

Thermal degradation kinetics of nutrients in orange juice heated by electromagnetic and conventional methods

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Abstract

Newer processing technologies like infrared, microwave processing are being harnessed to optimize the processes to ensure minimum loss of the vital nutrients in processed foods. Vitamin C is an important nutrient known for its potential antioxidant, anti-cancerous and other health promoting properties. Orange juice is a very popular and rich source of vitamin C. The present research focuses on the status of the vitamin C during thermal treatment of orange juice heated by different methods. The study includes a comparative study of kinetics of vitamin degradation and changes in visual colour as an index of carotenoids. The degradation kinetics of vitamin C and colour in terms of reaction rate constant, destruction kinetics, enthalpy and entropy for different methods of heating are discussed. The destruction of vitamin C was influenced by the method of heating and the temperature of processing. The degradation was highest during microwave heating due to uncontrolled temperature generated during processing. Out of the four methods studied, ohmic heating gave the best result facilitating better vitamin retention at all temperatures. The visual colour is generally used as an index of the carotenoid content. The activation energies for both vitamin and colour were within the range of literature reported values of 7.54–125.6 kJ/mol. The activation enthalpies agreed with the literature values of vitamin destruction of other food products. The *z* values also were within the literature values of 20–30 °C for vitamin destruction, except for microwave heating.

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1. Introduction

There are a number of potential opportunities for exploiting the benefits of Electromagnetic technologies like ohmic, infrared and microwave heating in food processing. These technologies can be applied in a variety of ways depending on the process requirements and the consumer demand. Processing of food products is a

necessary requirement for extending the shelf life. However, such processing generally involves heat treatment that can reduce organoleptic quality. Over the years, researchers have optimized time/temperature profiles to minimize the exposure of food to heat. Further, the newer process technologies may have the potential to reduce or even eliminate heat exposure. Some of these processes are not new, but have recently made significant advances towards commercialization.

Methods for quantitative measurement of any compound are imperative for probing its function. This is especially true for compounds such as vitamin C because of its ubiquitous presence in all biological systems and participation in biological processes as diverse as

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digestion, absorption, endocrinology, anti-carcinogenicity, collagen formation, cataract prevention and detoxification. The potential antioxidant effect of vitamin C has been the subject of numerous studies. It is reported that vitamin C helps in the prevention of cancer (Byers & Perry, 1992; Wittes, 1985). The evidence that the antioxidant nutrient such as vitamin C will play an important role in our health and well being is growing rapidly (Johnson, 1995).

Interest in nutrient evaluation as one of the quality parameters has been triggered by the government regulation in nutritional labeling of processed products. The relative instability of vitamin C under usual conditions of food storage and processing is well documented (Erdman & Klein, 1982). However, the prediction of vitamin C losses is complicated due to lack of information about the mechanism of degradation and the factors that influence its stability during processing.

Fruits and vegetables are important low cost foods containing low levels of fat and high levels of vitamins, minerals and fibres (Masrizal, Giraud, & Driskell, 1997) and are the major sources of vitamin C in particular. There has been an increase in the number of people adapting to vegetarian diet due to various health benefits. Pasteurization of fruit juice is a widely used method for the thermal preservation of fruit juices. As the consumption of such products continues to increase, so does their contribution in providing important amounts of vitamin C, provitamin A and antioxidant activity in the diet (Hussein et al., 2000).

To predict the nutrient deterioration, the knowledge of kinetics of vitamin C degradation including the reaction rate as a function of temperature of processing is required. This kinetics has been examined extensively in model systems with particular attention to intermediate moisture foods and during storage studies. Data available for the vitamin C losses in actual food systems are insufficient to calculate the kinetic parameters during heat treatment. Hence, it is necessary to study the effect of different processing temperatures on the retention of vitamin C in the product and kinetic modeling to predict the losses during processing by different heating methods. Visual colour of fruits has been correlated with the carotenoid concentration (Ahmed, Kaur, & Shivhare, 2002). Attempts have been made to study the kinetics of thermal degradation of fruits during processing (Ahmed, Shivhare, & Sandhu, 2002; Kajuna, Bilanski, & Mittal, 1998). However, not many reports are available on kinetics of colour degradation of orange juice.

