KINETIC MODELING, TOTAL PHENOLIC CONTENT AND COLOUR CHANGES OF MANGO PEELS DURING HOT AIR DRYING

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***Abstract*— In this work, drying of mango peels was kinetically investigated within temperature range of 40-100 ºC and the applicability of several models available in literature was statistically evaluated. Midilli's model provided the best description of the kinetics of drying mango peels with constant drying rates (*k*-and *b*-values) varying from 0.0190 to 0.0399 min^{-n} and from 0.000404 to 0.000903 min^{-1}, respectively, in temperature range of 40-100 ºC. High concentration of total phenolic content was obtained when drying temperature was increased up to 80 ºC. Finally, the polyphenolic degradation could be accompanied by CIELB parameters related to the yellowness of the dried samples.**

***Keywords*— mango peels; drying; kinetic modeling; colour; polyphenols.**

I. INTRODUCTION

Utilization of plant residues as source of bioactive compounds and antioxidant dietary fibers for food and pharmaceutical applications is an increasing trend (Larrauri *et al.*, 1996; Kim *et al*., 2010; Dorta *et al*., 2012) because it reflects on final product costs and represents a way of waste management, one of the major challenges of food industries.

Mangoes are one of the most important tropical fruits, which processing by-products represent approximately 35% of the fruit (Larrauri *et al*., 1996). The by-products from mango processing show great potential to be used as a functional compound for being source of carotenoids, phenolics, anthocyanins, minerals, chlorophylls, vitamins, fibers, fat and natural antioxidants (Kim *et al*., 2010; Dorta *et al*., 2012). Incorporation of mango residue powder in macaroni and biscuits showed interesting results due to the increase in the antioxidant power and fiber content with good acceptance of the final product (Aroga, 1999; Ajila *et al*., 2010).

In fruit waste management, drying may be a critical operation to reduce water activity, weight and volume of the residue, enhancing its stability and resulting in packaging, transport, handling and storage cost reduction (Vashith *et al*., 2011). Despite of new technologies for food drying for minimizing thermal degradation of labile compounds, like lyophilization, convective forced air drying remains as the most widely used industrial method for food drying (Vashith *et al*., 2011). Dorta *et al*. (2012) compared freeze, static and forced air drying (70 °C) of mango peels and verified that high temperatures affected anthocyanin, phenolic and antioxidant components in the dried plant material. Binomial time-temperature is the main process parameter of this technology, since it is also responsible to inactivate pathogen microorganisms and spoilage enzymes. Applied research with a view over kinetic modelling of mango peel drying and the impact of this technique on polyphenols content and colour properties in a larger range of temperature is scientific and engineered essential, but scarce.

The aim of the present work is to investigate the drying kinetics of mango peels, statistically evaluating the adequacy of experimental data to several models available in literature and to observe the effect of the drying temperature on the phenolic content and on the colour changes of the dried material. On this basis, the association of the colour changes and the thermal degradation of polyphenols was determined.

II. METHODS

A. Plant material

Mangoes (*Mangifera indica* cv. "Tommy Atkins"), purchased from local market (Porto Alegre, RS, Brazil) in 2011, were washed and manually peeled. Peels, with averaged moisture content of 83.5% (wet basis) and thickness of 1 mm, were placed in plastic containers and stored at -42 ºC in the dark. Samples were thawed overnight at 4 ºC before drying analysis.

B. Drying procedure

Drying experiments were performed in an industrial pilot dryer like described elsewhere (Cassini *et al*., 2007). The dryer equipment was composed of a centrifugal fan (1200/3700 rpm, 15 m^3 min^{-1}), three electrical resistances in parallel (180 C at 330 m^3 min^{-1}), a drying chamber with mobile sidewalls (that makes possible the inversion of the drying air stream between ascendant and descendant) and a recipient for product disposal (area of 0.04 m^2). Air flow in the drier was cross-sectional through sample tray, with mesh with opening of 0.7 mm, controlled by continuous velocity meter (Nykon Dwyler, Michigan, USA).

