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Kinetics of Color Development in Fortified Cookies

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Abstract: The objective of this work was to study kinetics of color development in protein-fortified cookies systems at three heat-treatment temperature (190, 220, 250 ºC) and three water content (23, 26, 29 %). Response surface methodology was used to analyze the effect of heat treatment and water added on reaction rate constant obtained for L*, a*, b*, Cab*, hab* and were compared with Arrhenius equation. Color parameters evolutions follow a first-order kinetic. The linear coefficients corresponding to the water added variable were no significant for all color parameter, meaning rate constants values were only heat treatment temperature dependent. The goodness of the model prediction was assessed by the mean absolute relative error (%). Results showed that both correlation method were adequate to predict kinetic coefficients in the technological conditions studied.

Keywords: Kinetic, Color, Cookies

1. Introduction

Cookies prepared from composite flours have been extensively used as protein fortification vehicles due to their long shelf-life and high acceptability [1]. The aim of protein fortification is to develop products with both enhanced protein content and quality, and good sensory acceptance. An important step in the development of composite flour products is the evaluation of the influence of composite flour ingredients on the product's nutritional, sensory and technological attributes [2].

One of the first attributes used by consumers in evaluating food quality is the visual appearance, which is significantly impacted by color. Color is also critically important in food choice and influences the perception of other sensory characteristics [3]. Maillard reaction plays a major role in the cookie manufacturing process. Color and flavors developed during the last steps of Maillard reaction contribute to the acceptability of cookies and other baked products [4,5]. Heating cookies at high temperatures promotes the development of brown color. Cookies become darker as temperature and time increase. The Maillard reaction is influenced by many factors, including reactant concentration, temperature, time, initial pH and water activity [6,7].

On the other hand, it has been established that the condensation reaction between reducing sugars and the amino side-chain of lysine during Maillard reaction leads to a severe loss of lysine availability [2]. Carboxymethyl-lysine is considered as stable advanced Maillard product with low reactivity and it is frequently selected as a marker compound in the context of severe heat treatments [8].

Quantitative measurement of browning rate (brown compounds production or color development) may be considered an indicator of severity of heat treatment or for evaluating the efficiency of technological industrial processes. The knowledge of kinetic parameters is necessary to control the extent of the reaction [9].

The aim of this study was to determine the reference cooking time by sensory evaluation and correlate it with kinetic parameters describing brown pigment development in protein-fortified cookies, operating at three different cooking temperatures and dough hydratation levels.

2. Material and Methods

2.1. Materials

Wheat flour provided by Molinos Sagemuller (Entre Rios-Argentina) with the following characteristics was used: moisture 13.6 g/100 g, protein 10.3 g/100 g (N x 5.7), fat 1.0 g/100 g and ash 0.68 g/100 g. Whey Protein Concentrate (WPC) with moisture 6.6 g/100 g, protein 30.3 g/100 g (N x 6.38) and ash 6.3 g/100 g, was from Milkaut (Santa Fe-

Argentina). Soybean flour (SF) with moisture 7.5 g/100 g, protein 45.1 g/100 g (N x 6.25) and ash 5.93 g/100 g, was from Ricedal Alimentos (Santa Fe-Argentina). Pea flour (PF) with moisture 9.6 g/100 g, protein 20.8 g/100 g (N x 6.25) and ash 2.63 g/100 g, was obtained according to Alasino et al. [10]. Sugar, fat, salt and baking powder used were of food quality.

2.2. Manufacture of Cookies

Cookies were manufactured according to the rotary molded formula proposed by Erben et al. [1]. Base formulation was: wheat flour (75 g) + WPC (3 g) + SF (19 g) + PF (3 g) = 100 g, sugar (25 g), fat (25 g), salt (0.8 g), baking powder (1 g) and water.

