Croatian Journal of Food Science and Technology
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Moisture desorption isotherms and drying kinetics of pasta enriched with pecan nut flour

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ARTICLE INFO

TYPE: Original scientific paper

https://doi.org/10.17508/CJFST.2026.18.1.10

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ARTICLE HISTORY

Received: April 1, 2026

Accepted: June 24, 2026

CITATION

Muchiutti GS, Genoveva Cámera N, Larrosa VJ. Moisture desorption isotherms and drying kinetics of pasta enriched with pecan nut flour. Croatian Journal of Food Science and Technology (2026) 18 (1) 134—146

KEYWORDS

pecan nut flour; sorption isotherms; drying kinetics; effective diffusivity

KEY CONTRIBUTION

First characterisation of moisture desorption isotherms of noodles with 50% pecan nut flour. Validation of the GAB model as the best fit for predicting sorption behaviour. Determination of drying kinetics and effective moisture diffusivity at different temperatures. Demonstration of safe moisture content and water activity levels under optimised drying conditions. Contribution to the valorisation of pecan nut flour as a functional ingredient in pasta production.

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ABSTRACT

The growing interest in nutritionally enhanced pasta products has driven the search for functional ingredients capable of improving their lipid, protein, and fibre profiles. Pecan nut flour, a by-product of the oil extraction industry, represents a promising alternative; however, its incorporation into pasta formulations modifies dough physicochemical properties and requires optimisation of the drying process to ensure product stability. This study characterised moisture desorption isotherms and drying kinetics of noodles formulated with a 1:1 wheat flour–pecan nut flour ratio. Isotherms were determined at 25 °C and 40 °C (a_w: 0.20–0.92) and fitted to GAB and Oswin models. Drying was performed at 30 °C and 50 °C with 40% relative humidity. The GAB model showed the best fit (R^2 = 0.99; E% < 5%) with monolayer moisture contents of 0.188 and 0.0996 g water/g dry matter at 25 °C and 40 °C, respectively. Drying kinetics were dominated by the falling rate period, described by Fick's second law, yielding diffusion coefficients of 6.62×10^{-12} m²/s (30 °C) and 1.80×10^{-11} m²/s (50 °C). Both conditions produced final moisture contents and water activity values complying with Argentine Food Code limits and microbiological stability thresholds, confirming the technological feasibility of pecan nut flour as a functional ingredient in dry pasta production.

Introduction

By-products obtained after oil extraction by cold pressing of seeds and nuts, commonly referred to as oil cakes or flours, possess high nutritional value and are widely used in the edible oil industry (Sarkis et al., 2014). Pecan nut coproducts, such as oil and press cake, are particularly rich in nutrients and phytochemical compounds (Atanasov et al., 2018; Wakeling et al., 2001), with protein contents ranging from 15% to 50%, depending on the variety, cultivation conditions, and extraction method. Although tocopherols and some phospholipids are partially removed during oil extraction, the phenolic content remains relatively high in the defatted residues. Despite their nutritional value, pecan nut cake or flour has low commercial value and is generally used as animal feed (Salvador et al., 2016). The utilisation of these residues as raw materials for food production not only adds value to the production chain but also helps reduce problems associated with industrial waste disposal (Girotto et al., 2015).

Pasta products are among the most widely consumed foods across all age groups. In Argentina, the sector comprises more than 1,500 companies, with an annual per capita consumption of approximately 9 kg (Ministerio de Agroindustria de la Nación, 2015). Pasta is generally considered a healthy food due to its low-fat content, high levels of complex carbohydrates, and moderate protein content (Sissons, 2004); however, noodles produced from refined wheat flour contain reduced levels of minerals and dietary fibre. In this context, the partial incorporation of pecan nut flour as a replacement for wheat flour represents a strategy to improve the nutritional profile of pasta by increasing its content of unsaturated fatty acids, dietary fibre, and minerals such as manganese, potassium, calcium, and iron. Nevertheless, this substitution modifies the physicochemical properties of the dough and the final product, making it necessary to study and optimise processing conditions, particularly the drying process. Drying is a unit operation aimed at extending shelf life by reducing moisture content through the application of heat, thereby inhibiting microbial growth and enzymatic activity (Chigal et al., 2019). In pasta production, drying kinetics depend on both intrinsic product properties—density, porosity, composition, and geometry—and operational conditions such as temperature, relative humidity, and air velocity (Veladat et al., 2012). The Argentine Food Code establishes a maximum moisture content of 14% (w/w) for dry pasta products, and inadequate drying may cause thermal and mechanical damage affecting texture, colour, and nutritional value (Migliori et al., 2005).