Before the beginning of the experiments, equipment was turned on and left for 15 min at desired temperature, when sample recipient, spread of a single layer of mango peels (1 mm of thickness), was put into the dryer. Dewatering experiments were performed at 40, 60, 80 and 100 °C and at a constant transversal air velocity of 1.57 m s^{-1}. The air pumped into the dryer presented relative humidity of about 70%. The moisture losses of mango peels were recorded every 5 min by a digital balance (Mettler, model BB3000, Mettler-Toledo AG, Grefensee, Switzerland) with accuracy of ±0.1 g, connected to the samples recipient disposal. In order to provide more uniform drying conditions, an inversion of the drying air stream was performed every 2.5min, switched between ascendant and descendant.

Experiments were stopped when drying rates were less than 0.1 g of water min^{-1}. The moisture content of the samples before and after the drying experiment was determined using the standard method of moisture content determination (AOAC, 1990).

C. Kinetic analysis

Several kinetic equations have been proposed to model drying of food materials and the main models used in recent publications are presented in Table 1.

In these equations, MR is the moisture rate. Drying kinetic models, in Table 1, are assumed to take place in a single step (Eq. 1-5) or in two rates (both exponential (Eq. 6-7)) or one exponential and other with a linear decay (Eq. 8)). k is the drying rate constant, n, c and a are shape constants in one step drying (Eqs. 1-5); k_1 and k_2 are the drying rate constants of the labile and resistant fraction, respectively, and a, b and n are constants (Eq. 6-7). In Eq. 8, k and b are the drying rates of the exponential and linear fraction. a and n are shape constants.

The logarithm representation of drying rate constants against the reciprocal temperature allows the activation energy (E_a) estimation, by the slope of the experimental data fitted to the Arrhenius' law equation. Arrhenius equation is given by the Eq. 9.

$$\ln k = \ln k_0 - \frac{E_a}{RT} \tag{9}$$

where k_0 is a constant (min^{-1}), R is the universal gas constant (8.314 J mol^{-1} K^{-1}), E_a the activation energy (J mol^{-1}) and T is the absolute temperature (K).

D. Comparison of the kinetic models

Experimental data of MR versus drying time were fitted to the models presented in Table 1. Adequacy was evaluated by non-linear regression using software *Statistica* 10.0, based in the Quasi-Newton method. Coefficient of determination (r^2), chi-square (χ^2) and root mean square error (RMSE) were the statistical criteria evaluated in order to test mathematical equation accuracies for reproducing experimental data.

Table 1. Kinetic models for drying process.

Model (nº) / Reference	Equation
Lewis (1) / Bruce (1985)	$MR = \exp(-kt)$
Henderson and Pabis (2) / Henderson and Pabis (1961)	$MR = a\exp(-kt)$
Page (3) / Page (1949)	$MR = \exp(-kt^n)$
Modified Page (4) / White *et al.* (1981)	$MR = \exp(-(kt)^n)$
Logarithmic (5) / Togrul and Pehlivan (2002)	$MR = a\exp(-kt) + c$
Two terms (6) / Hendersen (1974)	$MR = a\exp(-k_1 t) + (1-a)\exp(-k_2 t)$
Approximation of diffusion (7) / Yaldiz *et al.* (2001)	$MR = a\exp(-kt) + (1-a)\exp(-kbt)$
Midilli (8) / Midilli *et al.*, 2002	$MR = a\exp(-kt^n) + bt$

Calculation of χ^2 is done by the equation:

$$\chi^2 = \frac{\sum (a_{mensured} - a_{predicted})^2}{(n-p)} \tag{10}$$

RMSE is defined as:

$$RMSE = \left[\frac{\sum (a_{measured} - a_{predicted})^2}{n} \right]^{1/2} \tag{11}$$

where n is the number of observations and p the number of parameters.

The model with the lowest χ^2 and RMSE, and higher r^2 for MR behavior through time to the studied temperature interval is the best choice for modeling the drying process through time (Midilli *et al*., 2002; Celma *et al*., 2009).

