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Thermodynamic properties and drying kinetics of 'okara'

Rafaiane M. Guimarães¹, Daniel E. C. de Oliveira², Osvaldo Resende³,
Jhessika de S. Silva⁴, Thaisa A. M. de Rezende⁴ & Mariana B. Egea¹

¹ Instituto Federal de Educação, Ciência e Tecnologia Goiano/Programa de Pós-Graduação em Tecnologia de Alimentos/Campus Rio Verde. Rio Verde, GO. E-mail: rafaiane.guimaraes@ifgoiano.edu.br (Corresponding author) - ORCID: 0000-0001-6117-8986; mariana.egea@ifgoiano.edu.br - ORCID: 0000-0001-7589-2718

² Instituto Federal de Educação, Ciência e Tecnologia Goiano/Campus Iporá. Iporá, GO. E-mail: oliveira.d.e.c@gmail.com - ORCID: 0000-0002-3824-994X

³ Instituto Federal de Educação, Ciência e Tecnologia Goiano/Diretoria de Pesquisa e Pós-Graduação/Campus Rio Verde. Rio Verde, GO. E-mail: osvaldo.resende@ifgoiano.edu.br - ORCID: 0000-0001-5089-7846

⁴ Instituto Federal de Educação, Ciência e Tecnologia Goiano/Campus Rio Verde. E-mail: jhessikasantanas@gmail.com - ORCID: 0000-0002-2634-1140; t-alves-matos@live.com - ORCID: 0000-0002-5213-5172

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ABSTRACT

'Okara' is the insoluble part obtained after the aqueous extraction of soybeans, generated in large quantities as a by-product of the 'tofu' industry or soybean water-soluble extract. This work aimed to study 'okara' convective drying kinetics, determine the effective diffusion coefficient, and obtain activation energy and thermodynamic properties under different drying conditions. The by-product 'okara' was obtained from the processing of BRS 257 soybean water-soluble extract, homogenized and dried in a forced-air oven at temperatures of 40, 50, 60 and 70 °C until constant weight. Among the analysed models, Wang & Singh was selected to represent the drying phenomenon. Effective diffusion coefficient increased with the temperature rise, and the activation energy for the net diffusion in the drying was 28.15 kJ mol⁻¹. Enthalpy and Gibbs free energy increased with the elevation of drying temperature.

Palavras-chave:

modelagem matemática
energia de ativação
coeficiente de difusão

Propriedades termodinâmicas e cinética de secagem de okara

RESUMO

Okara é a parte insolúvel obtida após a extração aquosa dos grãos de soja, gerado em grande quantidade como um subproduto da indústria de tofu ou de extrato hidrossolúvel de soja. Objetivou-se estudar a cinética de secagem convectiva do okara, determinar o coeficiente de difusão efetivo, obter a energia de ativação e as propriedades termodinâmicas em diferentes condições de secagem. O subproduto okara foi obtido a partir do processamento de 'extrato hidrossolúvel de soja' de grãos de soja BRS 257, homogeneizado e submetido à secagem em estufa de ventilação forçada nas temperaturas de 40, 50, 60 e 70 °C até atingirem peso constante. Dentre os modelos analisados, Wang & Singh foi o selecionado para representar o fenômeno de secagem. O coeficiente de difusão efetivo aumentou com a elevação da temperatura e a energia de ativação para a difusão líquida na secagem foi de 28,15 kJ mol⁻¹. A entalpia decresceu com o aumento da temperatura de secagem, enquanto a entropia e energia livre de Gibbs aumentaram com o acréscimo da temperatura de secagem.



INTRODUCTION

'Okara' is the by-product of the 'tofu' production process or soybean water-soluble extract, and is the insoluble part left after aqueous extraction from soybeans (Mateos-Aparicio et al., 2010). Due to its high moisture content, 'okara' is very perishable; however, when dry (in the form of flour), it has rich nutritional composition and yet is normally undervalued by the food industry and widely used as animal feed (Vong et al., 2016).

The interest in new forms of using 'okara' and its incorporation in human diet has increased in the last two decades, since it contains approximately 40-60% of carbohydrates (especially insoluble fiber), 20-30% of proteins and 10-20% of lipids on dry basis (Li et al., 2011).

