

# Kinetics of colour change of raspberry, sweet (*Prunus avium*) and sour (*Prunus cerasus*) cherries preserves packed in glass containers: light and room temperature effects

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## Abstract

This work studied the kinetics of colour change in preserves of raspberries, sweet and sour cherries exposed to different lighting conditions (light and darkness), at constant temperature, as well as stored at several temperatures. In all the preserves and treatments, the colour diminished noticeably during the first 90 days of storage to remain constant from then on. The kinetics of colour change was evaluated using the concept of fractional conversion, and a first-order kinetics was found in relation to both the effect of lighting conditions and temperature. The corresponding reaction rate constants were determined by regression analysis of the experimental data. For samples stored at different lighting conditions, the resulting constants of the different preserves were not alike: constants were higher in samples kept in the presence of light than the corresponding samples stored in the dark. However, the statistical analysis of the results indicated those not significant differences among the different light treatments. The statistical analysis of reaction rate constants obtained for the samples stored at constant temperatures, of 4°C, 20°C and 40°C, did indicate significant differences between treatments. In this case, applying one (A. Arabshai, D.B. Lund, Journal of Food Process Engineering 7 (1985) 239) and two-step (Arrhenius model) methods, the corresponding values of the pre-exponential factor ( $\ln k_0$ ) and the energy of activation ( $E_a$ ) were obtained and compared for the three preserves analysed. © 2001 Elsevier Science Ltd. All rights reserved.

**Keywords:** Raspberries; Cherries; Colour degradation; Kinetics; Modelling

## 1. Introduction

The production of small fruit preserves, such as sweet and sour cherries and raspberries, bears great economic importance in the north-west area of Chubut and south-west of Río Negro, Argentina, as they constitute exclusive products with strong demand not only in the retail market but also in the institutional one. Preserves are elaborated by packing fresh fruit together with low-concentration sucrose syrup in transparent glass containers. Owing to the characteristics of the container and to manufacturers' unwillingness about using additives of any type, the colour of the preserves becomes an im-

portant parameter to test product visual quality. Previous literature on the subject indicates that the colour of these fruits is caused by anthocyanin pigments (Rommel, Heatherbell, & Wrolstad, 1990; Daravingas & Cain, 1965, 1968; Markakis, Livingston, & Fellers, 1957; Ponting, Sanshuck, & Brekke, 1960; Spanos & Wrolstad, 1987; Kessler, Ochoa, & Vullioud, 1997; Von Elbe & Schaller, 1968; Fischer & Von Elbe, 1970; Forni, Polesello, & Torregiani, 1993).

These pigments are degraded by heat (Kessler et al., 1997; Rommel et al., 1990; Daravingas & Cain, 1965, 1968) and by the oxygen present in the air of the head space of the container (Markakis et al., 1957). According to Daravingas and Cain (1965), degradation is also intensified by the presence of ascorbic acid and sugar concentration. In all cases, the acidity of the medium and of course the exposure time are of great importance. No mention has been found about the effect of light on the natural pigments of small fruit preserves.

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Notation			
$C$	concentration parameter of colour (%)	$k$	reaction rate constant (days <sup>-1</sup> )
$C_{(t_j, T_i)}$	concentration of $C$ at time $t_j$ and temperature $T_i$	$k_{(T_i)}$	reaction rate constant for temperature $T_i$
$C_0$	initial concentration parameter of colour (%)	$k_0$	reaction rate constant at infinite temperature or pre-exponential factor (days <sup>-1</sup> )
$C_{0(T_i)}$	initial concentration at time zero for temperature $T_i$	$N$	number of set of data concentrations versus time
$C_{eq}$	equilibrium concentration parameter of colour (%)	$R$	universal gas constant (kJ/mol K)
$E_a$	energy of activation (kJ/mol)	$T$	absolute temperature (K)

In the fruit preserves analysed here, the sugar concentration, oxygen and ascorbic acid contents, and the acidity and thermal treatment are all parameters fixed by the raw materials and the preparation method. So, it becomes important to evaluate the action of light and temperature on pigment stability as a function of storage time. Then, it will be possible to recommend actions to minimise visual quality losses.