E. Extraction and analysis of total phenolic content

Extraction of total phenolic content (TPC) from dried samples (1 g) was performed accordingly to Ajila *et al*. (2010) with 20 mL of 80% acetone (80:20, v/v) for 1 h. TPC in the extracts was determined by the Folin-Ciocalteau method (Singleton and Rossi, 1965) using gallic acid as standard. The absorbance of the reaction mixture was measured at 765 nm by UV-1600 spectrophotometer (Pró-Análise, Brazil), and results were expressed as mg gallic acid equivalent per gram of dry bagasse weight (mg GAE g^{-1}).

F. Color parameters

Color parameters were measured using Hunter Lab D25-9 solid colorimeter. Response parameters on Hunter Lab scale were: L (lightness or black-white axis), a (green-red axis) and b (blue-yellow axis). Chroma (C^*) and hue angle (Hue^o) were calculated as follows:

$$C^* = \sqrt{a^2 + b^2} \tag{12}$$

$$Hue^o = \tan^{-1}\left(a^* / b^*\right) \tag{13}$$

G. Statistical analysis

Similarities and dissimilarities among the colour parameters and the TPC were elucidated by principal component analysis (PCA). PCA was performed using the *Statistica* 11 software (Statsoft, Tulsa, OK, USA). Multivariate analysis and a pattern recognition method called hierarchical grouping (or cluster analysis) were used to identify similarities among the samples. Drying kinetic parameters, total phenolic content and colour properties were conducted in triplicate and averages of two independent tests were calculated. Obtained values were compared using Tukey's test by *Statistica* 11, and differences were considered statistically significant when p<0.05.

III. RESULTS AND DICUSSION

A. Kinetic modeling

Figure 1 shows experimental data for drying of mango peels in temperature range of 40-100 ºC with transversal air velocity of 1.57 m s^{-1}.

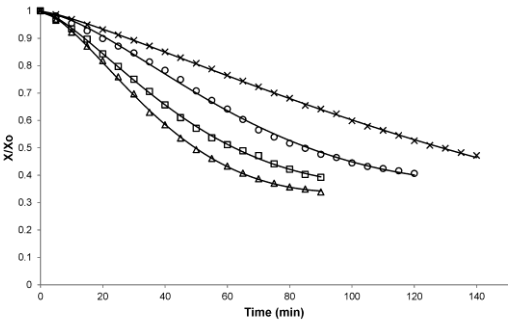

Fig. 1. Drying kinetics of mango peels at 100 (Δ), 80 (□), 60 (o) and 40 ºC (x). Data presented, fitted to a modified Midilli's model, are average values from two independent experiments with standard errors less than 6%.

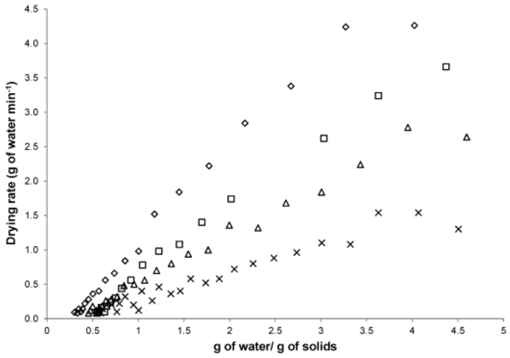

Fig. 2. Curves of drying rates versus moisture content during drying of mango peels at 100 (Δ), 80 (□), 60 (o) and 40 ºC (x).

As expected, *MR* decreased faster when processing temperature was increased due to the higher energy given to the dewatering process. Curves of drying rates versus moisture content (Fig. 2) show that there was no constant drying rate period, and the majority of drying process occurred during a falling rate period. The absence of a constant rate period might be due to no constant supply of water for an appreciable period of time at initial stages of drying (Xiao *et al*., 2010).

A short increasing drying rate period in the beginning of the process is possibly due to heat transfer through the solids, heating-up the food material (Barbosa-Cánovas and Vega-Mercado, 1996). After, drying rates decreased linearly with moisture content, becoming very slow at the last stages during the drying process. Results show that diffusion was the dominant physical mechanism, governing moisture movement from interior to surface of mango peels during drying process.