Knowledge of moisture sorption properties is essential for modelling and optimising the drying process. Water sorption isotherms relate equilibrium moisture content to water activity (a_w) at a given temperature and constitute a fundamental thermodynamic tool for predicting product stability and selecting appropriate packaging materials (Labuza and Altunakar, 2020; Chirife and del Pilar Buera, 1994). Water activity also indicates the availability of water to participate in deterioration reactions, such as lipid oxidation, enzymatic reactions, and Maillard browning, and is widely used as an indicator of shelf-life (Comaposada et al., 2000). Among the mathematical models proposed to describe sorption behaviour, the Guggenheim–Anderson–de Boer (GAB) model is one of the most widely used due to its theoretical basis and broad applicability (van den Berg and Bruin, 1981), whereas the Oswin model is frequently applied for cereal-based products due to its simplicity and adequate fitting capacity (Al-Muhtaseb et al., 2002). Drying kinetics can additionally be analysed through diffusion-based approaches derived from Fick's second law, which allow estimation of effective moisture diffusivity (Crank, 1975; Doymaz, 2012). Although sorption and drying studies have been reported for pasta made from conventional and alternative flours such as corn, rice, and legumes, information regarding pasta enriched with pecan nut flour remains scarce. In particular, studies characterising desorption isotherms and drying kinetics of such products are still limited, restricting the possibility of optimising their industrial processing and ensuring product stability during storage.

The present work aimed to characterise the drying process of noodles made with partial substitution of wheat flour by pecan nut flour, through the study of moisture desorption isotherms and the analysis of drying kinetics under different temperature conditions. Specifically, the objectives were: (i) to determine moisture desorption isotherms at 25 °C and 40 °C and fit the experimental data to the GAB and Oswin models, to establish the reference equilibrium moisture content for safe product storage; and (ii) to study the effect of drying temperature (30 °C and 50 °C) on moisture loss kinetics, to model the drying curves using Fick's second law, and to verify compliance with regulatory requirements for final product moisture content and water activity.

Materials and methods

Noodle preparation

Noodles were prepared by partially replacing wheat flour (0000 type, PUREZA®, Argentina) with pecan nut flour at a 1:1 ratio. The formulation consisted of 31.98 g of wheat flour, 31.98 g of pecan nut flour, 26.87 g of fresh egg (Grade N1, SyB S.A.), 8.88 g of potable water, and 0.28 g of fine salt (TRESAL®), expressed as grams per 100 g of total formulation.

Manufacturing process

Noodle manufacture was carried out using a commercial food processor (LILIANA, Model AM 323, Argentina). Dry ingredients were homogenised for 1 minute at 400 rpm, after which fresh egg and water were incorporated sequentially, and kneading was extended for 5 minutes until a homogeneous, cohesive, and non-adhesive dough was obtained. The dough was allowed to rest for 10 minutes at 20 ± 2 °C to promote gluten network relaxation and was then sheeted and cut into ribbons of approximately 30 cm × 7 mm × 1.2 mm (n = 10, measured with a digital calliper).

Sorption isotherms

Moisture desorption isotherms were determined using the static gravimetric method with saturated salt solutions as reference atmospheres (Larrosa, 2014). Equilibrium moisture content (Xw) was determined in duplicate according to AACC Method 44-40 (AACC, 1984), by drying samples at 105 °C to constant mass. Water activity was measured using a portable water activity meter (LabSwift-a_w, Novasina, Switzerland). Isotherms were constructed at 25 °C and 40 °C over a water activity range of 0.20–0.92, covering both the monolayer and multilayer regions.