## 2. Materials and methods

### 2.1. Plant material

Freshly harvested fruit coming from the same plant were utilised here. The varieties used were: raspberry (Schöeneman), sour cherry (Montmorency), and sweet cherry (Napolitana).

**Preparation of samples:** A mass of 200 g of fresh fruit, having the same surface colour quality, was packed in 360 cm<sup>3</sup> cylindrical glass containers (diameter: 0.073 m, height: 0.012 m). A 200 cm<sup>3</sup> of 25° Brix solution of sucrose in water was added, as covering liquid to complete the volume. The container was closed using a metallic lid to carry out the pasteurisation process in a water bath at 90°C for 20 min, the final headspace was 2 mm. The container was quickly cooled down to room temperature. Turning the container upside down for 48 h tested hermeticity of the lids.

**Storage of samples:** As soon as the preserves were prepared, they were stored for 10 months under light and dark conditions at the same temperature, to evaluate the influence of light. Illuminated samples were exposed, all the time at 60 W fluorescent light in a 2.00 × 2.00 × 2.40 m<sup>3</sup> white room at 20 ± 2°C, on the 0.85 m height table. In the same room dark samples were stored in a closed closet. Concerning the temperature influence on the concentration of natural pigments, this was tested by storing samples in temperature controlled ovens at constant temperatures values of 4 ± 0.3°C, 20 ± 0.3°C and 40 ± 0.3°C for five months.

**Determinations:** The initial samples, as well as those taken then periodically along storage, were analysed for the following determinations:

**Colour of covering liquid:** It was determined by using a spectrophotometer at 527 nm. To this end, the liquid

was diluted with distilled water as necessary. Colour was expressed in absorbance units per gram of (covering) liquid.

**Colour of fruit:** Pigments were extracted by using a mixture of methanol and hydrochloric acid (1%), and the absorbance was measured by spectrophotometry at 527 nm. Colour was expressed in absorbance units per gram of fruit.

**Analysis of the results:** These were examined by analysis of variance (ANOVA) and the mean values were compared by the LSD test with  $\alpha = 0.05$ .

## 3. Results and discussion

### 3.1. Effect of lighting conditions

Figs. 1–3 show the absorbance at 527 nm per gram for preserves of sweet and sour cherries and raspberry both for fruit and covering liquid as a function of the lighting conditions. Fig. 1 indicates that the colour of sweet cherries decreases sharply during the first 10 days. Then it decreases more slowly up to the 90th day to finally stabilise from then on ( $P < 0.05$ ). In sour cherries (Fig. 2), a sudden absorbance drop is also observed, but in the first two days. Then, the decrease becomes slower up to the 90th day to keep then stable ( $P < 0.05$ ). In raspberry preserve (Fig. 3), the sharp absorbance decrease takes place over the first day. As in the preserves mentioned before, the decrease occurs then more slowly until 80–90 days of storage, to stabilise for longer times. The behaviours of samples stored in the dark were alike those of samples kept in the presence of light ( $P < 0.05$ ).

In the different covering liquids, a maximum colour was recorded during the first day in raspberries (Fig. 3, enlarged zone in inset graph), at two days in sour cherries (Fig. 2, enlarged zone in inset graph) and at 10 days in sweet cherries (Fig. 1, enlarged zone in inset graph). This colour increase of the covering liquid coincides in time with the sharp decrease observed in the absorbance measurements of the fruit. After reaching the maximum, the absorbance decreases until about the 90th day, remaining then constant ( $P < 0.05$ ).