With reference to kinetics of destruction of vitamin C in food systems, different buffer systems and real products with different processing methods have been examined (Frias, Oliveira, Cunha, & Oliveira, 1998; Hussein et al., 2000; Lathrop & Leung, 1980; Lee & Nagy, 1988; Lima, 1997; Lima, Heskitt, Burianek, Nokes, &

Sastry, 1999; Rao, Lee, Katz, & Cooley, 1981; Robertson & Samaniego, 1986; van den Broeck, Ludikhuyze, Weemaes, Loey, & Hendrickx, 1998). Many of these studies are related to the storage of pasteurized juices or higher temperatures of processing. It has been reported that vitamins are heat labile and high processing temperatures can degrade these nutrients. Several studies have been carried out to quantify the rate of destruction of nutrients at elevated temperatures. Large differences in kinetic parameters, activation energy and frequency factors have been observed. This variation can be attributed to the fact that nutrient destruction is a complex function of many variables like, pH, oxygen, salt, sugar, presence of enzymes, amino acids and metal catalysis (Fennema, 1996).

Conventional pasteurization requires longer heating time leading to deterioration of product quality. Electromagnetic heating, on the other hand, has been successfully used for the efficient pasteurization of food products in the recent years. Though, the influence of temperature on the degradation kinetics of vitamin C has been extensively studied and reported, the influence of method of processing and in particular, electromagnetic heating has not been much studied.

The objective of the present study is to examine the retention and kinetics of degradation of vitamin C during the electromagnetic heating in comparison with conventional heating. The electromagnetic processing includes infrared, ohmic and microwave heating.

2. Materials and methods

The fresh sweet orange (*Citrus sinensis* Osbeck) fruits used for this investigation were purchased from the local fruit market and was kept at 4°C until the experiments were carried out (not more than 1 day).

2.1. Experimental setup

2.1.1. Ohmic heating system

Since a standard system was not available, a laboratory setup was developed at the Food Engineering Centre of this institute. The ohmic heating system (Fig. 1) consists of a PVC Tee (0.05 m cm id), with straight ends, closed with rubber stoppers and stainless steel circular electrodes. The electrodes were held in place by silicon sealant (GE Bayers Silicones, India) to prevent leakage of constituents. The distance between the two electrodes was 55 mm. Diameter of the electrode was 50 mm. The setup provided a working volume of 100 ml of orange juice. Power connection was from 230 V, single-phase AC line. The sample was subjected to 42 V/cm electric field with a minimum of 2.5 A to a maximum of 5.0 A during heating. A built-in temperature controller maintained the set temperature during processing within $\pm 1^\circ\text{C}$.

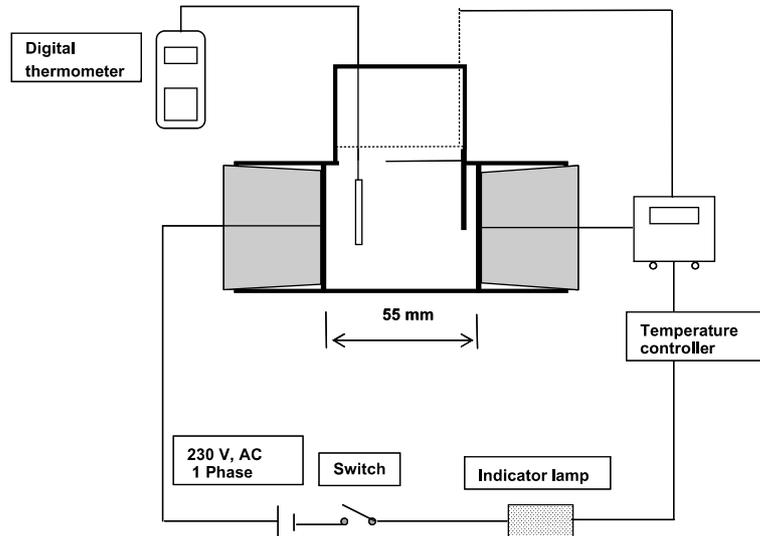


Fig. 1. Experimental setup of ohmic heater.

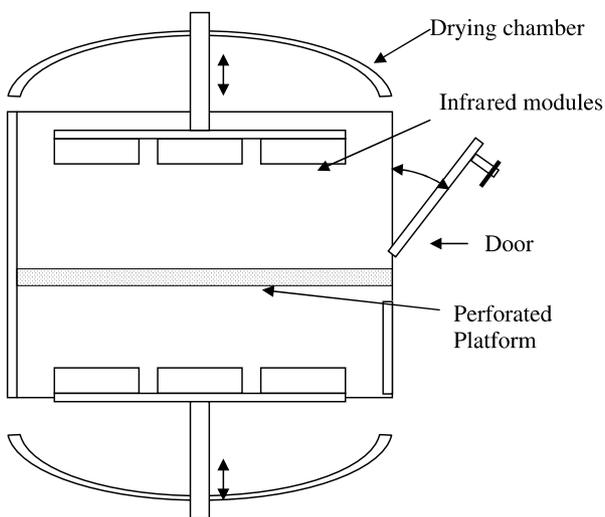


Fig. 2. Batch type infrared heater.