Drying processes are widely used as methods of conservation in the food industry, because they reduce moisture content in foods and limit the deterioration rate. However, to obtain the desired final product with the lowest energy demand possible, it is necessary to study product characteristics and drying conditions. In this context, mathematical modeling stands out, and thus dried 'okara' can be conserved for a longer time and added as ingredient in various foods (Perussello et al., 2012).

In addition to the importance of mathematical models in predicting the drying behavior of a certain product, the thermodynamic properties in the drying steps are relevant sources of information to design dryers, calculate the energy required in the step, study the properties of the adsorbed water, evaluate food microstructure and study physical phenomena that occur on food surface (Oliveira et al., 2011).

Mathematical modeling depends on the drying conditions, such as time, temperature and type of equipment used. Perussello et al. (2012) have already studied other 'okara' drying processes using devices such as spray-dryer adapted with pneumatic pipe (130, 150 and 170 °C), rotary drum (50, 60 and 70 °C) and fixed-bed dryer (130, 150 and 170 °C), but the diffusion coefficient and thermodynamic properties were not investigated by these authors.

Therefore, this work aimed to study the drying phenomena and fit mathematical models, determine and evaluate the effective diffusion coefficient, obtain activation energy and thermodynamic properties of 'okara' drying to obtain flour at temperatures of 40, 50, 60 and 70 °C, which are commonly used in food production.

MATERIAL AND METHODS

Grain samples of the soybean variety BRS 257 were donated by the SL Alimentos - Paraná, Brazil (23° 44' 20" S; 51° 18' 36" W; 722 m) to the Laboratory of Postharvest of Plant Products of the Federal Institute of Goiás (IF Goiano) - Campus of Rio Verde. The grains were pre-processed to separate physical impurities (rocks, leaves and earth residues) and washed in running drinking water to remove the remaining dirt. 'Okara' was obtained through the extraction method described by Baú et al. (2015). The creamy mass of 'okara' obtained was then homogenized and stored at -18 °C until the moment of drying.

'Okara' was thawed at room temperature immediately before drying, which was conducted at temperatures of 40, 50,

60 and 70 °C, under internal relative humidity of 19.1, 8.5, 6.4, 4.6%, respectively, in a forced-air oven (Ethik Technology/400-4ND). Drying air temperatures and room temperature were monitored by thermometers inside and outside the dryer, and the relative humidity inside the oven was obtained based on the basic principles of psychrometry, using the computer program GRAPSI.

Wet 'okara' was arranged in four 250 g portions on metal trays (23 cm long x 8.5 cm wide) in 1.5 cm thick layer of mass. The trays were weighed to obtain the moisture content of 0.118 (decimal, dry basis), and the moisture content was calculated by difference between masses at the times, considering the initial moisture content. Initial moisture content was 3.848 (decimal, dry basis) whereas final moisture content was 0.118 (dry basis).

Moisture content ratios in 'okara' during drying were determined using Eq. 1:

$$RX = \frac{X - X_e}{X_i - X_e} \quad (1)$$

where:

RX - moisture content ratio of the product, dimensionless;
X - moisture content of the product at a certain time instant, decimal, d.b.;

X_i - initial moisture content of the product, decimal, d.b.;
and,

X_e - equilibrium moisture content of the product (decimal, d.b.).

'Okara' drying was predicted using 11 mathematical models, presented in Eq. 2 to 12:

- Wang & Singh

$$RX = 1 + a \cdot t + b \cdot t^2 \quad (2)$$

- Verma

$$RX = a \cdot \exp(-k \cdot t) + (1 - a) \exp(-k_1 \cdot t) \quad (3)$$

- Thompson

$$RX = \exp \left\{ \frac{\left[-a - \left(a^2 + 4 \cdot b \cdot t \right)^{\frac{1}{2}} \right]}{2 \cdot b} \right\} \quad (4)$$