Sorption isotherm modelling

Experimental equilibrium moisture content data were fitted by nonlinear least-squares regression to the Guggenheim–Anderson–de Boer (GAB) model (Eq. 1) and the Oswin model (Eq. 2), both of which have previously been applied with satisfactory results to pasta products of similar composition (Sefrienda et al., 2022). The GAB model was selected because of its theoretical basis in multilayer adsorption theory and its broad applicability (van den Berg and Bruin, 1981), whereas the Oswin model was included because of its simplicity and adequate fitting capacity for cereal-based matrices (Al-Muhtaseb et al., 2002). The GAB model is expressed as:

$$X_w = (X_m * C * aw)/[(1 - K * aw) * (1 + (C - 1) * K * aw)] \quad (1)$$

where Xw is the equilibrium moisture content (g water/g dry matter); Xm is the monolayer moisture content (g water/g dry matter); K is a correction factor related to the heat of multilayer sorption; and C is the Guggenheim constant related to the heat of monolayer sorption.
The Oswin model is expressed as:

$$X_w = A * [aw/(1 - aw)]^B \quad (2)$$

where A and B are empirical constants characteristic of each food system.
The monolayer moisture content (Xm) was used as a stability reference indicator, given that values below Xm are associated with minimised deterioration reaction rates (Labuza and Altunakar, 2020). The target moisture content for safe storage was established as the Xw corresponding to $a_w \leq 0.63$ at 25 °C, below which the development of most spoilage microorganisms is inhibited (Chirife and del Pilar Buera, 1994).

Experimental drying curves

Equipment and operating conditions

Drying experiments were conducted in a laboratory-scale tray dehydrator with forced air circulation. Temperature was controlled using a digital controller connected to a type-K thermocouple, and relative humidity (RH) was monitored with a digital thermo-hygrometer (Model 30.5002, TFA Dostmann, Germany). RH of the drying air was maintained using an ultrasonic humidifier (Model HM20B, GADNIC, China). Two drying temperatures, 30 °C and 50 °C, were evaluated, both at 40% RH and 1.2 m/s air velocity, corresponding to low-temperature treatments commonly applied in the pasta industry to preserve the nutritional and sensory product attributes (Larrosa, 2014; Yu et al., 2018; Zhang et al., 2020).

Experimental procedure and moisture content determination

For each condition, 100 g of ribbon-type noodles were placed on a tray fitted with a metallic grid to allow air circulation on both sides. Mass loss was recorded every 10 minutes using an analytical balance until a constant mass was reached. Drying curves were expressed as dry basis moisture content (Xw, g water/g dry matter) as a function of time. Initial and final moisture contents were determined according to AACC Method 44-40 (AACC, 1984) by drying approximately 2.0–3.0 g of ground sample at 105 °C to constant mass and were expressed on both a wet basis (g water/100 g sample) and a dry basis (g water/g dry matter).
Before definitive experiments, a preliminary test was conducted for each condition to determine the minimum drying time required to reach an Xw compatible with product stability, defined as the equilibrium moisture content corresponding to $a_w \leq 0.63$ at 25 °C (Chirife and del Pilar Buera, 1994).

Mathematical modelling of drying curves

Moisture transport during drying was modelled on the assumption that water migration by molecular diffusion from the interior of the solid matrix to the surface, in accordance with Fick's second law for unsteady-state mass transfer (Geankoplis, 1983), consistent with the predominance of the falling rate period observed experimentally (Crank, 1975). Noodle strips were geometrically approximated as infinite flat plates, with moisture loss occurring exclusively through both flat faces. This approximation was validated by calculating aspect ratios relative to the half-thickness (se = 0.65 mm): the length-to-half-thickness and width-to-half-thickness ratios were 462 and 11.1, respectively, both exceeding the minimum criterion of 10 for neglecting edge effects (Doymaz, 2012). The following simplifying assumptions were adopted: (i) the effective diffusivity (D) is constant and independent of local moisture content; (ii) no external resistance exists at the solid-air interface, so surface moisture remains in equilibrium with the drying air; (iii) dimensional shrinkage is negligible; and (iv) the initial moisture distribution is uniform. Under these conditions, the analytical solution of Fick's second law reduces to a single-term Fourier series approximation (Crank, 1975):