2.1.2. Infrared heating system

The infrared heating system (Fig. 2) developed at the Food Engineering Centre of the Institute was used for the study. The system consisted of a heating chamber with infrared modules (250 W), with reflectors to direct infrared waves on to a platform. The chamber had a door for easy handling of samples during processing. The distance between platform and IR source could be adjusted for varying the intensity. Power connection was from 230 V, single-phase AC line. A temperature controller maintained the set temperature during processing to $\pm 1^\circ\text{C}$.

2.1.3. Microwave heating system

The domestic microwave oven (BPL SANYO, BMC 900 T, India) was used for processing. Juice (100 ml)

was transferred into a clean 150 ml beaker (Borosil, India.) and processed at different power levels. The beaker was placed towards the periphery of the turntable, where maximum power incidence was observed (Ramesh, Wolf, Tevini, & Bogнар, 2002). Temperature was measured intermittently stopping the oven in a separate experiment, keeping the experimental conditions constant.

2.1.4. Conventional processing

Juice (100 ml) was transferred into a clean 250 ml conical flask (Borosil, India) and was processed at different temperatures in a shaker water bath (Haake, Germany). Shaking the juice during heating simulating the turbulent conditions of a conventional heating system.

A digital temperature indicator (Ebro, Germany) was used to measure the product temperature during the experiments.

2.1.5. Sample preparation

Oranges were squeezed manually and the juice was collected in a conical flask kept in an ice bath. The samples were processed at different time temperature combinations as shown in Table 1. Zero time was set at the instant the juice reached the desired temperature as indicated by the thermocouple and it took about 1–3 min for electromagnetic heating and upto 10 min for conventional heating. The lag time depends on the targeted temperature, longer time being associated with lower temperature. For microwave heating, time was set on the microwave oven panel and was initiated with the pressing of the start button. The sample was immediately removed after processing for the desired time and stored at 4°C till analysis.

Table 1
Thermal treatment conditions for orange juice heating

| Conventional, ohmic, infrared Temperature (°C) | Microwave | | Time (min) |
|---|------------------|---------------------------------------|-------------------|
| | Power levels (W) | Approximate temperature attained (°C) | |
| 50 | 245 | 100 | 1, 5, 8, 10, 15 |
| 60 | 315 | 105 | 1, 2, 3, 5, 10 |
| 75 | 385 | 110 | 1, 5, 8, 10 |
| 90 | 455 | 125 | 0.5, 1, 1.5, 2, 3 |

2.1.6. Sampling

For each processing temperature, unprocessed sample was stored at 4°C, and designated as control. From the processed juice sample, 20 ml juice was centrifuged at 6000 rpm for 15 min and at -4°C, in a refrigerated centrifuge (Remi Pvt. Ltd., India.). The supernatant was filtered through filter paper (Whatman number 3); 1 ml of filtrate was diluted with 1 ml of 0.8% meta-phosphoric acid and was filtered through a 0.45° Millipore filter. 10 µl of the juice was injected into HPLC. Three injections were prepared from samples corresponding to each processed sample.

3. Methodology

3.1. Vitamin C

The quantitative analysis of vitamin C were carried out by high performance liquid chromatography (HPLC) (LC 6 A, Shimadzu, Kyoto, Japan) using a C18 (ODS) column (5 µm × 30 cm, Shimadzu) with an UV detector set at 254 nm. Meta-phosphoric acid (0.8%) was used as the mobile phase at a flow rate of 0.5 ml/min. The analyses were carried out at ambient temperature in triplicate. Standard calibration graph was used to calculate the concentrations.

Based on the peak areas corresponding to the retention time of 8.5 min, the concentration was calculated from the standard graph as mg/ml on moisture free basis. The percentage retention of vitamin C was calculated using Eq. (1) (Murphy, Criner, & Gray, 1975).

%Retention

$$= \frac{\text{Concentration of vitamin C in processed juice (A)}}{\text{Concentration of vitamin C in un-processed juice (A}_0\text{)}} \times 100 \quad (1)$$

The values were plotted as indicated in Fig. 3.

4. Kinetic data analysis

Nutrient destruction is described using the reaction rate and the dependence of reaction rate on temperature. Parameters used are the reaction rate constant (k) and the Arrhenius activation energy (E_a). The kinetic data were analyzed as described by Van Boekel (1996).