Several models were statistically evaluated for describing *MR* loss of the fruit residue through time in the temperature interval of 40-100 ºC and results are shown in Table 2.

All models tested presented good fit to experimental data, with high values of r^2 and low values of χ^2 and RSME. However, choosing the best mathematical model to represent processing curves is essential in order to minimize processing errors, maximize final product quality and facilitate designing and simulation of industrial processes. Midilli's model clearly better described the hot air dewatering process of mango peels, with r^2-values in the interval of 0.9995-0.9999, and the lowest values of χ^2 and RSME, ranging from 1.96E-5 to 1.27E-4 and from 1.47E-5 to 3.81E-5, respectively. Midilli's model is a semi-empirical equation proposed by Midilli *et al.* (2002) for single layer drying, which has been used to model the drying curves of mushrooms, pistachio, pollen (Midilli *et al*., 2002) and grape by-products (Celma *et al.*, 2009). The main difference between Page's, Lewis' and two fractions model in comparison to the equation proposed by Midilli *et al*. (2002) is the "*bt*" term, which plays considerable correction of tendency curves, mainly when processing moisture is close to equilibrium. It suggests that, in the end of the drying process, *MR* decays linearly through time, unlike exponentially as proposed by the other equations (Hendersen, 1974; Bruce, 1985; Henderson and Pabis, 1961).

Table 3 shows kinetic parameters estimated by non-linear regression. The *n*-values computed to be less than 1 indicate the 'tailing' phenomena with the retention curve having an upward concavity (Corradini and Peleg, 2004). This might be inferred as an indication of the presence of a strongly bounded fraction of water to the residue matrix.

Table 2. Maximum and minimum error parameters for fitting experimental data to different models in temperature range of 40-100 C.

Model	Lewis (1)
r^2	[0.9943;0.9983]
χ^2	[3.22E-4;1.13E-3]
RSME	[1.45E-4;5.07E-4]
Model	Henderson and Pabis (2)
r^2	[0.9952;0.9985]
χ^2	[3.39-4;1.00E-3]
RSME	[1.35E-4;1. 60E-3]
Model	Page (3)
r^2	[0.9961;0.9985]
χ^2	[2.15E-4;5.89E-4]
RSME	[8.60E-5;2.35E-4]
Model	Modified Page (4)
r^2	[0.9961;0.9985]
χ^2	[2.15E-4;5.89E-4]
RSME	[8.60E-5;2.35E-4]
Model	Logarithmic (5)
r^2	[0.9992;0.9997]
χ^2	[3.10E-5;1.40E-4]
RSME	[1.25E-5;1.63E-4]
Model	Two terms (6)
r^2	[0.9945;0.9998]
χ^2	[3.52E-5;9.45-4]
RSME	[1.06E-5;8.01E-4]
Model	Approximation of diffusion (7)
r^2	[0.9939;0.9997]
χ^2	[3.16E-5;3.16E-4]
RSME	[7.91E-3;7.90E-4]
Model	Midilli (8)
r^2	[0.9995;0.9999]
χ^2	[1.96E-5;1.27E-4]
RSME	[1.47E-5;3.81E-5]

Table 3. Kinetic parameters from Modified Midilli's model for drying mango peels.

T(ºC)		
40	*k* (min^{-n})	0.0203±0.0008[a]
	b (min^{-1})*	0.000399[a]
	N	0.975
60	*k* (min^{-n})	0.0256±0.0012[b]
	b (min^{-1})*	0.000419[b]
	N	1.011
80	*k* (min^{-n})	0.0405±0.0017[c]
	b (min^{-1})*	0.000547[b]
	N	0.961
100	*k* (min^{-n})	0.0367±0.0012[c]
	b (min^{-1})*	0.000997[c]
	N	1.067

Table 4. Total phenolic content and CIELAB colour parameter of dried mango peels.