All solid ingredients were placed in an Oster mixer and mixed during 1 min and after that time water was added and mixed during 6 min. After mixing, the dough was rolled on a wood table with 2 mm aluminium strips at both sides and then it was allowed to rest for 1 min. The dough was cut with a dough cutter of 4 cm diameter.

Samples (eight cookies) were then heat treated at three different temperatures in a rotary oven without steam. During heat treatment, cookies were removed from the oven at different cooking times (from 2 to 9 minutes according to the temperature used) to obtain samples ranging from raw dough to overcooked product (very dark brown). At each sampling time, sensory evaluation and color measurement of cookies were performed.

2.3. Sensory Evaluation

Visual and textural evaluation of cookies to determine degree of cooking: raw/cooked/overcooked, was performed by a trained panel in a number of three.

2.4. Measurement of Color

The color of cookies was measured using a Minolta spectrophotometer (Model CM-508d/8, Minolta Co. Ltd., Osaka, Japan) with illuminant D65, 10° standard observer angle and specular component excluded. Three cookies for treatment and five measurements for each cookie were performed.

Color characteristics were presented in CIE L* a* b* system. CIE L* a* b* system defines color by parameters such as lightness, L* (0: black and 100: white), a* (coordinate of red and green color; a* < 0: greenness and a* > 0: redness), and b* (coordinate of yellow and blue color; b* < 0: blueness and b* > 0: yellowness). Chroma value: [$C_{ab}^* = (a^{*2} + b^{*2})^{0.5}$] and hue angle: [$h_{ab}^*$ = arctangent (b*/a*)], were also determinated [3,11].

2.5. Mathematical Modelling of Color Changes

The rate of color parameter changes can be described by the following general equation (Eq. (1)):

$$\pm dQ/dt = kq\,[Q]^n \quad (1)$$

Where: Q = color parameter; t = time; n = reaction order; k_q = change rate constant for the color parameter Q. The sign (+) refers to attributes with increasing values (a*, b* and C_{ab}*), and the sign (-) to attributes with decreasing values (L*and h_{ab}*).

The regression analysis is used to determine the kinetic order (n) of the quality change rate. Analysis of the coefficient of determination (R^2), the differences between observed and fitted data points (residuals), and the estimated value of the intercept would enable determination of the reaction order [12].

2.6. Experimental Design and Models Validation

The experiments were based on a three-level, two-factor factorial design, with 2 replicates in the central point (11 runs; Table 1). The two independent variables were the heat-treatment temperature (T = 190, 220, 250ºC) and the water added (W = 23, 26 and 29%).

Table 1. *Independent variables and their levels used in the experimental design.*

Independent variable	Symbol	variable levels		
Heat treatment temperature (ºC)	T	190	220	250
Water added (%)	W	23	26	29

A quadratic polynomial regression model, Eq. (2), was assumed for predicting individual k_q as a function of T and W:

$$k_q = bk_0 + bk_1T + bk_2W + bk_{11}T + bk_{22}W + bk_{12}TW \quad (2)$$

Where the bk_0, bk_i, bk_{ii}, bk_{ij} were regression coefficients and T and W the independent variables (Table 1).

Color parameters L*, a*, b*, C_{ab}* and h_{ab}* values predicted integrating equation 1 for the determined pseudoreaction order n were compared with experimental values, and the percentage error (E %) was calculated using the following formula (Eq. (3)).

$$E\,(\%) = [(Q^*_{(experimental)} - Q^*_{(predicted)}) / Q^*_{(experimental)}] \times 100 \quad (3)$$

The influence of temperature on the change rate constants (k_q) for each level of water added can be also described using the Arrhenius equation (Eq. 4) [6,12]:

$$k_q\,(T) = k_0 \exp\,[-Ea / RT] \quad (4)$$

Where: k_q (T) = change rate constant for each color parameter; k_0 = pre-exponential factor; Ea = activation energy, [J mol^{-1}]; R = universal gas constant, [8.3145 J K^{-1} mol^{-1}]; T = absolute temperature, [K]. The Ea for each color parameter at each water level is obtained by regression analysis.