The sharp absorbance decrease in fruits observed along with the absorbance increase in the covering liquid can be attributed to pigment transfer from the former to

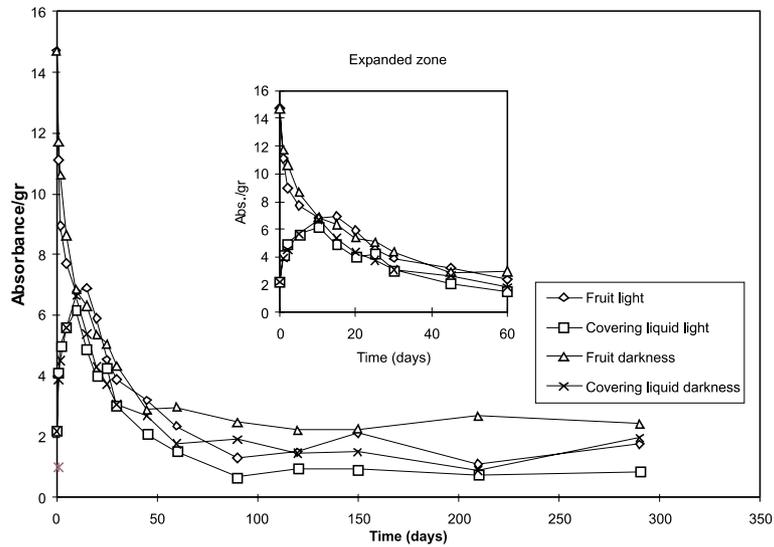


Fig. 1. Sweet cherry preserve: colour concentration (absorbance units/gram) in fruit and covering liquid, with and without lighting.

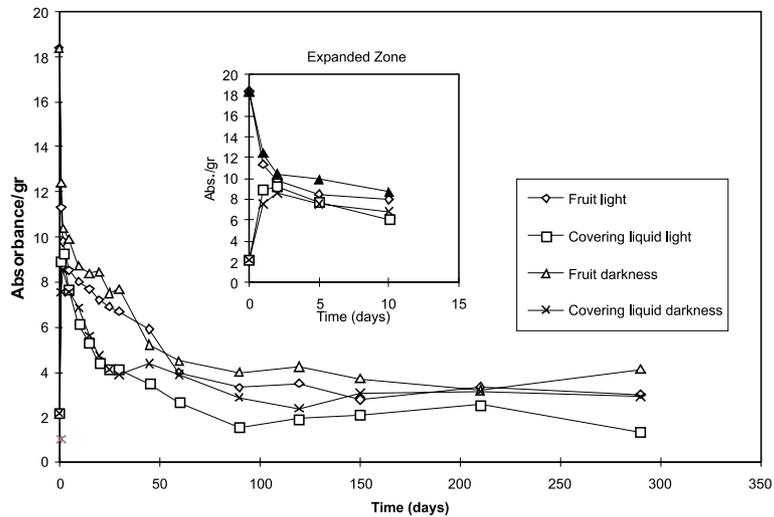


Fig. 2. Sour cherry preserve: colour concentration (absorbance units/gram) in fruit and covering liquid, with and without lighting.

the latter, which then comes to equilibrium. The times required to reach equilibrium are specific of each fruit. In raspberry, the transfer is fast because the drupes are damaged during the harvest since they are separated from the calyx, which remains in the plant (DeMichelis, Riadigos, & Martinez, 1993). In the case of sour cherries, a relatively fast equilibrium can be expected because of a high percentage of peel rupture of fruits that takes place during the pasteurisation thermal treatment (Márquez & De Michelis, 1997). On the contrary, the stabilisation time is longer in sweet cherries because fruits do not present any physical damage during harvest and/or preserve preparation.