Vitamin loss is generally considered to follow first order kinetics.

$$-dC/dt = kC \quad (2)$$

where C is the concentration of nutrient, t is time and k is the reaction constant (time^{-1}). If C_0 is the concentration of the vitamin C at time zero, integration of the Eq. (2) yields:

$$C = C_0 \exp(-kt) \quad (3)$$

Taking natural logarithm, we have

$$\ln C = \ln C_0 - kt \quad (4)$$

For the first order reaction, a plot of 'ln C ' against time ' t ' will be a straight line, and the rate constant is represented by the slope. The rate constant values for the degradation of vitamin C are reported in Table 2.

Temperature dependence of a reaction is described by the Arrhenius equation:

$$k = k_0 \exp(-E_a/RT) \quad (5)$$

where, k_0 = frequency factor or the Arrhenius constant (time^{-1}), R is the universal gas constant (8.3145 J/molK), and T is the absolute temperature (K).

Substituting for k from Eq. (5) in (4)

$$\ln C/C_0 = k_0(\exp -E_a/RT) \cdot t \quad (6)$$

The decimal reduction time (D values), the time required to reduce the vitamin C concentration by 90%, was related to reaction rate constants by

$$D = 2.303/k \quad (7)$$

The D values were then described as direct exponential function of temperature:

$$\ln \left(\frac{D_1}{D_2} \right) = \frac{(T_2 - T_1)}{z} \quad (8)$$

where D_1 and D_2 are decimal reduction time at temperatures T_1 and T_2 , respectively, with z representing the temperature required for 1 log cycle reduction in D value.

Enthalpy (ΔH) and entropy (ΔS) of activation were obtained by the regression analysis of $\ln(k)$ on $1/T$ from the equation derived from transition state theory (Ariahu & Ogunsua, 2000)

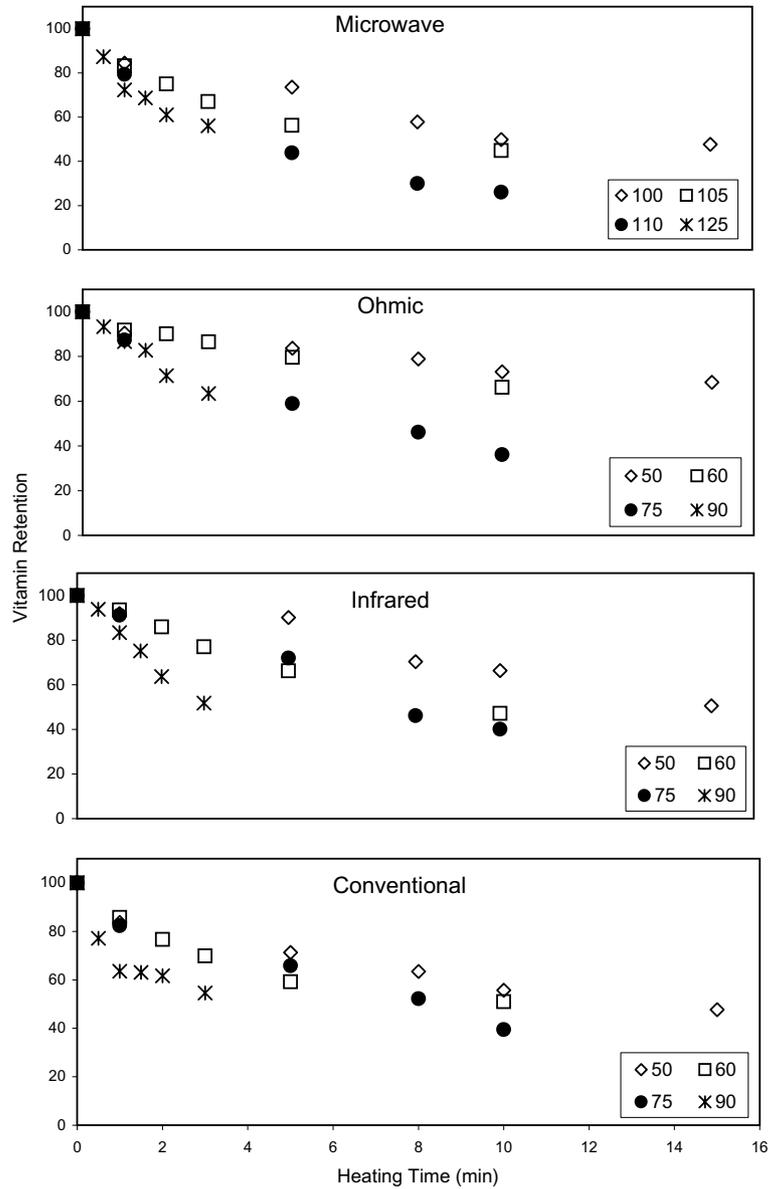


Fig. 3. Vitamin C retention during heating by different methods at different temperatures.