$$X^* = (8/\pi^2) * \exp[-D * t * (\pi/2se)^2] \tag{3}$$

where X* = (Xt − Xw) / (Xo − Xw) is the dimensionless moisture content, with Xt, Xo, and Xw being the dry-basis moisture content at time t, initial, and equilibrium, respectively (g water/g dry matter); t is the drying time (s); se the half-thickness (m); and D the apparent diffusion coefficient (m^2/s).
Applicability of the single-term solution requires the long-time condition $M_t/M_\infty > 0.6$ (equivalent to $X^* < 0.4$), where:

$$M_t/M_\infty = (X_t - X_o)/(X_w - X_o) \tag{4}$$

This condition was verified for both drying temperatures prior to model application, and the critical time (t_critical) from which it is satisfied is reported in Table 2. The apparent diffusion coefficient, D, was estimated by nonlinear regression of experimental X* data against time, using exclusively the data from the falling rate period that satisfied the long-time condition, with OriginPro 2021 software (OriginLab Corporation, Northampton, MA, USA).

Statistical analysis
All sorption experiments were conducted in duplicate. Goodness of fit of the models was evaluated using the coefficient of determination (R^2), chi-square statistic (χ^2), and mean relative error (E%), calculated as:

$$E\% = (100/n)\sum|X_{exp} - X_{cal}|/X_{exp} \tag{5}$$

where X_{exp} and X_{cal} are the experimental and model-predicted values, respectively, and n is the number of data points. A fit was considered satisfactory when E% < 10%. Model parameters are reported together with their confidence intervals, and results are expressed as mean ± standard deviation. All data processing and regression analyses were performed using OriginPro 2021 software.

Results and discussion

Sorption isotherm modelling
The moisture desorption isotherms of pecan nut flour-enriched noodles were determined at 25 °C and 40 °C within a water activity range of 0.20–0.92 (Figure 1). In both cases, the isotherms exhibited a typical sigmoidal shape, characteristic of type II behaviour according to the BET classification, in which equilibrium moisture content increases monotonically with water activity, consistent with that reported for food products of mixed composition (Iglesias and Chirife, 1982, 1984; Prasantha, 2018). The GAB model showed the best overall fit at both temperatures, and the Oswin model also provided a

satisfactory fit, albeit with lower precision, particularly at 25 °C. Model parameters are presented in Table 1. The Xm values obtained were slightly above the range reported for conventional pasta (0.05–0.15 g water/g dry matter; Lagoudaki et al., 1993), attributable to the higher density of active adsorption sites provided by the protein matrix and phenolic content of pecan nut flour — a compositional feature that distinguishes this product from standard wheat pasta and is relevant for establishing accurate stability criteria. The decrease in Xm with temperature is consistent with the inhibitory effect of temperature on water adsorption discussed in Section 3.2. From a technological standpoint, Xm constitutes a practical reference below which deterioration reactions — particularly lipid oxidation, which is especially relevant given the high unsaturated fat content of pecan nut flour — are minimised (Labuza and Altunakar, 2020). Despite its lower fitting precision, the Oswin model was selected for equilibrium moisture content interpolation through its temperature-modified version (Chen and Morey, 1989).