As a result of the presence of absorbance maximum values in the covering liquid, and of the sharp drop of it in the fruit, it is difficult to analyse which kinetic func-

tion represents the resulting curves. Hence, a measure of the concentration of pigments was obtained by adding the absorbances of covering liquid and fruit (Daravingas & Cain, 1965) for each preserve studied. In the same way and a consequence of the different initial pigments concentration in the different fruits (see Table 1), and to compare the behaviour of each preserve, Fig. 4 represents the total colour retention with respect to the initial content (considered as 100%) as a function of time along storage, both for preserves kept in the light and in the dark.

In Fig. 4 as in Figs. 1–3, it is observed that the concentration of pigments decreases with time until about 90 storage days to tend then to remain constant.

The conventional equation for degradation kinetics data is

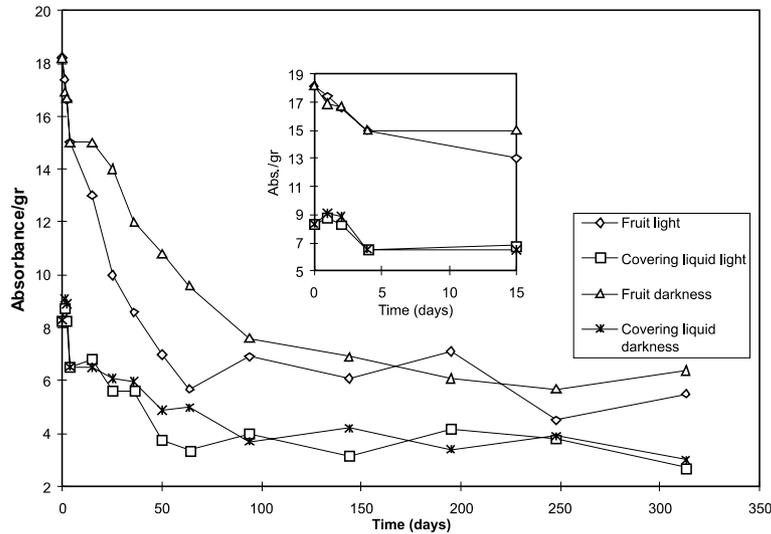


Fig. 3. Raspberry preserve: colour concentration (absorbance units/gram) in fruit and covering liquid, with and without lighting.

Table 1  
Characteristics of the fruits used and pH of the preserve of each fruit

Fruits	pH of fruits	Refractometric soluble solids (°Brix)	Acidity (mg of citric acid/100 g)	Ascorbic acid (mg/100 g)	Anthocyanins (mg cyanidin-3-glucosid/100 g)	pH of preserves
Raspberry	2.98	11.5	1.54	17.4	27.3	3.15
Sour cherry <sup>a</sup>	3.50	19.6	1.63	2.24	25.2	3.42
Sweet cherry <sup>a</sup>	4.19	25.8	0.48	1.35	3.67	4.27

<sup>a</sup> Expressed on fruit without stones. Sour cherry: 7.8% of stones; sweet cherry: 4.9% of stones.

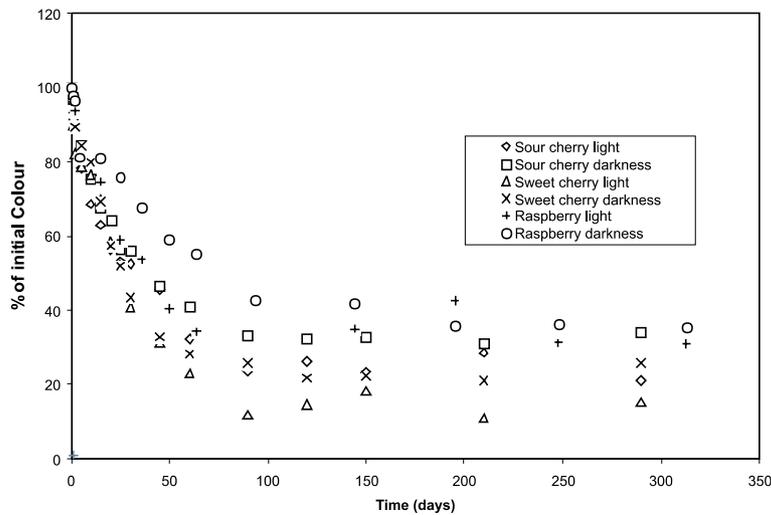


Fig. 4. Total pigment content (fruit plus covering liquid) as percentage of the initial value.