$$\ln\left(\frac{k}{T}\right) = \left[\ln\left(\frac{k_s}{h}\right) + \left(\frac{\Delta S}{R}\right)\right] - \left[\left(\frac{\Delta H}{R}\right)\left(\frac{1}{T}\right)\right] \quad (9)$$

where k_s is Boltzmann constant ($1.380658 \times 10^{-23} \text{ J K}^{-1}$), h is Planck's constant ($6.6260755 \times 10^{-34} \text{ J s}$) and R is universal gas constant ($8.3145 \text{ J mol}^{-1} \text{ K}^{-1}$).

4.1. Colour

Degradation of colour was based on Hunter 'a' and 'b' values and the combination Hunter ($a \times b$) value was used to represent the thermal colour change as suggested by Ahmed et al. (2002) for papaya puree. Visual colour was measured using Hunter colorimeter model D25 optical sensor (Hunter Associates Laboratory, Reston, VA, USA) in terms of L (lightness), a (redness

and greenness) and b (yellowness and blueness). The instrument ($45^\circ/0^\circ$, 2° observer) was calibrated with a standard white tile ($L = 90.55$, $a = -0.71$, $b = 0.39$). A glass petri dish (50.5 mm internal diameter) containing the processed juice was placed above the light source and covered with a white plate and post process Hunter L , a , b values were recorded.

4.2. Fractional conversion kinetics model

Degradation has been found to be first order reaction kinetics (Ahmed et al., 2002; Ahmed, Shivhare, & Raghavan, 2000; Ahmed, Shivhare, & Singh, 2000; Gunawan & Barringer, 2000; Hutchings, 1994; Shin & Bhowmik, 1994; Steet & Tong, 1996; Weemaes, Ooms,

Table 2
Rate constant and thermal resistance parameters for vitamin C degradation

| Heating method | Temperature (°C) | <i>k</i> -value (min ⁻¹) | <i>D</i> -value (min) |
|----------------|------------------|--------------------------------------|-----------------------|
| Conventional | 50 | 0.0351 | 65.67 |
| | 60 | 0.0462 | 49.81 |
| | 75 | 0.0852 | 27.02 |
| | 90 | 0.1784 | 12.91 |
| Ohmic | 50 | 0.0240 | 95.96 |
| | 60 | 0.0393 | 58.55 |
| | 75 | 0.0971 | 23.72 |
| | 90 | 0.1571 | 14.66 |
| Infrared | 50 | 0.0444 | 51.91 |
| | 60 | 0.0760 | 30.32 |
| | 75 | 0.0969 | 23.76 |
| | 90 | 0.2284 | 10.08 |
| Microwave | 100 | 0.0504 | 45.69 |
| | 105 | 0.0760 | 30.32 |
| | 110 | 0.1352 | 17.04 |
| | 125 | 0.1944 | 11.84 |

Loey, & Hendrickx, 1999). Following this, a convenient fractional conversion factor has been derived (Ahmed et al., 2002). For an irreversible first order reaction kinetics, the rate constant at any constant temperature can be determined through fractional conversion, *f*:

$$f = \frac{(C_0 - C)}{(C_0 - C_\infty)} \quad (10)$$

First order reaction in terms of the fractional conversion may be represented as

$$\ln(1 - f) = -kt \quad (11)$$

The reaction rate values for the degradation of colour is given in Table 3. The Arrhenius parameters and *E_a* values were further calculated as described earlier.

Table 3
Rate constant and thermal resistance parameters for colour degradation

| Heating method | Temperature (°C) | <i>k</i> -value (min ⁻¹) | <i>D</i> -value (min) |
|----------------|------------------|--------------------------------------|-----------------------|
| Ohmic | 50 | 0.0183 | 125.85 |
| | 60 | 0.1103 | 20.88 |
| | 75 | 0.2885 | 7.98 |
| | 90 | 0.5697 | 4.04 |
| Infrared | 50 | 0.0706 | 32.62 |
| | 60 | 0.1294 | 17.80 |
| | 75 | 0.3256 | 7.07 |
| | 90 | 0.6108 | 3.77 |
| Microwave | 100 | 0.5848 | 3.94 |
| | 105 | 0.6291 | 3.66 |
| | 110 | 0.6875 | 3.35 |
| | 125 | 0.7817 | 2.95 |

4.3. Statistical analysis

The data were analyzed and the Kinetic parameters were determined using 'Microsoft Excel' software (Version 5, Microsoft Corporation, Redmond, WA, USA).