- Page

$$RX = \exp(-k \cdot t^n) \quad (5)$$

- Newton

$$RX = \exp(-k \cdot t) \quad (6)$$

- Midilli

$$RX = a \cdot \exp(-k \cdot t^n) + b \cdot t \quad (7)$$

- Logarithmic

$$RX = a \cdot \exp(-k \cdot t) + c \tag{8}$$

- Henderson & Pabis

$$RX = a \cdot \exp(-k \cdot t) \tag{9}$$

- Two-Term Exponential

$$RX = a \cdot \exp(-k \cdot t) + (1-a) \exp(-k \cdot a \cdot t) \tag{10}$$

- Two Terms

$$RX = a \cdot \exp(-k_0 \cdot t) + b \cdot \exp(-k_1 \cdot t) \tag{11}$$

- Approximation of Diffusion

$$RX = a \cdot \exp(-k \cdot t) + (1-a) \cdot \exp(-k \cdot b \cdot t) \tag{12}$$

where:

- t - drying time (h);
- k, k₀, k₁ - drying constants (h⁻¹); and,
- a, b, c, n - coefficients of the models.

The mathematical models were fitted using nonlinear regression analysis by Gauss-Newton method in the statistical program Statistica® 7.0. The degree of fit of the models to the experimental data was evaluated based on the magnitude of the adjusted coefficient of determination (R²), mean estimated error (SE) and mean relative error (P).

Net diffusion was described using the flat plate model, with approximation of eight terms, according to Eq. 13:

$$RX = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2 \cdot n + 1)^2} \cdot \exp \left[- \frac{(2 \cdot n + 1)^2 \cdot \pi^2 \cdot D_{ef} \cdot t}{4} \cdot \left(\frac{S}{V} \right)^2 \right] \tag{13}$$

where:

- D_{ef} - effective diffusion coefficient, m² s⁻¹;
- n - number of terms;
- t - time, s;
- S - surface area of the 'okara' layer, m²; and,
- V - volume of the 'okara' layer, m³.

The relationship between effective diffusion coefficient and increase in drying air temperature was described by the Arrhenius equation (Eq. 14):

$$D_{ef} = D_0 \exp \left(\frac{-E_a}{R \cdot T_a} \right) \tag{14}$$

where:

- D₀ - pre-exponential factor, m² s⁻¹;
- E_a - activation energy, kJ mol⁻¹;

- R - universal gas constant, 8.134 kJ kmol⁻¹ K⁻¹; and
- T_a - absolute temperature, K.

Thermodynamic properties of 'okara' drying were obtained using the method described by Jideani & Mpotokwana (2009), according to Eqs. 15 to 17.

$$\Delta H = E_a - R \cdot T \tag{15}$$

$$\Delta S = R \cdot \left(\ln A - \ln \frac{k_B}{h_p} - \ln T_{abs} \right) \tag{16}$$

$$\Delta G = \Delta H - T_{abs} \cdot \Delta S \tag{17}$$

where:

- ΔH - enthalpy, J mol⁻¹;
- ΔS - entropy, J mol⁻¹;
- ΔG - Gibbs free energy, J mol⁻¹;
- k_B - Boltzmann constant, 1.38 x 10⁻²³ J K⁻¹; and,
- h_p - Planck constant, 6.626 x 10⁻³⁴ J s⁻¹.

RESULTS AND DISCUSSION

The moisture content ratios of the flours for different temperatures and drying times were fitted to eleven mathematical models, to define which one adequately described the drying process. The coefficients of determination (R²) and mean relative errors (P) of the models fitted are presented in Table 1.

The coefficients of determination of all models were higher than 0.97 for all drying temperatures, which indicates a satisfactory fit of the mathematical models to the experimental data (Kashaninejad et al., 2007). Besides the coefficient of determination, SE and P values were also considered to select the models of drying for 'okara' flour production.

According to Draper & Smith (1998), the capacity of a model to faithfully describe a certain physical process is inversely proportional to the standard deviation of the estimate. Thus, the lower the SE values, the better the model's fit to the observed data. Based on this, the models Wang & Singh (2) and Midilli (7) showed the lowest SE values and the magnitude of P for these models was below 10%, thus showing a satisfactory fit to the experimental results. However, the Wang & Singh model was selected because it is the simplest one among the others analysed and has the lowest parameters (SE and P), consequently a satisfactory fit.

