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Modeling of Drying and Adsorption Isotherms of the Fish Feed

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ABSTRACT

The aim of this work was to study the drying curves and equilibrium isotherms of extruded fish feed. The drying curves were determined at air temperatures of 50, 60, 70 and 80°C and airflow velocities of 1.5, 2.5 and 3.5 m/s). The equilibrium isotherms of relative humidity of air were obtained between 10 and 80% at 30, 40, 50, and 70°C. The experimental data were fitted for non-linear regression by using STATISTICA® to the models reported in the literature. The results showed that the drying curves and the equilibrium isotherms were significantly influenced by variations of the air temperature in a similar way to solid materials as described in the literature. The statistical results for models of Page and Peleg showed that the fitting of the experimental drying curve and isotherm data were satisfactory.

Key words: extruded fish feed, adsorption isotherms, equilibrium moisture

INTRODUCTION

Aquaculture is becoming an important productive component of the food safety in animal protein. It has been well developed in Brazil and in the world in the last decades. Factors such as the decrease of the natural fishing, environmental restrictions, governmental incentives, and medical recommendations for a healthier diet have contributed to an increase in the demand of fish meat. The success of the aquaculture depends on the quality and final product cost to the consumers. According to Magliano (2007), the feed cost may correspond up to 70% of the fish meat production cost of a fish breeding station. With an aim to make aquaculture an economically and environmentally sustainable activity, attempts have been made to develop the fish feed formulation of low cost with minimum environmental impact (Kubitza, 1997; El-Sayed, 1999; Faria et al., 2001a and 2001b; Al-Ruqaie, 2008). Other studies have been conducted to improve the nutritional and sensorial aspects of the feeds aiming the appropriate consumption and a greater gain of weight for animals (Kubitza, 1995; Souza and Hayashi, 2003; Teixeira et al., 2007). Sussel (2008) reported that the large increase in the fish breeding was due to the development of pelletized feed and later of extruded feed. The high temperature and pressure conditions are used in extruded feed to promote the suitable physical-chemical modifications in the ingredients and improve the feeding efficiency of fishes. Furthermore, the extruded feed can float on the

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water, reducing the nutrient leaching causing environmental impact (FAPEAM, 2009; Botaro, 2007).

After the extrusion, the feed is dried to a moisture level that prevents the proliferation of microorganisms to ensure its quality during handling and transportation. Drying requires large amount of energy and may increase the production costs, especially if the equipment operating conditions are not appropriate. Nevertheless, drying must be optimized for the production of a good quality product at an accessible cost to the consumer. This requires knowledge of the drying kinetics of feed, because it influences the design, operation, and efficient control of the dryer.

The drying kinetics describes the moisture variation profile of the material with time. Models that represent the drying curves have been proposed based on the variables that may interfere on product drying. According to Brooker et al. (1992), empirical and semi-empirical models are the best options to estimate the moisture variation of materials. Some examples of application of these models were identified in the analyses of drying of urucum (*Bixa orellana L.*) using the Lewis, Page, Thompson, Overhults, Brooker, Henderson and Motta Lima (Guedes and Faria, 2000), of beans (*Phaseolus vulgaris* L.) using the correlations of Wangh and Singh, Verna, Thompson, Page, Midilli, Logarithmic, Henderson and Pabis, Henderson and modified Pabis, two-term exponential (Corrêa et al., 2007) and of fertilizers using the Lewis, Brooker, Page, Overhults, Henderson and Henderson (Arruda, 2008) models. The utilization of these models requires the knowledge of the drying data and system operation conditions. When these data are not available, dimensionless moisture data of the product are experimentally obtained as a function of time.

The concern with feed quality and cost reduction is fundamental for the production and commercialization of the feed. Therefore, both the feed producer and consumer need to know the adsorption behavior in several ambient conditions to prevent losses during the storage and supply of the feed of acceptable quality to the market.