2.7. Statistical Analysis

All data were analyzed using Statgraphics Plus (Manugistic, Inc., Rockville, MD, USA) and Desing Expert 7.0.0. The data were fitted to the corresponding models. Regression analyses and the corresponding analysis of variance were carried out. Differences among mean values were established using LSD Multiple Range Test. Mean values were considered significantly different when $p \leq 0.05$.

3. Results and Discussion

3.1. Sensory Evaluation

In order to determine the optimal cooking time in protein-fortified cookies baked at various temperatures and with different formulations, it is necessary to define an accurate and pertinent indicator. Generally, the temperature in the core of the biscuit is a useful indicator, but in cookies this parameter is not very accurate due to the very low thickness of the product. Therefore, other parameters must be evaluated in parallel, such as the water loss and water activity. However, these two parameters are, respectively, inaccurate and time-consuming [13]. A first assessment of the development of browning at cookies surface during baking can be done by visual inspection. It can be seen that the color intensity of samples increased with baking time, as is expected.

In this study, the browning level of commercial unfortified cookies was used as a reference. In turn, to determine the reference baking time for each formula and each baking temperature we evaluated the browning development in the whole cookie by visual and textural evaluation (Table 2).

Table 2. *Optimal cooking time determined by sensory evaluation in protein-fortified cookies.*

Parameter	Water (%)	Temperature (ºC)		
		190	220	250
	23	6.5	5.0	3.5
Heating time (min)	26	7.0	6.0	4.0
	29	8.0	6.5	4.5

As shown in Table 2, the time needed to reach those values was dependent on the temperature and water concentration employed. For the same dough water content, optimal cooking time decreased as the baking temperature increased and at the same temperature, optimal cooking time increased with the amount of water added in the dough.

3.2. Measurement of Color

Selected commercial unfortified cookies had similar general characteristics to the ones in our study.

Mean values of color parameters from three commercial cookies are presented in Table 3.

Table 3. *Color parameters (L*, a*, b*, C_{ab}*, h_{ab}*) in commercial unfortified cookies.*

Color parameter	Mean ± SD (1)
L*	71.69 ± 2.01
a*	5.20 ± 2.01
b*	25.79 ± 3.51
C_{ab}*	26.34 ± 3.83
h_{ab}*	78.91 ± 2.69

(1) Mean value ± standard deviation from three commercial unfortified cookies.

In Table 4 are presented the L*, a*, b*, C_{ab}* and h_{ab}* mean values corresponding to the protein-fortified cookies at the reference cooking time selected by the panel. Time needed to reach those values was dependent on the heat treatment temperature and dough water content employed, as we show in Table 2.

Table 4. *Color parameters (L*, a*, b*, C_{ab}*, h_{ab}*) in protein-fortified cookies at the selected reference cooking time.*

Color parameter	Water (%)	Temperature (ºC)		
		190	220	250
	23	68.4 ± 1.0 (1) abC (2)	69.4 ± 0.6 bAB	70.5 ± 1.3 aAB
L*	26	69.8 ± 0.4 abB	68.6 ± 1.2 bB	70.2 ± 0.8 aB
	29	69.3 ± 0.9 abA	68.4 ± 2.0 bC	68.5 ± 0.6 aC
	23	5.4 ± 0.8 bC	5.2 ± 0.4 aC	4.6 ± 0.4 bC
a*	26	5.2 ± 0.5 bB	6.8 ± 1.1 aB	5.6 ± 0.8 bB
	29	6.3 ± 0.7 bA	8.7 ± 1.6 aA	7.1 ± 1.1 bA
	23	28.7 ± 1.2 bC	28.2 ± 0.9 aC	28.3 ± 1.0 aC
b*	26	26.9 ± 1.0 bB	31.1 ± 1.1 aB	30.6 ± 1.2 aB
	29	29.4 ± 0.5 bA	30.4 ± 1.9 aA	31.6 ± 1.9 aA
	23	29.2 ± 1.3 bC	28.7 ± 0.9 aC	28.7 ± 1.0 aC
C_{ab}*	26	27.3 ± 1.0 bB	31.8 ± 1.3 aB	31.1 ± 1.3 aB
	29	30.1 ± 0.5 bA	31.6 ± 1.9 aA	32.4 ± 0.9 aA
	23	79.4 ± 1.1 aA	79.5 ± 0.8 bA	80.8 ± 0.5 aA
h_{ab}*	26	79.2 ± 0.8 aB	77.8 ± 1.5 bB	79.8 ± 1.0 aB
	29	77.9 ± 1.2 aC	74.1 ± 2.7 bC	± 1.9 aC

