H_{cr} and T_{cr} for samples S1–S4 were determined and are presented in Table 1. Fig. 6 shows the T_{cr} as a function of WC together with literature data of T_{cr} for fresh Kiwifruit and T_{cr} for apple, pear, strawberry, peach and melon, fresh and OD in

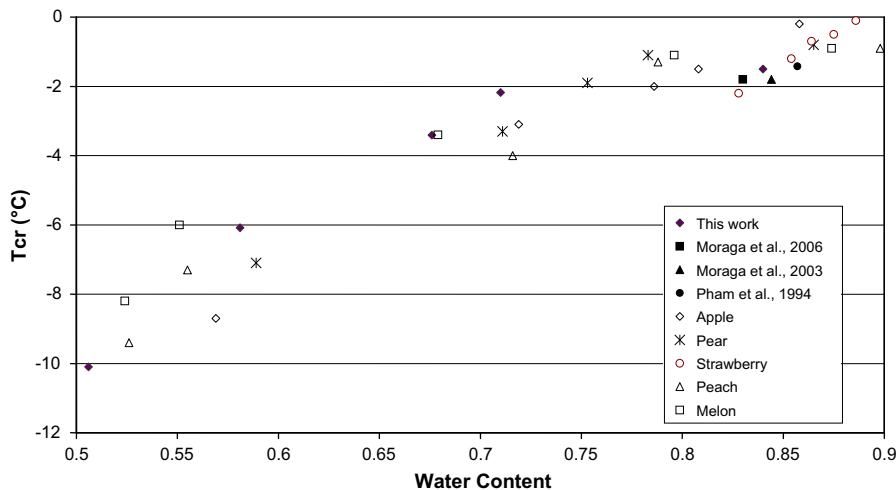


Fig. 6. Experimental data of T_{cr} vs WC for fresh and OD fruits. Filled symbols: Kiwifruit; empty symbols: other fruits.

sucrose solution, taken from previous works by the authors (Tocci et al., 1998; Tocci and Mascheroni, 1998). Despite differences in tissue composition among the different fruits studied and the cited problems in accuracy of measurements, these results show a clear trend between WC and all measured T_{cr} .

3.1.3. Density

Experimental results of the variation of ρ with T above and below T_{cr} , for samples S1, S3 and S5 are presented in Fig. 7. Two distinct characteristics can be seen:

- A clear decrease of ρ in the frozen zone, and
 - At equal temperatures, a higher value the lower WC is.
- These trends are related to the increase in total solids content of higher ρ than liquid water or ice with increased dehydration.

3.2. Prediction equations

3.2.1. Enthalpy and heat capacity

Table 3 presents the fitted values of constants in Eqs. (5) and (7) to the experimental H vs T data together with their respective correlation coefficients. In all cases, the fits are very good, with very high correlation coefficients (higher than 0.99 in most cases). Fig. 4 shows the experimental data together with the prediction equations. The good fits support the sound physical basis of Eqs. (5) and (7).

The analysis covers a wide range from untreated fruits with WC = 0.84 and no added solids (S1) to OD fruit with WC = 0.58 and three times the SS of fresh fruit (S4). The main limitation for the use of this method is that no systematic way to predict the values of constants in Eqs. (5) and (7) as a function of known variables was found. These constant must always be obtained by fitting specific experimental data. Therefore, these equations cannot be used for WC different from those for which experimental data is available.

In Eq. (7) constant D is the value of H_{cr} predicted by the method of Succar and Hayakawa. Table 3 shows that the experimental H_{cr} is less than 2% different from the fitted value of D .

3.2.2. T_{cr}

Experimental data of T_{cr} vs WC for Kiwifruit (both own data and from the literature Pham et al., 1994; Moraga et al., 2003; Moraga et al., 2006), were fitted to linear, logarithmic and polynomial functions of second and third order. The two that had the best accuracy were

Logarithmic : $T_{cr} = 14.733 \text{Ln}(\text{WC}) + 1.3307$,
 $R^2 = 0.9008$ (11)

Polynomial : $T_{cr} = 129.37(\text{WC})^3 - 356.67(\text{WC})^2 + 326.11(\text{WC}) - 100.55$,
 $R^2 = 0.9954$ (12)

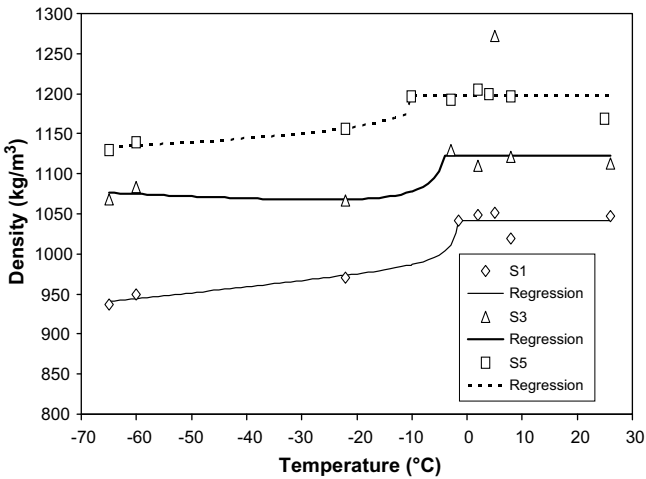


Fig. 7. Variation of density of fresh and dehydrated Kiwifruit with temperature. Symbols: experimental data; lines: predicted by Eq. (9).