Figure 1 shows the estimated moisture content data (A) and the moisture content ratio over time with values obtained experimentally and estimated by the Wang & Singh model (B). In this case, it is noted that the lines of the estimated values were close to those of the experimental data of 'okara' drying, showing along with the other parameters evaluated that the fit is satisfactory.

Moisture content ratio decreased with the increment of temperature and drying time. Drying speed for all temperatures tested increased in the first 3 hours and gradually decreased as time progressed.

Table 1. Mean relative error (P, %), coefficient of determination (R^2) and mean estimated error (SE, decimal) for different conditions of 'okara' drying

Model	Temperature (°C)											
	40			50			60			70		
	P	R^2	SE	P	R^2	SE	P	R^2	SE	P	R^2	SE
Wang & Singh	1.98	0.9993	0.010	1.53	0.9992	0.010	5.98	0.9995	0.007	2.06	0.9992	0.009
Verma	31.20	0.9870	0.043	20.08	0.9958	0.023	18.68	0.9980	0.015	4.09	0.9987	0.011
Thompson	31.20	0.9870	0.042	40.20	0.9861	0.041	60.08	0.9840	0.042	25.61	0.9783	0.046
Page	14.72	0.9960	0.024	20.66	0.9948	0.025	25.82	0.9968	0.019	10.69	0.9969	0.017
Newton	31.20	0.9870	0.041	40.20	0.9861	0.040	60.07	0.9840	0.040	25.60	0.9783	0.044
Midilli	5.15	0.9990	0.012	7.44	0.9991	0.011	9.57	0.9991	0.011	1.04	0.9999	0.004
Logarithmic	7.22	0.9988	0.013	8.56	0.9991	0.011	16.82	0.9984	0.014	2.59	0.9995	0.007
Henderson & Pabis	28.77	0.9887	0.040	36.97	0.9877	0.038	51.65	0.9886	0.035	21.37	0.9860	0.037
Two-Term Exponential	16.33	0.9963	0.023	40.20	0.9861	0.041	60.07	0.9840	0.042	25.61	0.9783	0.046
Two Terms	13.66	0.9973	0.020	36.96	0.9877	0.041	25.42	0.9973	0.018	21.37	0.9860	0.039
Approximation of Diffusion	14.84	0.9967	0.022	20.08	0.9958	0.023	18.68	0.9980	0.015	4.09	0.9987	0.011

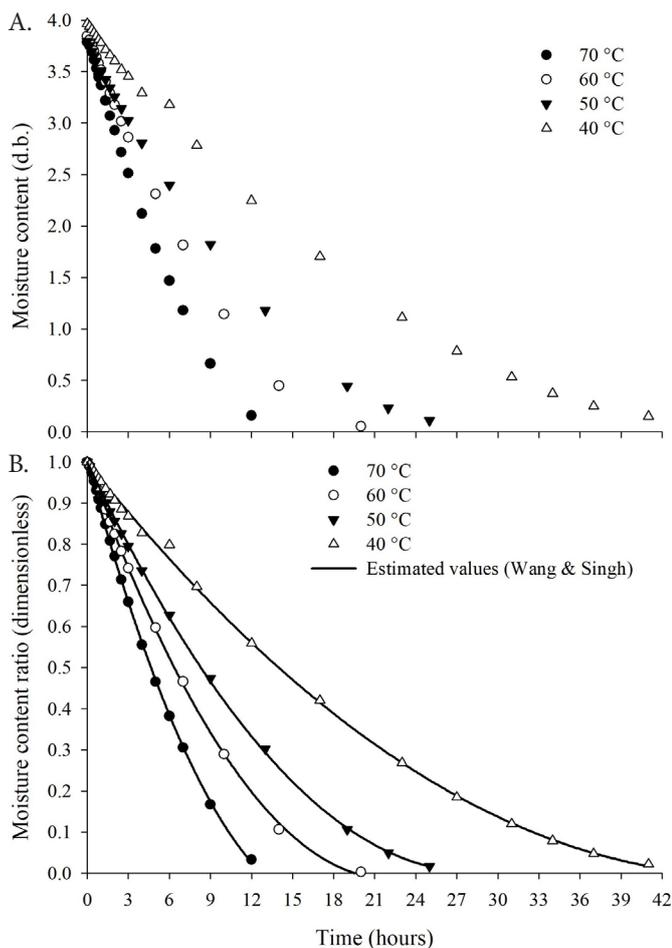


Figure 1. Experimental moisture content (A) and moisture content ratio (B) of 'okara' (decimal, d.b.) with values obtained experimentally and estimated by the Wang & Singh model, as a function of drying time (hours) for the temperatures of 40, 50, 60 and 70 °C