T (ºC)	TPC (mg GAE g^{-1})	*Colour parameter*	
100	12.72±0.22[a]	*a**	6.70±0.28
		*b**	25.10±0.42
		*L**	40.65±0.64
		Hue	75.09±0.34
		*C**	25.98±0.34
80	15.60±0.51[b]	*a**	6.40±0.42
		*b**	28.50±1.02
		*L**	43.75±0.49
		Hue	77.34±1.70
		*C**	29.22±1.74
60	15.36±0.10[b]	*a**	5.85±1.01
		*b**	30.80±0.85
		*L**	50.70±0.28
		Hue	79.28±2.53
		*C**	31.35±1.32
40	16.50±1.84[b]	*a**	5.24±0.49
		*b**	35.20±0.28
		*L**	49.05±0.64
		Hue	81.58±0.79
		*C**	35.59±0.13
Fresh	19.98±1.72[c]	-	-

As expected, drying rate constants (*k*- and *b*-values) increased with increasing processing temperature. For the process at 100 and 80 ºC, *k*-values did not differ significantly (p>0.05). *k*-values varied from 0.0203 to 0.0405 min^{-n}, meanwhile *b*-values from 0.000399 to 0.000997 min^{-1} in temperature range of 40-100 ºC. Lower values of the *b*-parameter in relation to the *k*-parameter, demonstrates that the "*bt*" term is related to a slower process of dewatering, in the end of drying. Activation energy (E_a), estimated by the Arrhenius approach, was of 10.72 kJ mol^{-1} for fitting *b*-values, while E_a from *k*-values was of 14.27 kJ mol^{-1}. These results corroborate to the premise that *b*-values are related to the final stage of drying, where *MR* is low, and removal of water from foods is more difficult, requiring higher energy (Barbosa-Cánovas and Vega-Mercado, 1996).

B. Total phenolic content

Beyond of modeling changes in *MR* through time, analysis of the effect of the process over bioactive compounds is an important step to the proper utilization of mango peel powder in the food and pharmaceutical industries. Table 4 shows the extractable TPC of the dried samples of mango peels. Fresh mango peel presented 19.98 mg GAE g^{-1}, meanwhile TPC from dried samples decreased significantly (p<0.05) varying from 16.50 to 12.72 mg GAE g^{-1} in the temperature interval of 40-100 ºC. The increase of the drying temperature, as expected, led to a decrease on the final concentration of polyphenols, resulted from the thermal degradation. Larrauri *et al*. (1997) observed that total phenolic content of red grape pomace was stable to the drying process happening at 60 ºC. Dorta *et al*. (2012) verified that mango peels dried at 70 ºC did not presented significant thermal degradation of phenolics. In the present work, it can be observed that TPC in mango peels did not change significantly when the drying temperature was increased up to 80 C. Similar results were verified by Vega-Gálvez *et al*. (2012) with drying procedures of apple slices at 80 C with air velocity of 1.5 m s^{-1}. The results suggest that it is possible to dry food materials at higher temperatures, which lead the procedure to be faster, with maximum preservation of the phenolic content.

C. Colour

CIELAB parameters of dried mango peels are presented in Table 4. *a** and *b**-values were positive with calculated *hue* in the range of 75-81, indicating that samples presented an yellow colour. *C** shows up as a function of parameters to represent a physical brightness the emitting/reflecting surface.

It ranged from 35.59 to 25.98, also decreasing with the increasing of the drying temperature as well as the *hue* and *L** values. The results from dried samples' CIELAB parameters indicate the degradation of pigment compounds to darker materials, possibly due to non-enzymatic reactions.

D. Tracking colour changes to degradation of total phenolic content

Usual methodologies to analyze bioactive compounds involve chromatography and/or chemical reaction turning them laborious, time consuming and consequently not interesting for industrial application. Thus, developing alternative methods for online quality control application is a topic of utmost relevance. For this propose the results of TPC and the CIELAB parameters were analyzed by PCA.