(1) Values are mean ± standard deviation.

(2) Different lowercase letters indicate a significant difference (p 0.05) for the temperature. Different capital letters indicate a significant difference (p 0.05) for the water content.

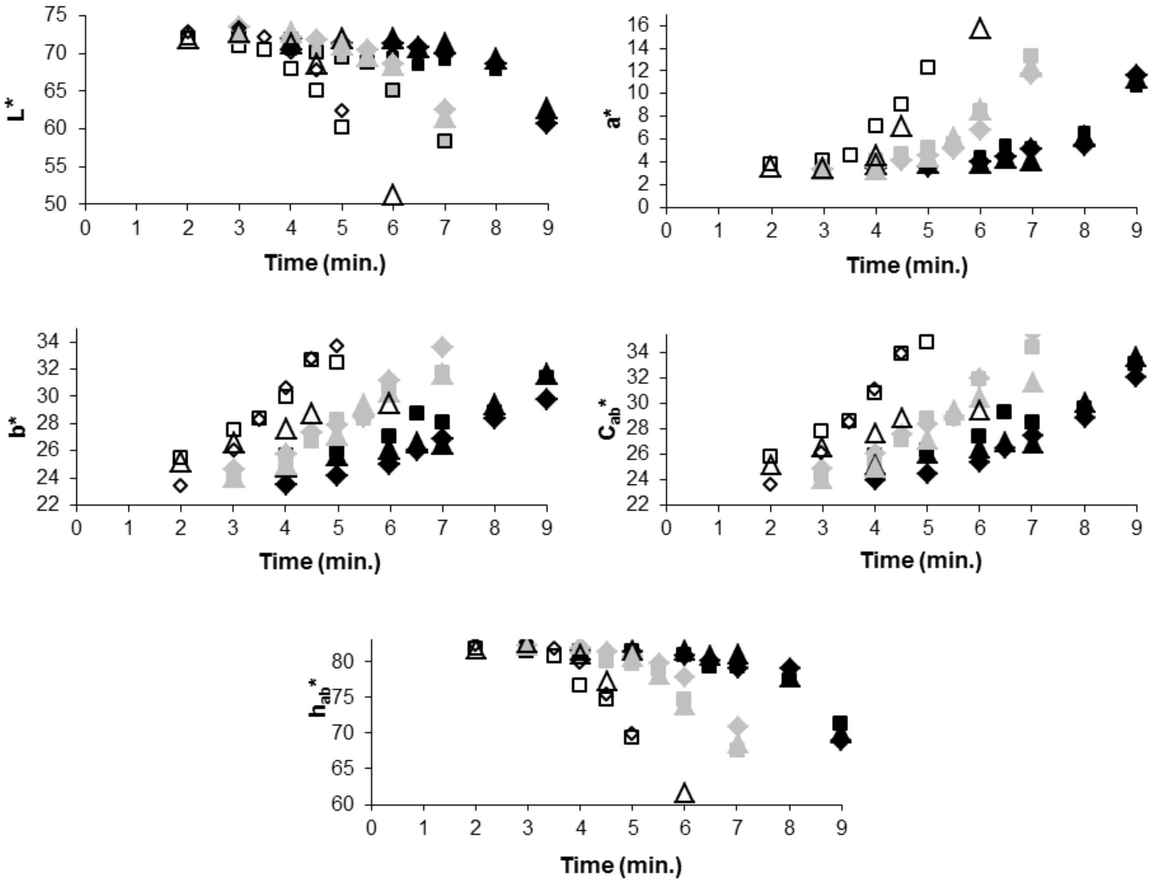
At the reference cooking time, color parameters range in protein-fortified cookies were L*: 68.4-70.5; a*: 4.6-7.1; b* 26.9-31.6; C_{ab}*: 27.3-32.4 and h_{ab}*: 74.1-80.8. As can be seen, parameters vary within a narrow range. However, statistical analysis revealed significant differences ($p < 0.05$) between color parameters of protein-fortified cookies at the selected reference cooking time, as shown in Table 4. Therefore, differences in L*, a*, b*, C_{ab}* and h_{ab}* values indicated that spectrophotometric method is necessary for characterizing these samples thanks to its higher sensitivity and objectivity compared with the sensory method used to determine optimal cooking time.