Initial moisture content in the flour was equal to 3.848 ± 0.082 (d.b.), decreasing to 0.118 ± 0.048 (d.b.) for dry 'okara', and the drying times required to reach this moisture content were 12, 20, 25 and 41 h for the temperatures of 70, 60, 50 and 40 °C, respectively.

According to Muliterno et al. (2017), when higher drying temperatures are used, there is greater mass transfer due to the higher water evaporation capacity of the air. Additionally, at higher temperatures, water diffusivity is greater, leading

to increased water transfer rate inside the sample. However, higher temperatures may result in significant heat losses to the environment and undesirable alterations in sensory quality.

Costa et al. (2011) and Smaniotto et al. (2017) fitted mathematical models to the drying data of crambe (*Crambe abyssinica*) seeds and sunflower grains, respectively, under different air conditions (temperature and relative humidity), and the Wang & Singh model was selected because it had the best fit to the experimental data.

The coefficients 'a' and 'b' fitted to the experimental data of 'okara' flour drying kinetics at the different temperatures studied are shown in Table 2.

Muliterno et al. (2017) modeled 'okara' drying kinetics and the Page model fitted well to the drying kinetics data. The difference between the model fitted by these authors and the one in the present study may be due to the different relative moisture ranges in the experiments, as well as to the model selection criterion used by these authors (only evaluation of coefficient of determination - R^2).

There was a linear increase in the effective diffusion coefficients with the increment in temperature, which indicates greater magnitude of water transfer from inside to the periphery of the product (Figure 2), as demonstrated by Silva et al. (2014), Oliveira et al. (2014a) and Smaniotto et al. (2017).

The dependence of the effective diffusion coefficient of 'okara' flour on drying air temperature and speed was also represented by the Arrhenius equation (Figure 2).

Activation energy can be defined as the minimum energy required for water molecules to begin the movement from inside to outside of the product (Corrêa et al., 2005). The activation energy for 'okara' net diffusion was $28.15 \text{ kJ mol}^{-1}$, within the temperature range from 40 to 70 °C. The value found here is within the range cited in the literature and lower than that of forced convection drying of pumpkin ($33.74 \text{ kJ mol}^{-1}$) (Guiné et al., 2011); drying of crambe seeds ($37.07 \text{ kJ mol}^{-1}$) for the temperature range from 30 to 70 °C (Costa et al., 2011);

Table 2. Wang & Singh model parameters fitted to the experimental data of 'okara' flour drying

Parameters	Temperature (°C)			
	40	50	60	70
a	-0.041815**	-0.070555**	-0.09207**	-0.123793**
b	0.000435**	0.001248**	0.002109**	0.003557**