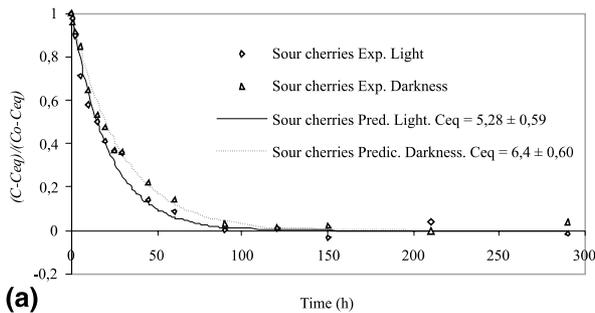
$$\frac{C}{C_0} = \exp(-kt), \tag{1}$$

where  $C_0$  is the initial parameter colour concentration,  $k$  the temperature dependent reaction rate ( $\text{days}^{-1}$ ),  $t$  the

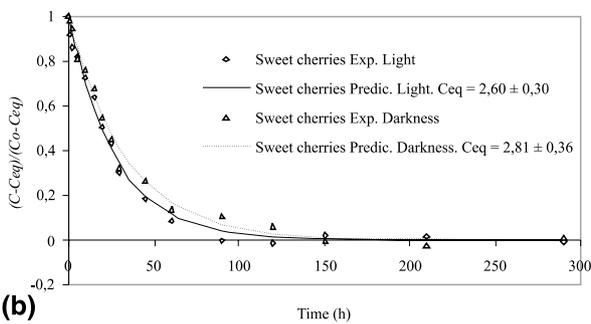
time, and  $C$  is the parameter colour concentration at any time. For a reaction following a first-order kinetic model, the plot of  $\ln(C/C_0)$  versus  $t$  would be a straight line and the slope, at a constant temperature, would equal  $-k$ . As a consequence of a final stabilisation of

parameter concentration colour, it is possible to obtain the corresponding kinetics constants applying the concept of fractional conversion for an irreversible first-order reaction (Levenspiel, 1972; Steet & Tong, 1996; Avila & Silva, 1999):

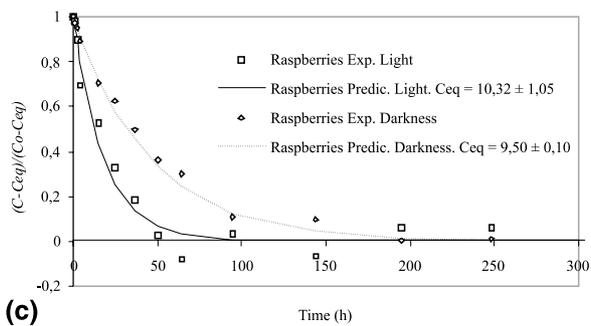
$$\frac{C - C_{eq}}{C_0 - C_{eq}} = \exp(-kt), \quad (2)$$



(a)



(b)



(c)

Fig. 5. Colour changes in: (a) sour cherries preserves; (b) sweet cherries preserves; (c) raspberries cherries preserves, light effects.

where  $C_{eq}$  is the final equilibrium value of the colour parameter concentration.

By regression of experimental data with Eq. (2)  $k$  values were obtained for studied preserves in both light and dark conditions.

In Figs. 5(a)–(c) (sour cherries, sweet cherries and raspberries preserves, respectively) the experimental values and, in full and dotted lines, predicted values obtained with Eq. (2) are presented. Experimental values are regressioned with Eq. (2) maximising the regression coefficient  $R^2$ . However, it shows experimental  $C_{eq}$  values obtained by linear regression of colour concentration parameter for times longer than 90 days.