3.3. Mathematical Modelling of Color Changes

In order to select an appropriate model to represent the cookie color changes, a regression analysis was performed to determine the kinetic order. The performance of the fitted models was analysed. Based on the best coefficient of determination (R^2), first order was the apparent order of the cookie color development for all color parameters evaluated. Further analysis determined the estimated value of the intercept of the first order model. Finally, the plots of residuals versus predicted values (not shown) for each model (zero and first order) indicated that the distribution around zero was more random for the first order model. Therefore, all these results were taken into account to select the first order reaction model to describe the color development of protein-fortified cookies during heat treatment (Eq. 5).

$$Q^*_{(t)} = Q^*_{(to)} \exp(\pm k_q t) \quad (5)$$

The rate of browning of many foods followed a pseudo-zero or first order rate law as illustrated for the color development during cracker baking by Zanoni et al. [14] and Broyart et al. [15]. The kinetic model developed by Zanoni et al. [14] predicts the color difference (ΔE) for bread crust color during baking. Broyart et al. [15] developed a kinetic model using first order kinetics, and product temperature and moisture content as parameters to predict lightness variation (L*) of the cracker surface. Moreover, Mundt & Wedzicha [16] evaluated a kinetic model for browning in the baking of biscuits and postulated that the rate of formation of colored product changes according to a first order kinetic process.



■ water 23 % - 190 ºC, ♦ water 26 % -190 ºC, ▲ water 29 % - 190 ºC, ■ water 23 % - 220 ºC, ♦ water 26 % - 220 ºC, ▲ water 29 % - 220 ºC, □ water 23 % - 250 ºC, ◊ water 26 % - 250 ºC and Δ water 29 % - 250 ºC.

Figure 1. *Experimental profile of L*, a*, b*, C_{ab}* and h_{ab}* parameters in protein-fortified cookies at different conditions and baking time.*

Fig. 1 illustrates the color profiles in protein-fortified cookies. The measurements show that color parameters values vary significantly through the baking process and seem to be very sensitive indicators of cooking conditions. During thermal treatment at 190, 220 and 250 ºC, all cookies became darker, redder and more yellow with baking time. L* and h_{ab}* decreased while the others color parameters increased. In all cases, at the highest temperature, the slope obtained in the color parameters evaluated was higher so the color development was faster.

Table 5, shows the change rate constants for each color parameter (k_q Eq. 4) at each cooking temperature and water added. Results indicate that for all the color parameters the dependence of rate constants follow the Arrhenius relationship (R^2 values ranged between 0.6096 and 0. 0.9694).