**Significant at 0.01 probability level by t-test

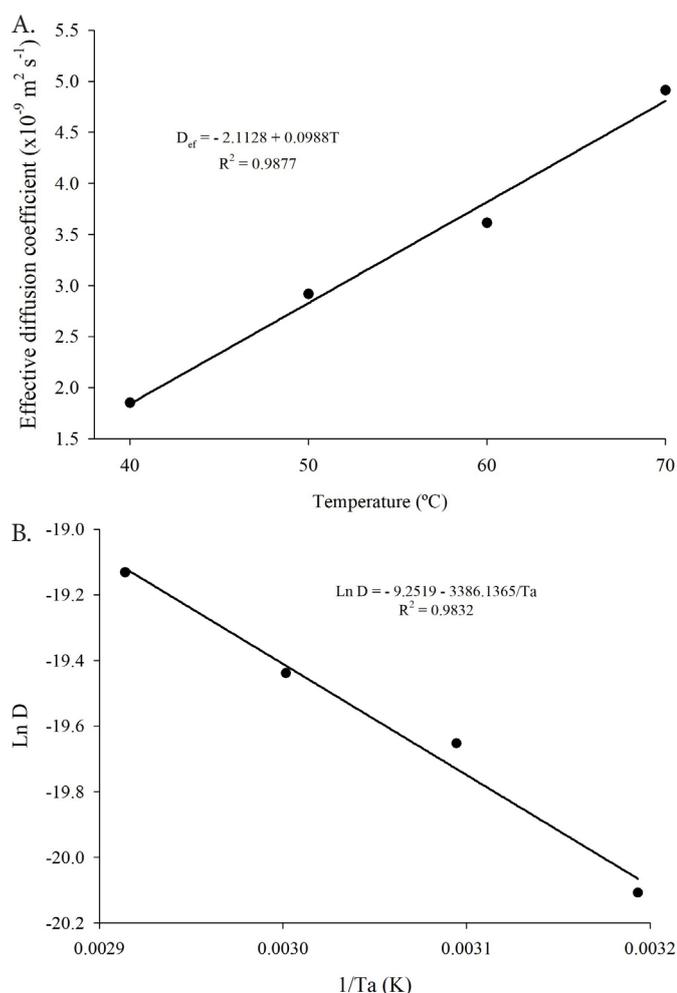


Figure 2. Mean values of the diffusion coefficient ($m^2 s^{-1}$) obtained for 'okara' drying (A) and Arrhenius representation for the diffusion coefficient as a function of drying air temperature (B)

and higher than that of soybean drying ($22.77 kJ mol^{-1}$) at temperatures of 40 to 100 $^{\circ}C$ reported by Oliveira et al. (2013).

The values of enthalpy, entropy and Gibbs free energy for the different drying conditions are shown in Table 3. With the increment in drying temperature, enthalpy decreases and Gibbs free energy increases.

Enthalpy is related to the energy needed to remove water bound to the product during the drying process. Thus, the higher the drying temperature, the lower the enthalpy (Oliveira et al., 2010). Higher enthalpy at lower temperatures indicates greater amount of energy required to promote the drying to obtain 'okara' flour.

Entropy is a thermodynamic property that can be associated with the level of disorder between water and product (Goneli et al., 2010). Entropy increased with the increment in drying air temperature. This pattern was expected because the elevation

Table 3. Values of enthalpy (ΔH), entropy (ΔS) and Gibbs free energy (ΔG) of 'okara' for different drying air conditions

Temperature ($^{\circ}C$)	Thermodynamic properties		
	ΔH ($J mol^{-1}$)	ΔS ($J mol^{-1} K^{-1}$)	ΔG ($J mol^{-1}$)
40	25548.51	-168.39	78281.17
50	25465.37	-168.66	79966.42
60	25382.23	-168.91	81654.25
70	25299.09	-169.15	83344.58

of drying temperature increases the excitation of water molecules in the product and, consequently, the order of the water-product system (Corrêa et al., 2010).

Gibbs free energy is related to the work needed to make the sorption sites available (Nkolo Meze'e et al., 2008). Gibbs free energy in the present study was positive and increased with the increment in the drying temperature.

Positive Gibbs free energy indicates non-spontaneous reaction, which requires the introduction of energy from the environment for the phenomenon to occur, in this case the drying of the product (Corrêa et al., 2011).

The results of the present study corroborate those of Oliveira et al. (2013, 2014b), for the drying of soybean grains, and Resende et al. (2014), for the drying of sorghum grains, who also obtained reduction of enthalpy and with increment of temperature and positive Gibbs free energy.

CONCLUSIONS

1. The Wang & Singh model satisfactorily represents 'okara' drying.
2. Effective diffusion coefficient increased with the elevation of drying temperature, evidencing that 'okara' drying speed depends on drying temperature, and the activation energy to obtain 'okara' drying was equal to $28.15 kJ mol^{-1}$.
3. Enthalpy decreased with the elevation of temperature, whereas entropy and Gibbs free energy increased.
4. The results obtained can be useful to develop industrial processes and design equipment, as well as help control the quality of products manufactured from 'okara' flour.

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