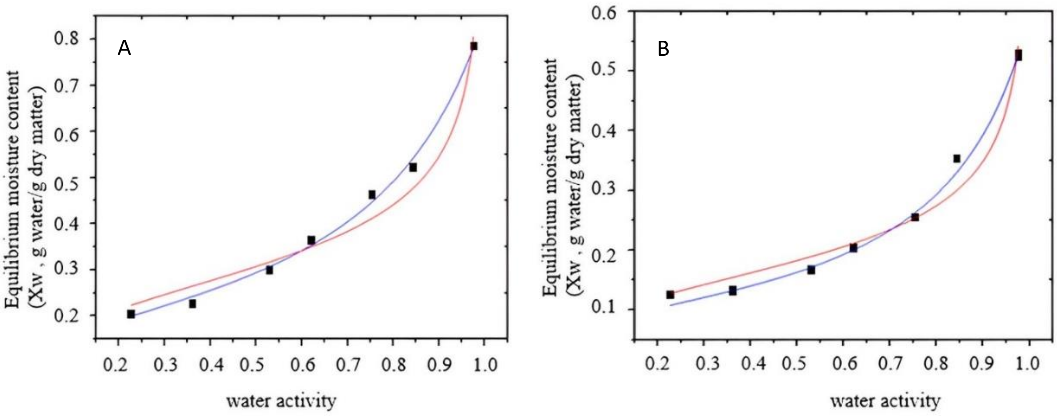

Figure 1. Desorption isotherm for noodles with pecan nut flour at 25 °C (A) and 40 °C (B) modelled with the the — GAB and — Oswin models; experimental data (■)

Table 1. Constants obtained from the GAB and Oswin models

Model	Parameter	25 °C	40 °C
GAB	Xm	0.188 ± 0.007	0.0996 ± 0.004
	C	31.09 ± 7.26	30.19 ± 13.03
	K	0.781 ± 0.009	0.833 ± 0.009
	R^2	0.99	0.99
	E%	3.18 ± 0.02	4.35 ± 0.05
Oswin	A	0.307 ± 0.011	0.182 ± 0.008
	B	0.261 ± 0.013	0.295 ± 0.016
	R^2	0.97	0.96
	E%	9.53 ± 0.05	7.02 ± 0.07

Effect of temperature on desorption isotherms

Figure 2 illustrates the effect of temperature on equilibrium moisture content. At a fixed a_w, X_w decreased with increasing temperature, while a constant moisture content, the corresponding aw was higher at elevated temperatures. This indicates that noodles stored at 25 °C exhibit greater hygroscopic capacity than those stored at 40 °C, confirming the inhibitory effect of temperature on water adsorption. In practical terms, storage at elevated temperatures reduces the risk of moisture absorption; however, this advantage must be balanced against the potential acceleration of temperature-dependent deterioration reactions, particularly relevant in products with high lipid and phenolic contents such as those studied here (Labuza and Altunakar, 2020).

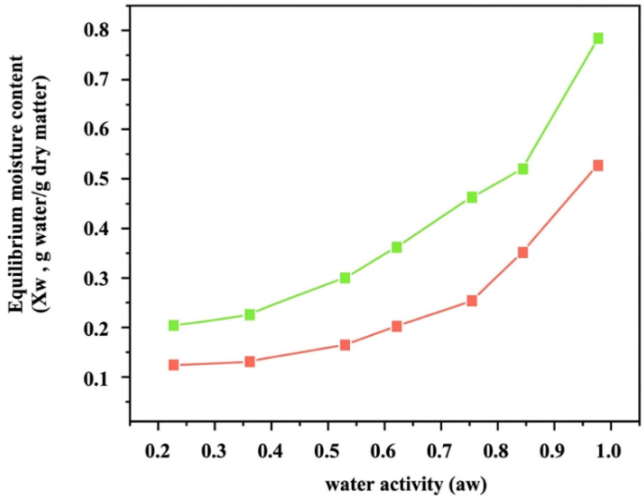

Figure 2. Average equilibrium moisture content of pasta expressed as g water/g dry matter for temperatures (-■-) 25 °C and (-■-) 40 °C

Determination of equilibrium moisture content for drying conditions

To account for the effect of temperature and enable interpolation at drying temperatures not experimentally evaluated, the modified Oswin model (Chen and Morey, 1989) was applied:

$$X_w = (A + B + T)[aw/(1 - aw)]^C \quad (6)$$

where A, B, and C are model parameters, and T is temperature (°C). Using this equation, the equilibrium moisture contents (Xw) of the isotherms were modelled as a function of water activity and temperature (Figure 3). Fitting parameters were A = 0.392, B = −0.006, and C = 0.282 (R^2 = 0.98). Relative humidity values were converted to water activity using aw = RH/100. The resulting equilibrium moisture contents were 0.082 g water/g dry matter at 50 °C (a_w = 0.4, condition 50-40) and 0.189 g water/g dry matter at 30 °C (a_w = 0.4, condition 30-40), which served as boundary conditions for subsequent modelling of the noodle drying curves.