Table 5. *Reaction rate (k_q) values and coefficient of determination (R^2) of color changes in protein-fortified cookies.*

Parameter	Water (%)	Temperature (°C)	Rate constant kq (min-1)	Coefficient of determination (R2)
L*	23	190	0.0253	0.6927
		220	0.0414	0.8459
		250	0.0641	0.8036
	26	190	0.0312	0.6568
		220	0.0447	0.8459
		250	0.0616	0.8036
	29	190	0.0221	0.6157
		220	0.0430	0.7548
		250	0.0777	0.7013
a*	23	190	0.1976	0.8592
		220	0.3013	0.9096
		250	0.4378	0.8985
	26	190	0.2109	0.7948
		220	0.3209	0.8929
		250	0.4654	0.8614
	29	190	0.2227	0.7227
		220	0.3124	0.9086
		250	0.4215	0.8663
b*	23	190	0.0414	0.9148
		220	0.0644	0.9657
		250	0.0953	0.9565
	26	190	0.0486	0.9767
		220	0.0811	0.9668
		250	0.1277	0.9868
	29	190	0.0411	0.874
		220	0.0531	0.9609
		250	0.0665	0.9504
C_{ab}*	23	190	0,0497	0.8996
		220	0,0776	0.9694
		250	0,1150	0.9627
	26	190	0,0575	0.9503
		220	0,0934	0.9529
		250	0,1434	0.9892
	29	190	0,0607	0.9503
		220	0,0723	0.9529
		250	0,0845	0.950
h_{ab}*	23	190	0,0249	0.7501
		220	0,0388	0.7682
		250	0,0575	0.7974
	26	190	0,0266	0.6096
		220	0,0375	0.7535
		250	0,0507	0.7109
	29	190	0,0262	0.6152
		220	0,0442	0.8154
		250	0,0701	0.7349

3.4. Experimental Design

Response surface methodology was used to analyze the effect of heat treatment temperature (T) and water added (W) on change rate constants obtained for L*, a*, b*, C_{ab}*, h_{ab}*. In all cases, the quadratic polynomial regression model was adequate for fitting reaction rate constants ($R^2 > 0.90$, no significant lack of fit), nevertheless all quadratic and interaction terms (T*W) were not significant ($p > 0.05$). Only heat treatment temperature affected change rate constants of color parameters (Fig. 1). Therefore a reduction to a linear model was done.

Eqs. (6-10) represented the mathematical models obtained for each color parameter to predict the k_q values for a given heat treatment temperature.

$$k_{L*} = - 0.10927 + 6.93333E\text{-}004 * T, (R^2{=}0{,}83) \quad (6)$$

$$k_{a*} = - 0.53386 + +3.85278E\text{-}003 * T_{,} (R^2{=} 0{,}95) \quad (7)$$

$$k_{b*} = - 012398 + 8.80000E\text{-}004 * T, (R^2{=} 0{,}64) \quad (8)$$

$$k_{Cab*} = - 0.12992 + 9.72222E\text{-}004 * T_{,} (R^2{=} 0{,}71) \quad (9)$$

$$k_{hab*} = - 0.082856 + 5.58889E\text{-}004 * T (R^2{=} 0{,}81) \quad (10)$$

Table 6. *Reaction rate (k_q) values and coefficient of determination (R^2) of color changes in protein-fortified cookies.*

Parameter	Temperature (°C)	Rate constant kq (min-1)	Coefficient of determination (R2)
	190	0.0263	0.6575
L*	220	0.0376	0.8108
	250	0.0516	0.7237
	Ea [kJ.mol-1] = 22.6		0.8907
	190	0.2144	0.8071
a*	220	0.3013	0.9164
	250	0.4182	0.8802
	Ea [kJ.mol-1] = 21.5		0.9257
	90	0.0473	0.9581
b*	20	0.0672	0.9810
	50	0.0916	0.8923
	Ea [kJ.mol-1] = 22.2		0.9017
	190	0.0563	0.9206
C_{ab}*	220	0.0793	0.9754
	250	0.1073	0.9384
	Ea [kJ.mol-1] = 21.6		0.8937
	90	0.0268	0.6593
h_{ab}*	20	0.0378	0.7901
	50	0.0512	0.7289
	Ea [kJ.mol-1] = 21.8		0.9290