Negative values of  $(C - C_{eq}) / (C_0 - C_{eq})$  obtained for longer times than 90 days correspond to the individual experimental values smaller than fitted  $C_{eq}$  values.

Values of  $k$  obtained and the confidence levels, together with the corresponding correlation coefficients are presented in Table 2.

The rate constants obtained for the three preserves studied were very similar. If the rate constants of the different treatments (presence and absence of light) are compared, it is observed that the  $k$  values are higher for light than they are for darkness in all the preserves tested here. However, when the statistical analysis of results is carried out no significant differences between treatments are found.

### 3.2. Effect of storage temperature

As indicated in Section 2, the samples prepared to evaluate the influence of storage temperature on pigment concentration were stored at the constant temperatures of 4°C, 20°C and 40°C.

In Fig. 6 the concentration of pigments in sweet cherry preserves is presented in units of absorbance per gram of covering liquid or fruit, as a function of time for the three storage temperatures tested. As it can be observed, the obtained curves are similar to those presented in Fig. 1, when the effects of lighting conditions were studied. A strong decrease can be observed in the absorbance of the fruit over the first 10 days of storage, and a maximum in the curves corresponding to the covering liquid at the same time. For sour cherries and

Table 2

Reaction rate constants and regression coefficients for the different preserves in conditions of light and darkness

Preserve of	Treatment	$k$ (days <sup>-1</sup> )	Regression coefficient
Sour cherries	Light	$0.0460 \pm 3.7 \times 10^{-3}$	0.986
Sour cherries	Darkness	$0.0347 \pm 2.6 \times 10^{-3}$	0.995
Sweet cherries	Light	$0.0360 \pm 3.1 \times 10^{-3}$	0.992
Sweet cherries	Darkness	$0.0300 \pm 3.6 \times 10^{-3}$	0.982
Raspberries	Light	$0.0550 \pm 3.5 \times 10^{-3}$	0.975
Raspberries	Darkness	$0.0220 \pm 1.2 \times 10^{-3}$	0.995

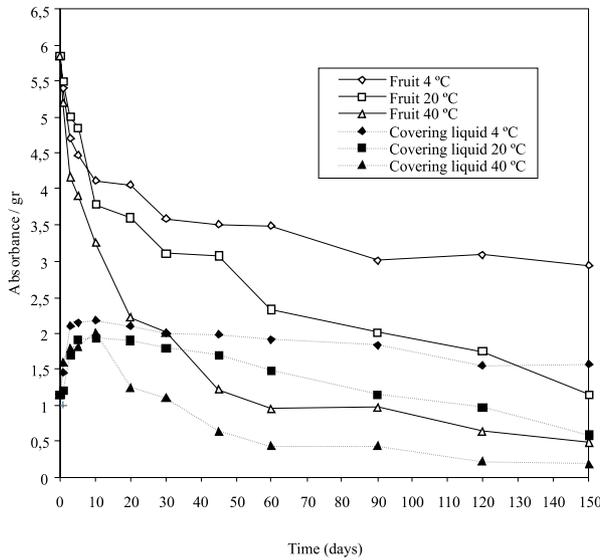


Fig. 6. Sweet cherry preserve: pigment concentration in fruit and covering liquid (absorbance units/gram) for the three storage temperatures tested.

raspberries, a similar behaviour was observed, i.e., the decrease in fruit colour and the colour maximum in the covering liquid take place at the same times as those of the study of the effect of light (Figs. 2 and 3). These effects are attributed to the same causes mentioned above.

On these grounds, and in order to evaluate the kinetics of colour degradation as a function of temperature in the preserves tested, Fig. 7 shows, in a semilogarithmic scale, the plots of the sum of the absorbances of fruit and covering liquid as a function of time for sweet cherry preserves exposed at different temperatures. As in Fig. 4, the results were presented as percentage of colour retention, taking the colour of the initial sample as 100%.