The moisture adsorption by the product on the shelf or after opening its package is related to the equilibrium moisture defined as the amount of corresponding moisture to the equilibrium between the water vapor pressure in the material and in the ambient. The correlation between the equilibrium moisture and water activity at a given temperature is called equilibrium isotherm (Karathanos et al., 1996). The equilibrium isotherms of certain products may be determined by using experimental data or estimated data by non-linear models found in the literature. As such, the BET, GAB, and Oswin models are the most common ones for accuracy and large application (Fadini et al., 2006). Recently, these and other models have been investigated by Gomes *et al.* (2002) in a study of adsorptions isotherms of acerola pulp applying BET, GAB, Oswin, and Smith, by Ascheri et al. (2007) in a study of the moisture adsorption of pre-gelatinized jabuticaba bagasse and rice applying Chung-Pfost, Halsey, GAB, BET, Oswin and modified Henderson, and by Alexandre et al. (2007) in the study of the adsorption isotherms of powdered pitanga applying GAB, Oswin, and Peleg correlations. However, the use of these models depends on the knowledge of the water activity or relative humidity of air, the drying temperature, and material-specific parameters. In the absence of these data, the equilibrium moisture data must be obtained in laboratory by gravimetry or hygrometry (Barrozo et al., 2000; Mujumdar, 1987).

Tithed aim of this work was to investigate the drying kinetics and adsorption moisture, and their fittings for mathematical models as tools for the design and simulation of industrial feed dryers.

MATERIALS AND METHODS

The feed samples were provided by a company in northern Paraná with spherical pellets (diameter 6 to 8 mm; initial moisture 0.30 (d.b.) The moisture was determined by drying at 105°C ± 2ºC for 24 h.

Drying kinetics data of feed

The drying data of the feed were obtained by using a bench scale convective dryer, as shown in Figure 1.

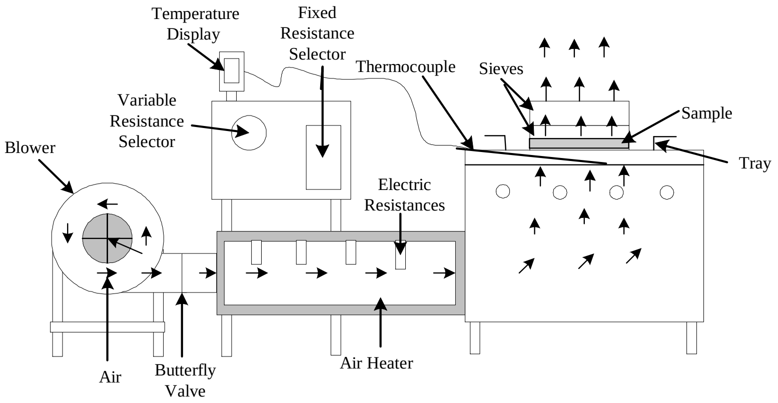

Figure 1 - Convective dryer scheme.

In this dryer, each assay was carried out with approximately 80 g of feed, amount sufficient to cover the drying bed constituted by two sieves with meshes of 200 and 270. Thermocouples were distributed under the dryer tray. The flow and air temperature were monitored by using an anemometer and a psychrometer, respectively. The stabilized drying conditions were registered with the ambient air data. Afterwards, the sieves with sample were set on the dryer tray and the drying was started. The sample-sieves set was weighed in an analytical balance every 30 s during the first 6 min due to the high evaporation in the beginning. Later, data were collected every minute until it completed the first hour of processing. In the next hour, the data were collected every 2 min, due to the low moisture variation in this period. The drying conditions were reregistered with the ambient air data. At the end, the feed dry mass was determined in the oven at 105ºC ±2 ºC for 24h.

Equation 1 was used to determine the solid moisture profile.

$$X_s = \frac{m - m_{ss}}{m_{ss}} \qquad (1)$$

Where X_s is the solid moisture (d.b.), m is the feed mass at each drying time (g) and m_{ss} is the feed sample mass in the oven (g).

The drying rate, N, was determined by Equation 2.

$$N = \frac{\Delta X_s}{\Delta t} \qquad (2)$$

Where N is drying rate (1/s), ΔX_s is the variation of the solid moisture (d.b.) and Δt is the time variation.

The previous procedures were repeated at 50, 60, 70, and 80°C under airflow velocities of 1.5, 2.5, and 3.5 m/s.

The dimensionless moisture ratio of the extruded feed, *MR*, at the different drying conditions was calculated by using Equation 3.

$$MR = \frac{X_s - X_e}{X_i - X_e} \qquad (3)$$

Where X_s is the solid moisture (d.b.), X_e is the solid equilibrium moisture (d.b.) and X_i is the solid initial moisture (d.b.).