5. Results and discussion

5.1. Degradation of vitamin C

The vitamin C degradation at various temperatures heated by different methods is indicated in Fig. 3. There were wide variations in the vitamin C content of the fresh juice (33.13 ± 11.55 mg/100 g). This is mainly due to the seasonal variations as the experiments were spread over few months during the season. This seasonal variation is caused as the vitamin C content increases with fruit maturation. The range of values obtained was comparable with the literature reported values (The Natural Food Hub, 2001; USDA, 2001). However, this variation was normalized by converting the values to percentage retention as explained earlier. The comparison indicated that the heating method had a definite influence on the retention of vitamin C. Under each heating method, temperature had a greater influence and the degradation was rapid at higher temperatures. The temperature profile for different method of heating is indicated in Fig. 4. Ohmic heating was most rapid followed by microwave, and infrared heating. This is mainly due to the internal heat generation under electromagnetic heating. Conventional method had a longer lag period and the time to reach the targeted temperature was about 5 min. Though ohmic heating was rapid, the temperature controller built into the system, monitored the temperature with in the range of targeted temperatures. However, with microwave heating, this facility was not available and the temperature increased uncontrolled, depending on the power level used. Ohmic heating method facilitated higher nutrient retention at all temperatures, compared to other methods, followed by infrared and conventional heating as indicated in Fig. 3. The degradation was highest in microwave, which may be attributed to uncontrolled temperatures generated during processing, which exceeded 100°C reaching 125°C and due to the heat labile nature of vitamin C.

The degradation of vitamin C for all methods of heating followed first order kinetics as indicated in Fig. 3. The reaction rate constants (*k*) and thermal resistance value (*D*) for vitamin C degradation during different methods of heating and at different temperatures are indicated in Table 2. The *k* value further confirmed the influence of temperature. The activation energy values obtained from these data were comparable to the reported values for vitamin C in orange juice. The highest

