Colour changes in thermally processed cupuaçu (*Theobroma grandiflorum*) puree: critical times and kinetics modelling

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Summary Colour changes in thermally treated cupuaçu (*Theobroma grandiflorum*) fruit puree were modelled mathematically. Isothermal experiments in the temperature range of 80–115 °C were performed and colour was measured by a tristimulus colorimeter. At each temperature total colour difference (TCD*) increased and normalized L* decreased with processing time, both following a power law model. The power of the model was temperature-dependent and described by the Arrhenius law. To estimate the model constants, a one-step non-linear regression was performed on all data. Activation energies of 31 and 36 kJ/mol were determined for TCD* and normalized L*, respectively. These results should prove useful in the design of pasteurization processes which minimize colour changes in cupuaçu puree.

Keywords Cupuaçu, Kinetics, pasteurization, total colour difference, tristimulus colorimeter.

Introduction

Cupuaçu (*Theobroma grandiflorum*) is a tropical fruit that grows in the Amazon area of Brazil and has a high economic potential due to its exotic flavour/aroma and good preservation properties. It has an hard husk that contains a soft pulp and seeds that can be separated from the pulp either manually, using scissors, or mechanically. The husk constitutes approximately 40%, seeds 15% and pulp 45% of the fruit. The pulp can be processed to produce nectar, ice cream, yoghurt flavour, chocolate filling and other products. This fruit pulp has an average pH of 3.4, a total acidity of 2.1% (citric acid), approximately 3% of reducing sugars and 5% of non-reducing sugars, and 10.7 °Brix (Venturieri, 1993).

On account of cupuaçu's low pH, a pasteurization process was recommended for its stabilization at ambient temperature (Silva & Silva, 1997). Although pasteurization stabilizes the product through the inactivation of micro-organisms and spoilage enzymes (Gaze & Betts, 1992), it can affect the product final quality. Colour changes may occur and some flavour and aroma compounds can be lost. Colour is often associated with the freshness of the fruit and is one of the most important quality attributes because it is perceived immediately by the consumer. This physical parameter can be used to inspect raw materials or final products and control the quality impact of a process. The colour can be correlated with other quality attributes such as sensory, nutritional and visual or non-visual defects, and help to control them indirectly (Kramer, 1976; Francis, 1995). Colour is also an indicator of heat treatment severity and can be used to predict the corresponding quality deterioration resulting from heat exposure (Shin & Bhowmik, 1995; Lozano & Ibarz, 1997).

Colour may be observed as the light transmitted through a solution containing substances extracted from the food in a transparent medium or as the light reflected from a surface of an opaque food. The conventional approach for

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measuring the reflectance of samples such as puree, which transmits some light, is to use a cell filled with sufficient sample so that a greater depth of sample does not change the reading (Francis, 1987). Colour can be assessed by a sensory panel or using an analytical instrument. Tristimulus colorimetry and spectrophotometry (Francis, 1987) are examples of analytical techniques that try to duplicate the response of the human eye, being more accurate than a sensory panel. The correlation between instrument and sensory panel evaluation of colour has been studied for other fruits and is very high. For instance, in cupuaçu colour-related fruits such as lemon juice and banana pulp, correlation coefficients of 0.98 (Anthoney *et al.*, 1984) and 0.97 (Wainwright & Hughes, 1989), respectively, have been observed. In addition, colour measurement with analytical instruments is quick, simple and cheap, in contrast to sensory tests.

Any colour is uniquely specified by a set of three imaginary red (X), green (Y) and blue (Z) primaries. These are the International Commission on Illumination (CIE) tristimulus values. To make colour data more intuitive and easier to interpret, these tristimulus values are usually converted to other colour scales, all of which are mathematical transforms of the tristimulus X, Y and Z. In 1952 Hunter developed the Lab colour system, while in 1976 the CIE established L*C*h°* and L*a*b* colour coordinates. The star symbol is used to distinguish between the two colour systems (Hutchings, 1994; Minolta, 1994). Colour lightness (value), L* (0: black to 100: white), measures how light/dark is the colour of the object; chroma or saturation, C* (0 – 60), measures how dull/vivid is the object colour; hue angle, h°* (0 – 360 °), expresses the characteristic/dominant colour; and a* and b* (– 60 – 60) measures the degree of greenness/redness and blueness/yellowness, respectively. Total colour difference (TCD*) is a parameter that quantifies the overall colour difference of a given sample (L*a*b*) when compared to a reference sample (L_o*a_o*b_o*) (Minolta, 1994):

$$TCD^* = \sqrt{(L^*-L_0^*)^2+(a^*-a_0)^2+(b^*-b_0^*)^2} \quad (1)$$

As pointed out previously, it is important to provide certain guidelines for colour grading in fruit products based on a somewhat analytical standpoint, such as tristimulus colour values, rather than subjective assessment (Lee, 1997). In the field of inks, differences in colour are classified as shown in Table 1 (Drlange, 1994). Based on this classification scale, critical times in terms of colour degradation can be established.