The linear trend of the results of Fig. 7 allows a first-order kinetics to be assumed on pigment degradation.

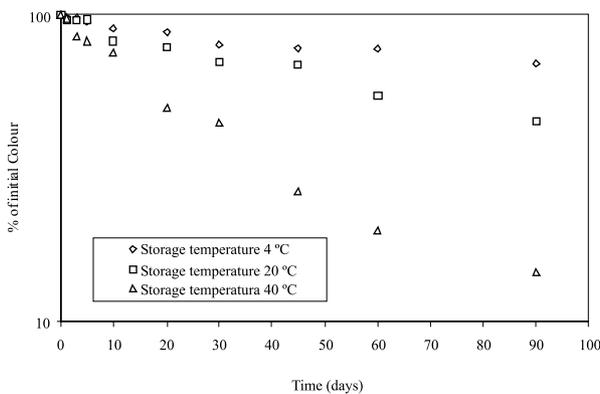


Fig. 7. Sweet cherry preserve: total pigment content as a percentage of initial value.

These trends are the same as in previous works by Kessler et al. (1997), and Barreiro, Milano, and Sandoval (1997). The reaction rate constants ( $k$ ) were calculated from the slopes of the straight lines obtained by regression of the experimental data. The values of  $k$  are presented together with the regression coefficients in Table 3.

The effect of temperature on the reaction rate constant can be evaluated by using the two-step method with Arrhenius equation, and/or applying the one-step method proposed by Arabshahi and Lund (1985).

In the two-step method, the temperature dependence of most reactions in foods can be expressed by the Arrhenius model (Lund, 1975; Kessler & Fink, 1986; Wells & Singh, 1988; Rhim, Nunes, Jones, & Swartzel, 1989; Lozano & Ibarz, 1997; Avila & Silva, 1999). Frequently applying linear regression analysis to the mean value of the rate constants,  $k$ , ignoring the variability of each  $k$  value even though this variability in  $k$  is usually calculated. The appropriate approach is to incorporate this variability in the rate constants by using weighted regression analysis (Arabshahi & Lund, 1985). For calculating the activation energy  $E_a$ , the Arrhenius model consists of two steps: first, regressing concentration on time at constant temperature to determine the rate constant, and second, regressing  $\ln k$  on reciprocal temperature to determine  $E_a$ . Generally this two-step procedure results in a relatively large standard deviation in  $E_a$  and particularly with a large confidence interval caused by the small number of degrees of freedom. To increase the degrees of freedom, thus narrowing the confidence interval, the equation presented by Arabshahi and Lund (1985) can be applied:

$$C_{(t_{ij}, T_i)} = C_{0(T_i)} \exp(-k_{(T_i)} t_{ij}), \tag{3}$$

where  $C_{(t_{ij}, T_i)}$  is the concentration of  $C$  at time  $t_{ij}$  and temperature  $T_i$ ; and  $C_{0(T_i)}$  the initial concentration at time zero for temperature  $T_i$ . Incorporating the Arrhenius equation into Eq. (3) results:

$$C_{(t_{ij}, T_i)} = C_{0(T_i)} \exp[-k_0 t_{ij} \exp(-E_a/RT_i)]. \tag{4}$$

Eq. (4) is a non-linear model in  $(N + 2)$  parameters:  $C_{0_1}, C_{0_2}, C_{0_3}, \dots, C_{0_N}, k_0, E_a$ .  $k_0$  is the rate constant at infinite temperature or pre-exponential factor;  $R$  the universal constant of gases, and  $N$  is the number of set of data concentrations versus time, that are for the same reaction at  $N$  different constant temperatures,  $T_1, T_2, T_3, \dots, T_N$ .