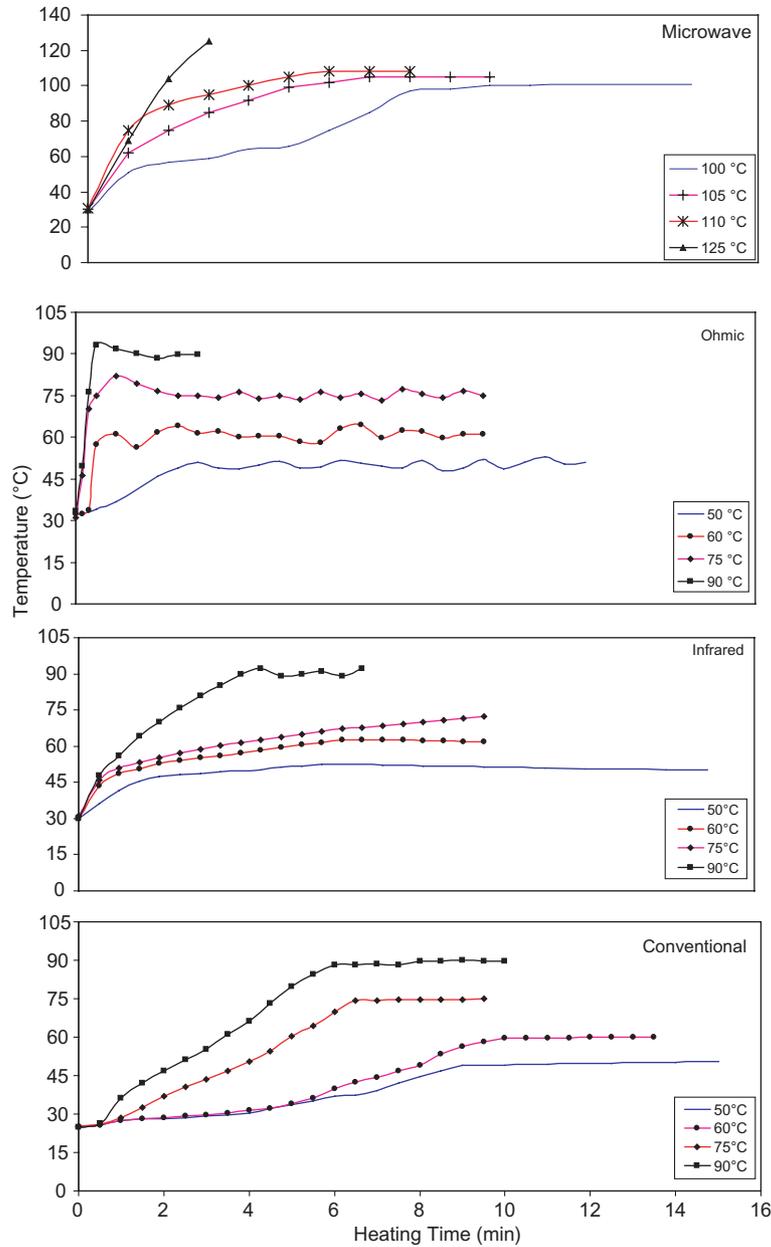


Fig. 4. Temperature profiles during heating by different method.

value was for microwave heating, due to internal heat generation and uncontrolled temperature increase. The next highest value was for ohmic heating, followed by conventional and infrared heating. The higher value of activation energy for ohmic heating is due to the higher dielectric properties of the juice and instantaneous heat generation due to passage of electric current. Higher activation energy implies that a smaller temperature change is needed to degrade a specific compound more rapidly. The activation energy values varied with the method of heating. The values were 64.78 ± 2.63 kJ/m K for microwave; 47.27 ± 0.80 kJ/m K for ohmic; 39.84 ± 0.61 kJ/m K for conventional and 37.12 ± 1.66 kJ/m K for infrared heating. In general the activa-

tion energy values for different heating methods were within the range of the reported values of (7.54–125.6 kJ/mol) (Lee & Labuza, 1975). The variation in the activation energy values for the microwave heating was high (SD = 2.63 kJ/m K) indicating the non-uniformity of temperature during processing. The activation energy values were also comparable to the literature values reported by several authors. Dio Alvarado and Viteri (1991) reported values ranging from 38.9–46.5 kJ/mol with a temperature range of 20–92°C during conventional heating, Johnson, Braddock, and Chen (1995) reported a value of 115.3 kJ/mol with a temperature range of 70–98°C during conventional heating, Lima et al., 1999 reported a value of 52.8 kJ/mol during

conventional heating and 52.3 kJ/mol for ohmic heating in the temperature range of 65–90°C and Assiry (1996) has reported a value of 29.3 kJ/mol in the temperature range of 40–80°C during ohmic heating. The lower value for infrared heating suggests that within the temperature range studied, vitamin C was more stable. This further strengthens the fact that vegetables are sensitive to higher temperatures and higher vitamin C deterioration occurs at higher temperatures of above 100°C.

The activation enthalpy (ΔH) and entropy (ΔS) for vitamin C degradation were found to vary with the method of heating. The values of ΔH and ΔS were 61.60 and 0.11 kJ/mK for microwave; 44.35 and 0.14 kJ/mK for ohmic; 37.03 and 0.16 kJ/mK for conventional and 34.20 and 0.17 kJ/mK for infrared heating, respectively. In general (ΔH) is a measure of energy barrier that must be overcome by reacting molecules and is related to the strength of the bonds, which are broken and made in the formation of the transition state from the reactants. ΔS is related to the number of molecules with appropriate energy that can actually react. The value of ΔS also includes steric and orientation requirements along with solvent effects.

The two values for different heating methods obtained were closer to that reported by Labuza (1980) obtained in the vitamin C degradation studies by Jensen (1969) for sea weed ($\Delta H = 31.32$ kJ/molK; $\Delta S = 0.18171$ kJ/molK) and by (Kirk, 1978) for cereal based foods ($\Delta H = 33.49$ kJ/molK; $\Delta S = 0.18631$ kJ/molK). The higher value for microwave heating is mainly due to the internal heat generation and uncontrolled temperature rise as explained above. The thermal resistance approach to vitamin C destruction in terms of z value were within the prescribed limit of vitamin destruction of 20–30°C (Hallstrom, Skjolderbrand, & Tragradh, 1988; Lund, 1977). The z values were 19.23 for microwave; 20.70 for ohmic; 24.39 for conventional and 26.25 for infrared heating, respectively. Also this value is comparable to the values of other vitamin destruction as well (van den Broeck et al., 1998; Viollota & Hawkes, 1986).