Table 1 Classification of colour differences (Drlange, 1994)

TCD*	Colour difference classification
0.0–0.2	not perceptible
0.2–0.5	very small
0.5–1.5	small
1.5–3.0	distinct
3.0–6.0	very distinct
6.0–12.0	great (GD)
>12.0	very great (VGD)

Colour can be used to design adequate thermal processing conditions for maximizing final product quality if its change kinetics are determined.

The main goal of this work was to study the effect of different pasteurization treatments on the final colour of cupuaçu puree. The specific objectives of this research were: (a) to present a review on colour change kinetics in fruits/vegetables, (b) for each temperature, to establish an approximate critical time after which the cupuaçu colour change is perceived by the consumer, (c) to model the kinetics of cupuaçu colour change in terms of TCD* and L*/L_o*.

Theoretical considerations and colour kinetics review

The deterioration of most food quality factors, including colour, follow zero (eqn 2) or first order (eqn 3) reaction kinetics (Villota & Hawkes, 1992). First order reversible reaction kinetics (eqn 4) have also been observed for colour change.

$$\frac{P}{P_o} = -kt \quad (2)$$

$$\frac{P}{P_o} = e^{-kt} \text{ or } \frac{P}{P_o} = 10^{-\frac{t}{D}} \quad (3)$$

$$\frac{P - P_\infty}{P_o - P_\infty} = e^{-kt} \quad (4)$$

where: P is the colour parameter, P_0 and P_∞ are the values of the colour parameter at time zero and infinite time, respectively, k the reaction rate, t is the time and D is the decimal reduction time.

In these kinetics, the temperature effect on the reaction rate constant, k, is described by the Arrhenius equation (eqn 5). The Bigellow model (eqn 6) can also be used for first order reaction kinetics (Saguy & Karel, 1980; Wells & Singh, 1988).

$$k = A \times l^{\left(-\frac{Ea}{R(T+273.15)}\right)} \tag{5}$$

$$D = D_{ref} \times 10^{\left(\frac{T_{ref}-T}{z}\right)} \tag{6}$$

where: A is the pre-exponential factor, E_a the activation energy, D_{ref} the decimal reduction time at reference temperature and z the number of degrees Celsius required to reduce D by a factor of 10.

Food colour changes in terms of L and TCD follow zero order (eqn 2) and first order (eqn 3) reaction kinetics, respectively (Rhim *et al.*, 1989; Barreiro *et al.*, 1997). Ávila & Silva (1996) considered zero order reaction kinetics for L, and Lozano & Ibarz (1997) assumed first order reversible (eqn 4) reaction kinetics for the TCD parameter. Tables 2 and 3 summarize a literature search on thermal change kinetics modelling of fruit and vegetable tristimulus colour coordinates or parameters resulting from their combination.

Materials and methods

Samples

Cupuaçu fruit (*T. grandiflorum*), 'Redondo variety', was bought at a local market in Belém, Brazil. The pulp was manually extracted, frozen and stored at −20 °C. The frozen pulp was air-shipped, using dry ice, to Portugal. To defrost before the experiment, the pulp was placed overnight at ambient temperature. Just prior to the experiment, the pulp was blended into a puree.