Two principal components accounted for 97.15% for the variance in the data (Figure 3). The first principal component (93.39% of the total data inertia) presented negative values for *b**, *L**, *C**, *hue* and TPC, while the chromatic coordinate *a** values were positive. The PC1 component was likely related to the degradation of TPC and the decrease of values of *hue, C**, *b** and increase in *a**-values on the dried mango peels.

Table 5 shows the Pearson correlation coefficients between the colour parameters and the TPC in the dried mango peels. TPC was highly related to the changes on *b**, *Hue* and *C** parameters with *r*-values close to 0.9. These CIELAB parameters are related to the yellowness intensity of the samples and results suggests that the degradation of TPC is related to the loss of yellow pigments from the mango peels.

Table 5. Pearson correlation coefficients between colour parameters and total phenolic content of dried mango peels.

*a**	*a**		*Hue*	-0.9884
	*b**	-0.9883	*C**	-0.9879
	*L**	-0.8530	TPC	-0.8147
*b**	*a**	-0.9883	*Hue*	0.9958
	*b**		*C**	1.0000
	*L**	0.8312	TPC	0.8885
*L**	*a**	-0.8530	*Hue*	0.8786
	*b**	0.8312	*C**	0.8274
	*L**		TPC	0.7526
Hue	*a**	-0.9884	*Hue*	
	*b**	0.9958	*C**	0.9951
	*L**	0.8786	TPC	0.8899
*C**	*a**	-0.9879	*Hue*	0.9951
	*b**	1.0000	*C**	
	*L**	0.8274	TPC	0.8884
TPC	*a**	-0.8147	*Hue*	0.8899
	*b**	0.8885	*C**	0.8884
	*L**	0.7526	TPC	

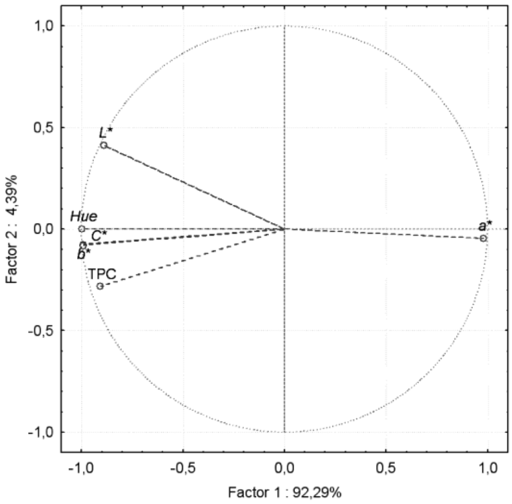

Figure 3. Projection of the color parameters and total phenolic content in dried mango peels in the plane defined by PCA.

The results are in agreement to Larrauri *et al*. (1997) who verified that degradation of TPC was related to colour changes in dried grape pomace, which can be also observed in work from Vega-Gálvez *et al*. (2012) for dried apple slices. In this sense, colour changes, measured by the CIELAB parameters, showed to be a fast and practical alternative to indirectly control TPC degradation of food materials subjected to hot air drying processes.

Plant materials are complex systems and caution must be exercised when tracking colour changes of molecular compounds during industrial applications. The degradation of nutritional compounds involves a complex network of reactions being also important to point out that foods may respond differently to the different processes and conditions employed in different parts of the world. However, it is clear that there is a relationship between the content of TPC and the colour parameters in dried mango peels. This suggests that a colorimetric technique may be appropriate to assess the quality of plant matrixes.

IV. CONCLUSION

A successful modeling of drying kinetics will enable the processors to modulate the process to achieve desirable relative moisture in the end of thermal process and better equipment sizing for industrial projects. Based on an isothermal experiment in the temperature range of 40-100 ºC, Midilli's model provided the best description of the kinetics of drying mango peels. TPC was sensitive to drying hot air, however it was possible to increase the drying temperature up to 80 ºC with no significant changes on TPC in the dried samples. Furthermore, the polyphenol degradation could be accompanied by CIELB parameters related to the yellowness of the dried samples. In this concern, the knowledge about kinetics and association of bioactive compounds in drying procedures in a larger spectrum of foods and processing conditions is an essential step to allow this approach to be used in industrial applications.

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