Applying a non-linear regression by using the Systat 5.02 software (SYSTAT, Inc, 1990–1993),  $k_0$  and  $E_a$  can be obtained by one-step method from the experimental data.

Values of  $\ln k_0$  and  $E_a$  obtained by two-step methods were presented in Table 3 for each preserve, with the corresponding correlation coefficient. In Table 4, values of  $E_a$  obtained by two- and one-step methods were

Table 3

Reaction rate constants at different temperatures, pre-exponential factor and energy of activation obtained by the two-step method for the preserves studied<sup>a</sup>

$T$ (°C)	$k$ (days <sup>-1</sup> ) $\times 10^3$					
	Sweet cherry		Sour cherry		Raspberry	
4	1.28 $\pm$ 0.11 (0.957)		1.10 $\pm$ 0.04 (0.991)		2.00 $\pm$ 0.09 (0.998)	
20	3.88 $\pm$ 0.16 (0.991)		2.49 $\pm$ 0.08 (0.998)		3.38 $\pm$ 0.11 (0.998)	
40	6.95 $\pm$ 0.50 (0.963)		5.37 $\pm$ 0.34 (0.975)		7.10 $\pm$ 0.46 (0.978)	
$\ln k_0$ (days <sup>-1</sup> )	$E_a$ (kJ/mol)			$E_a$ (kJ/mol)		
	Sweet cherry	Sour cherry	Raspberry	Sweet cherry	Sour cherry	Raspberry
	8.378 $\pm$ 0.167 (0.999)	7.280 $\pm$ 0.709 (0.998)	5.048 $\pm$ 0.675 (0.998)	34.757 $\pm$ 5.407 (0.999)	35.520 $\pm$ 4.817 (0.998)	26.099 $\pm$ 5.190 (0.998)

<sup>a</sup> Regression coefficients within parenthesis.

Table 4

Energy of activation ( $E_a$ ) and confidence interval, for the colour degradation of sweet, sour cherry and raspberry preserves, obtained by one and two-step methods

Method preserve	Two-step method		One-step method	
	$E_a$ (kJ/mol)	Confidence interval	$E_a$ (kJ/mol)	Confidence interval
Sweet cherry	34.757	$\pm$ 5.407	34.816	$\pm$ 2.559
Sour cherry	35.520	$\pm$ 4.817	32.495	$\pm$ 2.278
Raspberry	26.099	$\pm$ 5.190	26.003	$\pm$ 2.459

presented with the confidence interval. As can be seen, values of  $E_a$  obtained by the two mentioned methods are very similar, but the confidence interval of the values obtained with one-step method is smaller than the one obtained by the two-step method, confirming a conclusion of the study of Arabshahi and Lund (1985).

As can be seen in Table 3, an increase in both the pre-exponential factor and the activation energy is observed for increasing pH in the preserves. The lower parameter values are shown by raspberry preserve, of lower pH (see Table 1), whereas the highest parameters correspond to sweet cherry preserve, for which the highest pH was determined. This tendency agrees with the results reported by Daravingas and Cain (1965) and the paper of Williams and Hrazdina (1979).

The statistical analysis of the results indicates significant differences among treatments.

#### 4. Conclusions

The objective measurement of colour using a simple spectrophotometric method, evaluating the absorbance at 527 nm proved to be a good indicator of total colour change in the studied preserves, in all of the conditions tested. When the concentration of total colour decreases with time, until a certain period, and then tends to re-

main constant, the fractional conversion concept for an irreversible first-order reaction is adequate to obtain the velocity reaction constants, since this method permits use of all experimental data.

When the numbers of temperatures tested were scarce, and do not allow to obtain a good linear regression, evaluating reaction constants temperature dependence with a non-linear regression using the one-step method allows to obtain kinetic parameters with confidence intervals smaller than the traditional two-step method using the Arrhenius model.

The values of  $\ln k_0$  and  $E_a$  obtained for the preserves tested allow estimating the total colour loss for different storage temperatures.

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