Experimental drying curves

Figure 4 presents the drying curves obtained at 30 °C and 50 °C with 40% RH. The product did not exhibit an induction period, indicating that surface mass transfer conditions were established immediately. A short constant-rate period was followed by a predominant falling-rate period, suggesting that internal moisture diffusion controlled the drying process, consistent with behaviour reported for pasta with partial wheat flour substitution (Chigal et al., 2019). Moisture content decreased from an initial value of 0.55 g water/g dry matter to 0.15 g water/g dry matter at the end of drying. This value remained slightly above the equilibrium moisture content predicted by the modified Oswin model (0.13 g water/g dry matter at 30 °C). Nevertheless, the measured final water activity was below 0.65 under both drying conditions, confirming microbiological stability (Chirife and del Pilar Buera, 1994). This apparent discrepancy can be explained by the fact that the measured aw reflects the actual thermodynamic state of the dried product at the time of measurement, whereas the equilibrium value, derived from the isotherm model assumes full equilibration, which may not be reached due to kinetic limitations during the final stages of the falling-rate period. Therefore, although equilibrium was not fully attained, both drying conditions produced a microbiologically stable product within the established regulatory limits. This behaviour may be directly attributed to the incorporation of pecan flour, whose residual lipid fraction may partially coat starch granules and protein structures, reduce pore connectivity and limit internal moisture transport. As a result, the final drying stage becomes slower and more diffusion-limited.

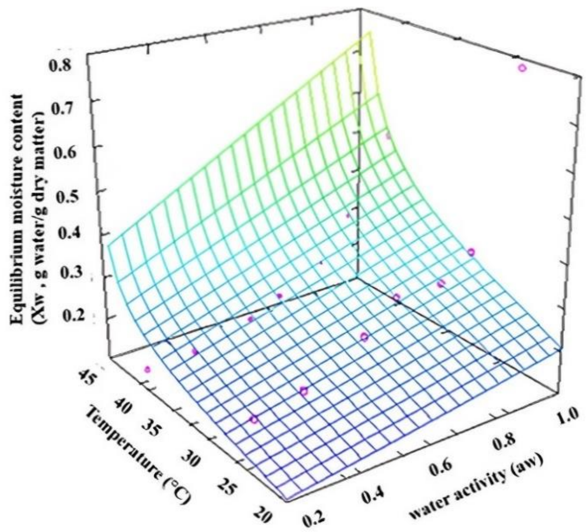

Figure 3. Modelling of equilibrium moisture contents Xw (g water/g dry matter) as a function of water activity and temperature

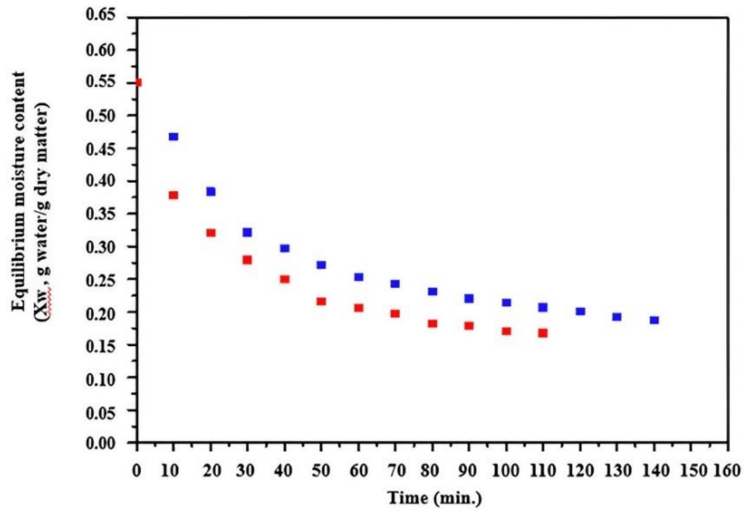

Figure 4. Drying curves of noodles showing the separation based on the observed drying periods (■ 30 °C – 40% RH; ■ 50 °C – 40% RH)

The drying time required to reach the established moisture content ranged between 110 and 140 minutes, being longer at 30 °C, consistent with the Arrhenius-type temperature dependence of effective diffusivity.