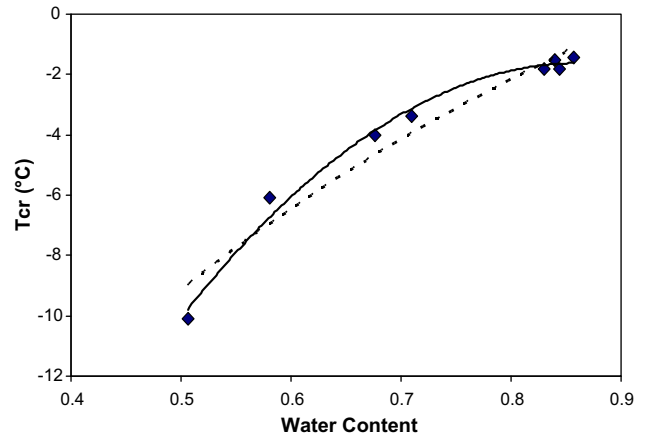


Fig. 8. Regression equations for the prediction of T_{cr} from WC: (♦) experimental data; regressions: full line: polynomial; dotted line: logarithmic.

Table 3

Values for constants in Eqs. (5) and (7) obtained from the fitting of experimental data of H vs T for fresh and OD Kiwifruit, together with their correlation coefficients

| A (kJ/(kg °C)) | B (kJ/(°C ⁿ)/kg) | C (kJ/kg) | n | R^2 (Eq. (5)) | H_{cr} (kJ/kg) | D (kJ/kg) | E (kJ/(kg °C)) | R^2 (Eq. (7)) |
|------------------|--------------------------------|-------------|-------|-----------------|------------------|-------------|------------------|-----------------|
| -1.521 | 58.216 | 0.078 | 0.803 | 0.998 | 236.0 | 232.6 | 2.679 | 0.985 |
| -3.617 | 41.159 | 0.734 | 0.582 | 0.999 | 186.1 | 186.3 | 1.996 | 0.996 |
| -4.312 | 51.901 | -0.562 | 0.624 | 0.998 | 175.9 | 175.3 | 0.876 | 0.998 |
| -19.110 | 56.968 | 1.754 | 0.301 | 0.998 | 142.0 | 139.7 | 0.709 | 0.983 |

Table 4

Values for constants in Eq. (9) obtained from the fitting of experimental data of density of fresh and OD Kiwifruit, together with their correlation coefficients

| Sample | F (kg/m ³) | G (kg/(m ³ °C)) | ρ_u (kg/m ³) | R^2 (Eq. (9)) |
|--------|--------------------------|------------------------------|-------------------------------|-----------------|
| S1 | 0.983962 | −0.000706026 | 1.0411986 | 0.993 |
| S3 | 1.04853 | 0.000383813 | 1.1299074 | 0.983 |
| S5 | 1.14608 | −0.000321857 | 1.1774727 | 0.987 |

Fig. 8 presents both regressions. It is clear that the best fit was the polynomial model (Eq. (12)).

3.2.3. Density

Table 4 and Fig. 7 present the results of the fitting of experimental data of ρ vs T to Eq. (9) for the three samples. Despite the scarce experimental data available, they follow the expected trend and are adequately predicted by Eq. (9). As in the case of H and C_p , the same prediction equation is valid for fresh and partially dehydrated Kiwifruit.

4. Conclusions

Measured values of SS and WC over time follow the expected trends for OD. Equilibrium concentrations are not reached during the test period (24 h).

There was a linear relationship between WC and SS so the use of only one of these values is sufficient to characterize food composition and thermal properties during OD. The relationship between SG and WL tended to be sample specific.

Measured values of H and C_p at the same temperature reduced and T_{cr} reduced as OD increased.

Density was constant above T_{cr} and reduced below T_{cr} , but there was an increase of ρ with greater dehydration, due to the higher solids content and the shrinkage of the food pieces.

Equations for H , C_p and ρ as functions of T accurately fitted the measured data for both fresh and dehydrated Kiwifruit but there was no obvious relationship between the values of the coefficients and the degree of dehydration so they cannot be used for WC different from those for which they were fitted.

Initial freezing temperature T_{cr} could be related to WC by a polynomial equation with high accuracy that was valid for both fresh and dehydrated Kiwifruit.

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