Table 2 Thermal kinetics of colour change in fruits

Reference	Product	Temp. range (°C)	Time range (min)	Colour parameter	Reaction* order	$D_{85\,°C}$ (min)	z (°C)	$k_{85\,°C}$ (min^{-1})	A (min^{-1})	E_a (kJ/mol)
Rhim *et al.* (1989)	grape juice	60–95	—	L	1	1609	23.6	1.43×10^{-3}	$7.8 \times 10^{+13}$	114.8
				a	1	717	20.5	3.21×10^{-3}	$5.4 \times 10^{+16}$	131.8
				TCD	0	—	—	8.34×10^{-2}	$2.9 \times 10^{+12}$	92.8
				C	1	949	22.3	2.43×10^{-3}	$1.2 \times 10^{+15}$	121.2
Ávila & Silva (1996)	peach puree	115–135	—	L	0	—	—	3.58×10^{-3}	$1.0 \times 10^{+13}$	106.0
				b	0	—	—	2.62×10^{-3}	$1.9 \times 10^{+13}$	108.8
				1/a	1^w	—	—	2.27×10^{-3}	$1.6 \times 10^{+13}$	108.7
				1/TCD	1^w	—	—	1.84×10^{-3}	$6.8 \times 10^{+13}$	113.6
Lozano & Ibarz (1997)	apple pulp	56–94	15–700	L	1	3988	52.2	5.77×10^{-4}	$2.4 \times 10^{+6}$	66.4
				TCD	1^w	—	—	2.28×10^{-5}	0.33	28.6
	peach pulp	56–94	15–700	L	1	3608	69.9	6.38×10^{-4}	$2.2 \times 10^{+3}$	45.1
				TCD	1^w	—	—	3.11×10^{-5}	$1.9 \times 10^{+1}$	39.7
	plum pulp	56–94	15–700	L	1	4862	58.6	4.74×10^{-4}	$3.0 \times 10^{+6}$	67.8
				TCD	1^w	—	—	3.26×10^{-6}	0.58	36.0
Barreiro *et al.* (1997)	tomato paste	70–100	5–90	L (phase I)	1	300	53.1	7.67×10^{-3}	$8.1 \times 10^{+4}$	48.2
				L (phase II)	1	2027	106.6	1.14×10^{-3}	3.6	24.0
				a	1	245	62.4	9.41×10^{-3}	$9.0 \times 10^{+3}$	41.0
				b	1	1735	29.8	1.33×10^{-3}	$4.4 \times 10^{+9}$	85.8
				a/b	1	196	89.3	1.17×10^{-2}	$1.8 \times 10^{+2}$	28.7
				TCD	0	—	—	0.24	$4.0 \times 10^{+5}$	42.7
				C	1	316	60.5	7.29×10^{-3}	$1.1 \times 10^{+4}$	42.3

*1^w reversible first order.

Table 3 Thermal kinetics of colour change in vegetables

Reference	Product	Temp. range (°C)	Time range (min)	Colour parameter	Reaction* order	$D_{121\,°C}$ (min)	z (°C)	$k_{121\,°C}$ (min^{-1})	A (min^{-1})	E_a (kJ/mol)
Hayakawa & Timbers (1977)	asparagus	80–150	0.5–120	−a/b	1	17	41.7	0.14	$3.7 \times 10^{+7}$	63.6
	green beans	80–150	0.5–120	−a/b	1	21	38.9	0.11	$1.1 \times 10^{+10}$	82.9
	green peas	80–150	0.5–120	−a/b	1	25	39.4	9.2×10^{2}	$9.9 \times 10^{+8}$	75.7
Rao *et al.* (1981)	peas	99–127	3–25	a/b	1	13	38.3	0.17	$8.9 \times 10^{+8}$	73.3
Shin & Bhowmik (1995)	pea puree	110–125	5–20	−a	1	30	45.1	7.6×10^{-2}	$1.4 \times 10^{+8}$	69.5
				−a/b	1	31	40.9	7.3×10^{-2}	$1.5 \times 10^{+8}$	70.1
				−(La)/b	1	31	42.9	7.4×10^{-2}	$6.8 \times 10^{+7}$	67.5
Steet & Tong (1996)	green pea puree	70–90	0–1440	−a	1^{w}	—	—	0.14	$1.7 \times 10^{+9}$	76.2

*1^{w} reversible first order.

Colour measurement

Quantitative evaluation of colour changes in cupuaçu puree samples was done using a portable tristimulus colorimeter (Minolta Chroma Meter CR 300, Osaka, Japan). The colorimeter has a beam diameter of 8 mm, three response detectors set at 0 ° viewing angle and a CIE standard illuminant C with diffuse illumination. This illuminant is accepted as having a spectral radiant power distribution closest to reflected diffuse daylight. A white calibration plate was used for calibration (L* = 98.15, C* = 1.92, h°* = 93.8, a* = −0.13, b* = 1.92). Each sample was placed in a clear glass Petri dish, and L*C*h°* and L*a*b* CIE colour measurements were taken in duplicate. Total colour difference (TCD*) was calculated using eqn 1. The reference sample (L_o*a_o*b_o*) used for the calculations was the non-heat-treated sample on the day of the experiment.