Drying curve modelling

The constant rate period was modelled by linear fitting of X* as a function of time according to X* = a + bt. Regression coefficients, the moisture content at the end of this period (Xvcte), and the transition time (tvcte) are presented in Table 2 for each condition (Larrosa et al., 2016). The falling-rate period was modelled using Eq. (3). The apparent diffusion coefficients obtained confirmed the expected increase in diffusivity with temperature, consistent with trends reported for composite noodles (Yebei et al., 2018). All values fall within the effective diffusivity range for low- and intermediate- moisture food products (10^{-9}–10^{-12} m²/s) and are comparable to those reported for corn pasta (2.8×10^{-11}–1.9×10^{-10} m²/s), durum wheat semolina pasta (3.0×10^{-13}–1.5×10^{-10} m²/s), and wheat flour pasta (2.50×10^{-12}–5.6×10^{-11} m²/s; Martínez-Navarrete et al., 1998). These differences may be attributed to the compositional characteristics of pecan flour. The high lipid content of pecan flour likely reduced moisture transport by partially blocking diffusion pathways and promoting a more compact internal structure. Additionally, the interaction between lipids, proteins, and starch may have increased matrix heterogeneity, producing greater resistance to internal water movement. The better model fit at 50 °C may reflect greater homogeneity of the diffusion process at elevated temperatures, whereas discrepancies at 30 °C may indicate additional transport mechanisms such as capillary diffusion or matrix contraction. Future work incorporating multi-term Fick solutions or variable diffusivity models could improve fitting accuracy. The critical time at which internal diffusion controlled the process was 60 min at 30 °C and 40 min at 50 °C. The earlier onset at higher temperature can be attributed to faster surface evaporation, which generates a steeper internal concentration gradient (Table 2). Figure 5 presents the experimental drying curves and model-predicted values for conditions (A) 30 °C–40% RH and (B) 50 °C–40% RH.

Table 2. Drying parameters for the constant rate and falling rate periods under each evaluated condition

		Condition	
Period	**Parameter**	**30-40**	**50-40**
Constant rate	Xvcte (g water/g d.m.)	0.32	0.29
	tvcte (min.)	30	20
	a (dimensionless)	0.546 ± 0.007	0.956 ± 0.097
	b (min^{-1})	−0.008 ± 0.0001	−0.0004 ± 0.0001
	R^2	0.99	0.84
Falling rate	D (m²/s)	$6.62 \times 10^{-12} \pm 1.63 \times 10^{-13}$	$1.80 \times 10^{-11} \pm 7.33 \times 10^{-12}$
	Critical time (min.)	60	40
	R^2	0.80	0.86

Final water activity and moisture content of the noodles

At 30 °C and 40% relative humidity, the product reached a water activity of 0.514 ± 0.002 and a moisture content of 10.39 ± 0.43 g water/100 g sample (11.6 ± 0.54 g water/g dry matter). Under the more severe drying condition (50 °C, 40% RH), lower values were obtained, with a final water activity of 0.463 ± 0.008 and a moisture content of 8.07 ± 0.42 g water/100 g sample (8.78 ± 0.50 g water/g dry matter). Both conditions satisfied the Argentine Food Code moisture limit (< 14% w/w; CAA, Chapter IX, 2018) and yielded water activity values below the critical microbiological stability threshold of 0.65 (Chirife and del Pilar Buera, 1994). The greater safety margin achieved at 50 °C is particularly relevant for a product with a high unsaturated lipid and phenolic content, since lower aw values more effectively limit not only microbial growth but also the oxidative and enzymatic deterioration reactions to which pecan-enriched pasta is specifically susceptible.