Since the influence of water content on darkening kinetic parameters is not of major importance the kinetic constant were recalculated considering heat treatment temperature only (Table 6). These results also follow the Arrhenius relationship (R^2 values ranged between 0.6575 and 0. 0.9810). In all cases, reaction rates increase with temperature growth and the Arrhenius activation energy (21.5-22.6) indicates similar sensitivity with temperature for all color parameters.

3.5. Models Validation

The goodness of the model prediction was assessed by the mean absolute relative error (%). Table 7 shows L*, a*, b*, C_{ab}* and h_{ab}* parameters experimentally determined in protein-fortified cookies at the selected reference cooking time versus predicted points by fitted models and Arrhenius equation.

Table 7. *L*, a*, b*, C_{ab}* and h_{ab}* parameters experimentally determined versus predicted points and percentage error.*

Parameter	Temp (ºC)	Experimental determination	Predicted by Model (1)	Error by Model (%)	Predicted by Arrhenius (2)	Error by Arrhenius (%)
	190	69.8	71.1	1.8	68.9	-1.4
L*	220	68.6	66.7	-2.9	68.6	0.0
	250	70.2	66.2	-6.1	69.1	-1.6
	190	5.2	4.5	-16.0	5.5	5.0
a*	220	6.8	5.4	-26.5	5.5	-23.6
	250	5.6	5.0	-11.3	5.3	-6.2
	190	26.9	27.1	0.8	27.9	3.6
b*	220	31.1	28.4	-9.6	28.0	-10.9
	250	28.2	28.0	-0.6	27.6	-2.1
	190	27.6	28.3	2.6	28.6	3.6
C_{ab}*	220	28.1	29.4	4.3	28.7	2.0
	250	28.6	28.7	0.3	28.1	-1.8
	190	79.9	80.2	0.3	78.3	-2.1
h_{ab}*	220	80.3	77.3	-3.9	78.2	-2.7
	250	81.3	77.4	-5.1	78.9	-3.0

(1)Predicted parameter by Model fitted (Eq. 5-10). **(2)**Predicted parameter by Arrhenius equation (Eq. 4 and 5). Values predicted at optimal time corresponding to 26 % of water added (Table 2). Experimental values also correspond to 26 % of water (Table 4).

Mathematical models obtained when $k_{(T)}$ values of each color parameter were correlated with variables by polynomial model and Arrhenius equation showed that both correlation method were adequate to predict the values of kinetic coefficients in the technological conditions studied. Thus, changes in system behavior when technological variables are changed could be predicted. In addition, model also estimate necessary cooking time to obtain optimal color parameters (L*, a*, b*, C_{ab}*, h_{ab}). For example the predicted optimal cooking times to obtain L*= 69.5 (Table 4) are 5-18 % lower than the ones determined by sensory evaluation (Table 2).

4. Conclusions

A spectrophotometric method is necessary to characterize color changes in protein-fortified cookies to its higher sensitivity and objectivity compared with visual determination. The performance of the fitted models for all parameters evaluated (L*, a*, b*, C_{ab}*, h_{ab}*) was analysed and first order was the order of the cookie color development. Response surface methodology showed that water content variable was not significantly affected the change rate constant at the studied levels, meaning that in the condition of this work color development in protein-fortified cookies is governed by temperature. Both correlation method employed (polynomial model and Arrhenius equation) were adequate to predict the values of kinetic coefficients in the technological conditions studied. The approach applied here demonstrated the usefulness of determining optimal cooking time based on color evaluation through spectrophotometric method and of the models obtained for predicting color of protein-fortified cookies at different baking conditions and could be applied for other bakery products.

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