Kinetic experiments

Isothermal experiments were performed at 80 °C, 90 °C (thermostatic water bath), 100 °C, 110 °C and 115 °C (thermostatic oil bath) for up to 120 min. The kinetic experiments were repeated at least twice for each temperature. This range of experimental temperatures was chosen, knowing that a pasteurization process is recommended for this product stabilization. Heating times up to 80 min (115 °C) to 120 min (80 °C, 90 °C) were chosen. A pasteurization value of 5 min at 95 °C is normally enough to stabilize the product. Although the heating times might be beyond those required for commercial processing, they were necessary so as to reach significant colour changes in order to obtain a more correct model for the colour change kinetics. Pyrex screw-capped test tubes of 10 cm height, and 0.86 cm and 1.2 cm internal and external diameter, respectively, were filled with cupuaçu puree almost to the top and closed. Care was taken to avoid incorporation of air in the cupuaçu puree samples. These Pyrex tubes were introduced in the thermostatic baths and taken out at pre-specified time intervals. The tubes were cooled immediately in an ice-water bath before colour analysis. The come-up time was determined at each temperature by inserting a thermocouple at the geometric centre of a tube containing puree, and monitoring the time required to heat up until the thermostatic bath temperature. An average come-up time of approximately 2 min was observed. The time intervals, with the exception of time = 0 min, for all the experimental colour data points were corrected by subtracting 60% of the come up time (= 1.2 min) (Ball & Olson, 1957).

Kinetics modelling

The experimental colour–time–temperature data were modelled mathematically using the statistical program STATA version 3.0 (Computing Resource Center, 1992). Several models were tested for each isothermal experiment, and the effect of temperature was investigated. Finally, a one-step non-linear regression was performed to all the data to estimate directly the kinetic parameters of the model (Lund, 1983; Arabshahi & Lund, 1985). This procedure avoided the calculation of unnecessary parameters, increased the number of degrees of freedom and decreased the confidence intervals estimated for the model parameters.

Results and discussion

The original, non-heated cupuaçu (the reference sample) exhibited a light yellow colour, corresponding to the following average values (± standard deviation) of the colour coordinates: $L_o^* = 70.04 \pm 1.57$, $C_o^* = 20.73 \pm 1.22$, $h_o^{o*} = 107.85 \pm 1.16$, $a_o^* = -6.36 \pm 0.56$ and $b_o^* = 19.73 \pm 1.16$. Visual darkening was observed in thermally treated cupuaçu puree. The darkening (browning) of this fruit after the thermal process was probably due to Maillard reactions (Fontana *et al.*, 1993). Enzymatic browning (polyphenoloxidase-PPO) did not occur. In fact, from previous experiments with cupuaçu, either this enzyme was not detected or very low amounts were found. Furthermore, Reed (1966) concluded that a mild heat treatment (e.g. 20 s at 78 °C) was effective for PPO inactivation in several fruit purees such as apple, pear, grape and apricot. As soon as the cupuaçu samples were exposed to high temperatures, during the first minute TCD* increased significantly. This is consistent with the loss of brightness and freshness that occurs whenever a food is cooked. After this period, TCD* changed linearly with time. The linear trend for this parameter was also observed by other authors (Rhim *et al.*, 1989; Barreiro *et al.*, 1997) (Table 2). With increasing time and/or temperature exposure, cupuaçu puree darkened (i.e. L* decreased), became more vivid in colour (i.e. C* slightly increased), and changed from a light yellow to a caramel-like colour (h°* decreased). Parameters a* and b* increased slightly. The parameters that changed most with thermal processing were L* (70.0–56.7) and h°* (107.8–84.3) and therefore, in terms of the individual colour coordinates, the L*C*h °* colour system was more appropriate. The other colour system (L*a*b*) was only useful for the calculation of the TCD* parameter, L* being the major contributor for colour change. In the most severe applied heat treatment (120 min at 110 °C), L*a*b* contributions for total change in colour were 55%, 32% and 13%, respectively. In a milder treatment (119 min at 80 °C) those contributions were 70%, 11% and 19%, respectively.

Colour critical times

Using the difference classification scale for TCD* (Table 1) it can be concluded that great differences (GD) and very great differences (VGD) in the colour of the processed cupuaçu puree were obtained (Fig. 1). For each temperature, these differences occurred after certain critical times. Very great differences (VGD) were observed at 110 °C and 115 °C after 77 min and 53 min, respectively. At 80 °C, 90 °C and 100 °C, processing times larger than 120 minutes would be required for VGD in colour. Great differences

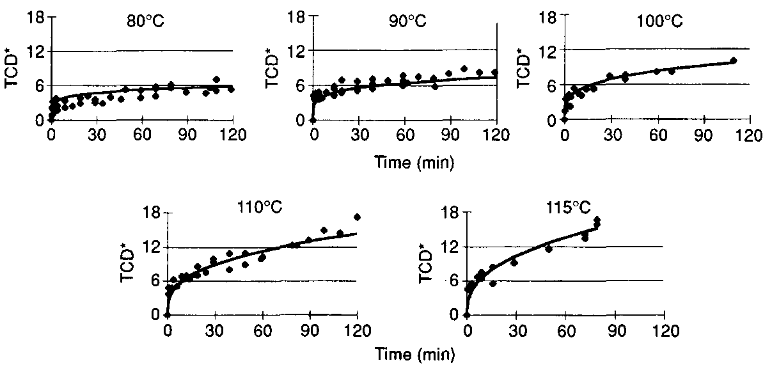

Figure 1 Processing temperature and time effects on total colour difference (TCD*): experimental (◆), predicted (—).