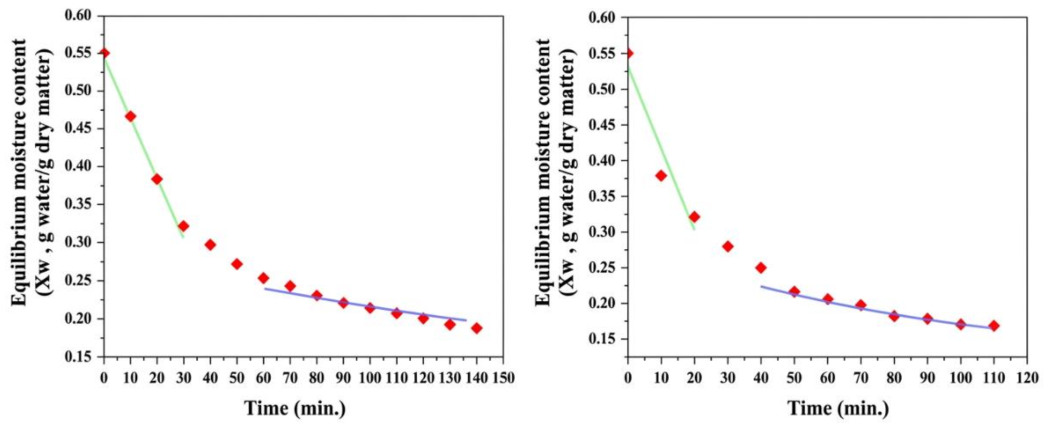

Figure 5. Drying curves (A) 30 °C–40% RH and (B) 50 °C–40% RH of the noodles: ■experimental data of the constant rate period (–) and diffusional model at long times (–)

Conclusion

The present study demonstrated, for the first time, that pasta formulated with a 1:1 wheat flour–pecan nut flour ratio is technologically feasible. The incorporation of pecan flour did not compromise the drying process, product stability, or final safety conditions, supporting its use as a functional ingredient for the production of value-added dry pasta from a nutrient-rich agro-industrial by-product. Desorption isotherms exhibited type II sigmoidal behaviour at both evaluated temperatures, with the GAB model providing the best fit (R^2 = 0.99; E% < 5%). Monolayer moisture values were higher than those reported for conventional pasta, reflecting the higher density of active water-binding sites attributable to the protein matrix and phenolic content of pecan nut flour — a compositional feature with direct implications for establishing accurate stability criteria for this novel product. Drying kinetics were governed by the falling rate period, with internal diffusion controlling moisture transport. The apparent diffusion coefficients (6.62×10^{-12} m²/s at 30 °C and 1.80×10^{-11} m²/s at 50 °C) are consistent with the structural influence of pecan flour's high unsaturated lipid content on reducing internal moisture mobility, a distinctive characteristic of this formulation relative to conventional pasta. Under both drying conditions, the final products complied with Argentine Food Code moisture requirements and presented water activity values below the microbiological stability threshold, confirming the safety and processability of the formulation. These results provide a quantitative basis for the industrial design of drying protocols and establish pecan nut flour as a viable ingredient for the development of nutritionally enriched, shelf-stable pasta products with added value along the nut oil production chain.

Author Contributions: GSM- Writing – Original Draft, Methodology, Investigation, Formal Analysis, Visualisation; NGC Writing – Original Draft, Methodology, Investigation, Formal Analysis, Visualisation; VJL - Writing – Review & Editing, Supervision, Conceptualisation. All authors reviewed the results and approved the final version of the manuscript.

Funding: This research was funded by the collaboration of the University National of Entre Ríos. Experiments were funded as part of the project PID 9189.

Acknowledgements: The authors would like to thank the Institute of Food Science and Technology of Entre Ríos (ICTAER-CONICET) and the National University of Entre Ríos (UNER) for the institutional and technical support provided during this research.

***Conflicts of Interest*:** The authors declare no conflict of interest.

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