5.2. Degradation of colour

The degradation of visual colour as expressed by the combination ($a \times b$) values followed first order kinetics

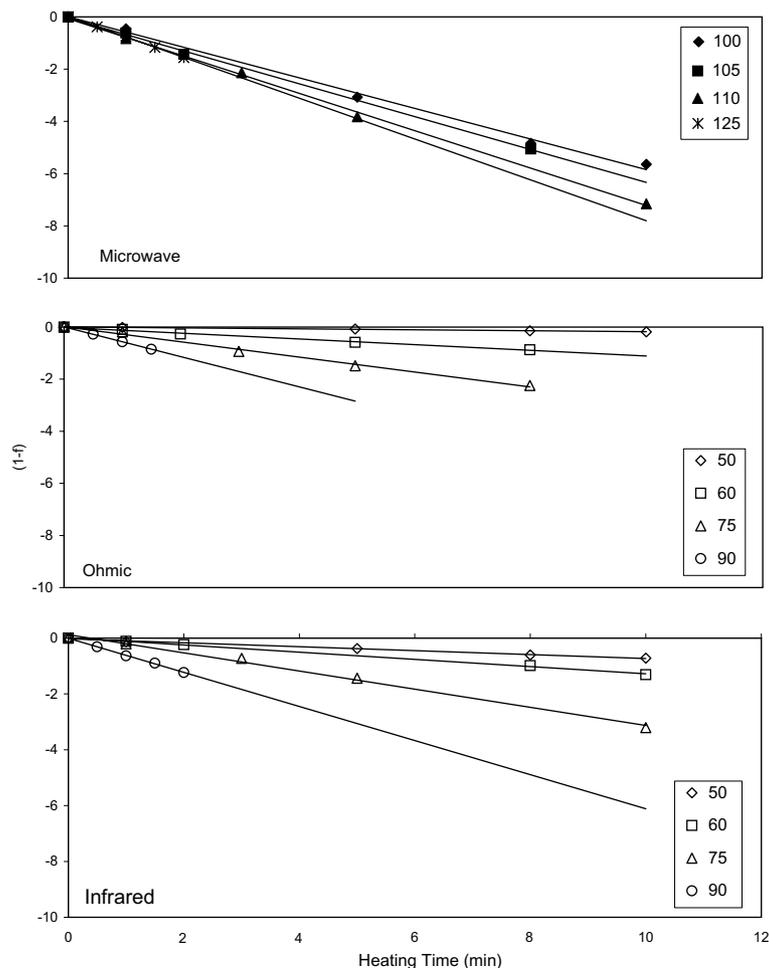


Fig. 5. Kinetics of visual colour at different temperatures.

for all methods of heating and a typical plot is shown in Fig. 5. The reaction rate constants (k) were obtained from these plots. The R^2 values were greater than 0.992 in all the cases. The k value confirmed the influence of temperature on colour degradation. The values are reported in Table 3. The activation energy obtained from these data for different heating methods fall in the range of the reported values of 7.54–125.6 kJ/mol (Lee & Labuza, 1975). The values were 14.15 ± 0.63 kJ/mK for microwave; 79.92 ± 0.68 kJ/mK for ohmic and 53.31 ± 0.97 kJ/mK for infrared heating. The highest value was for ohmic heating followed by infrared and least for microwave. This may be due to the instantaneous heat generation in ohmic heating due to the passage of current and higher dielectric properties of the juice. Higher activation energy implies that a smaller temperature change is needed to degrade colour more rapidly.

The activation enthalpy (ΔH) and entropy (ΔS) for colour degradation were 10.95 and 0.17 kJ/mK for microwave; 77.08 and 0.04 kJ/mK for ohmic and 50.47 and 0.11 kJ/mK for infrared heating, respectively. In general ΔH and ΔS had a broader range than vitamin C indicating the influence of heating and temperature on colour degradation. The ΔH values were closer to E_a values in the degradation of both vitamin C and colour. The thermal resistance z value were 87.72 for microwave; 12.32 for ohmic and 18.35 for infrared heating, respectively. This value of z is within the prescribed limit of vitamin destruction of 20–30°C (Hallstrom et al., 1988; Lund, 1977) except for microwave. This indicates that by microwave heating, the colour degradation requires higher temperature change. This is further confirmed by the lower activation energy for colour degradation by microwave heating.

The influence of different heating methods and temperatures on the vitamin and colour degradation were studied. This study of kinetics of nutrient degradation will facilitate the optimization of the heating process. The study indicated that microwave processing leads to higher degradation of vitamin C but lower degradation of colour. Ohmic heating indicated lower degradation of vitamin C than microwave and infrared heating. Of the four methods studied ohmic heating resulted in maximum retention of vitamin C. Further investigation needs to be carried out to optimize the nutrient (vitamin C, carotenoids) and colour retention and sterilization effect of infrared heating to arrive at a suitable time-temperature profile.

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