(GD) in colour were obtained for all the temperatures tested. Temperatures of 80 °C, 90 °C and 100 °C required approximately 90 min, 40 min and 20 min, respectively, to exhibit great differences. However, at 110 °C and 115 °C only a few minutes were enough to cause a great difference in colour.

Kinetics modelling

Several colour parameters or their combination were analysed. The TCD* and L^*/L_o^* were modelled because these were the colour factors that changed most with the thermal treatment. L_o^* is the L* of the non-heated original cupuaçu sample. For each temperature, TCD* increased and L^*/L_o^* decreased with processing time, following a power law equation:

$$TCD^* = c \times t^d \quad (7)$$

$$L^*/L_o^* = 1 - c \times t^d \quad (8)$$

The power parameter, d, was temperature-dependent and was expressed by the Arrhenius law:

$$d_T = d_{100°C} \times 1^{-\frac{Ea}{R}\left(\frac{1}{T+273.15} - \frac{1}{373.15}\right)} \quad (9)$$

where: d_T is the power parameter at a given temperature, $d_{100\,°C}$ is the power parameter at 100 °C, E_a the activation energy (J/mol), R the universal gas constant and T the temperature (°C).

For TCD*, the model's constants with its 95% confidence intervals (R^2 = 0.98) were c = 2.85 ± 0.25, $d_{100\,°C}$ = 0.26 ± 0.02 and E_a = 31.0 ± 4.0 kJ/mol. Figure 1 shows the model adjustment to the experimental data for all temperatures. In the first minutes a sudden rise in the TCD* was observed for all the temperatures, but after a few minutes a linear relationship was observed. The model's constants estimated for the L^*/L_o^* parameter (R^2 = 0.99) and its 95% confidence intervals were c = 0.044 ± 0.004, $d_{100\,°C}$ = 0.20 ± 0.02, E_a = 36.0 ± 6.1 kJ/mol (Fig. 2). Although in previous literature the colour parameters were modelled well with first and zero order kinetics (Tables 2 and 3), for the time–temperature ranges investigated in this study a power law equation modelled the colour degradation in cupuaçu puree much more effectively.

Nomenclature

a	Degree of greenness/redness colour
A	Pre-exponential factor (min^{-1})
b	Degree of blueness/yellowness colour
c	Power law model constant
C	Colour chroma or saturation
d	Power parameter
D	Decimal reduction time: time (min) to reduce by a factor 10, at a constant temperature
E_a	Activation energy (J/mol)
GD	Great differences in colour
h°	Colour hue angle (°)
k	Reaction rate (min^{-1})
L	Colour lightness
P	Colour parameter
R	Universal gas constant (8.314 J mol^{-1} K^{-1})
t	Time (min)
T	Temperature (°C)
TCD	Total colour difference
VGD	Very great differences in colour
z z-value	number of degrees Celsius required to reduce the *d*-value by a factor of 10

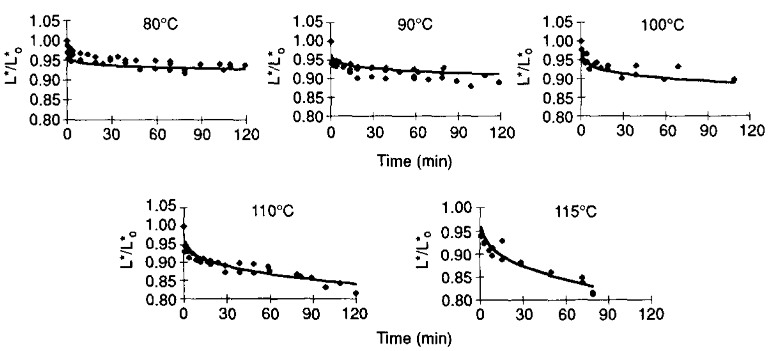

Figure 2 Processing temperature and time effects on L^*/L_o^*: experimental (◆), predicted (—).

Subscripts
o Refers to initial time, time zero, non-heated cupuaçu sample
ref Reference temperature
∞ Refers to infinite time or time to reach the equilibrium colour value

Superscript
* Refers to CIE colour space coordinates

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