Fitting of the extruded feed drying curves

The extruded feed drying curves were fitted to the mathematical models of Wang and Singh, Thompson and Page as follow.

- Wang and Singh (Wang and Singh, 1978)

$$MR = 1 + at + bt^2 \qquad (4)$$

- Thompson (Thompson et al., 1968)

$$MR = \exp((-a - (a^2 + 4bt)^{0,5})/2b) \qquad (5)$$

- Page (Page, 1949)

$$MR = \exp(-kt^n) \qquad (6)$$

Where a, b, c, n are model constants, k is the drying constant, t is the drying time (s), and MR is the dimensionless product moisture ratio.

The regression analysis was performed using computer software, namely, STATISTICA® version 5.0. Regression work was done based on the Quasi-Newton method and convergence criterion of 0.0001.

The parameters used to evaluate the curve fittings were the coefficient of determination (R^2), the relative mean deviation, P, and the estimated error, EE. Values of P and EE were calculated by Equations 7 and 8, respectively.

$$P = \frac{100}{n}\sum_{i=1}^{n}\frac{\left|MR_{exp} - MR_{calc}\right|}{MR_{exp}} \tag{7}$$

$$EE = \sqrt{\frac{\sum\left(MR_{exp} - MR_{calc}\right)^2}{DOF}} \tag{8}$$

Where MR_{exp} is the experimental feed moisture ratio, MR_{calc} is the calculated feed moisture ration is the number of experimental observations, and DOF is the number of degrees of freedom (number of observations minus the number of observed parameters of the model).

Equilibrium isotherms

The classical gravimetric method was used to obtain the solid adsorption equilibrium isotherms, as follow.

The feed samples were initially dried in an oven for 24 h at 105°C ± 2°C to determine the dry mass and guarantee the moisture adsorption by using salt solutions. The samples were stored in basket-like recipients that were weighed empty and with sample in an analytical balance. The determination of the mass of the empty recipient facilitated the weighting of the samples in the following steps. Afterwards, saturated samples were prepared and warmed, resulting relative humidity of air between 10 and 80%, as reported by Arnosti Jr. (1997) for LiCl, CH_3CO_2K, $MgCl_2.6H_2O$, $NaNO_2$, NaCl, and KCl. The solutions were prepared in duplicate with the salt and water masses informed by Perry and Chilton (1980). The samples and saline solutions were kept in closed jars to prevent their direct contact as shown in Figure 2.

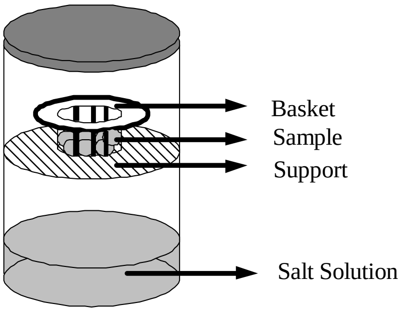

Figure 2 - Experimental jar

After storing the samples and solutions, the jars were kept in the oven at 50°C. The samples were weighed in the baskets at regular intervals until that the mass was constant, indicating that hygroscopic equilibrium was reached. The equilibrium moisture (d.b.), X_e, was calculated by Equation (1).

These procedures were repeated for new feed samples at 30, 40, and 70°C.

Fitting of the equilibrium isotherms

The extruded feed equilibrium isotherms were fitted to the mathematical models of Peleg, Henderson–Thompson and Keey, as follow.

- Peleg model (Peleg, 1993)

$$X_e = a a_w{}^{b} + c a_w{}^{d} \tag{9}$$

- Henderson -Thompson model (Thompson et al., 1968)

$$X_e = \left[\frac{\ln(1-a_w)}{-a(T+c)}\right]^{1/b} \tag{10}$$

- KEEY model (Keey, 1991)

$$X_e = \frac{a}{1 + bT^3 \ln\left(\frac{1}{a_w}\right)} \qquad (11)$$

Where a, b, c, d are model parameters, a_w is the water activity, and T is the feed drying temperature (K).

The regression analysis was performed using computer software STATISTICA® version 5.0. Regression work was done based on the Rosenbrook and Quasi-Newton and convergence criterion of 0.0001.

The parameters used to evaluate the model fitting were the coefficient of determination, R^2, the test F, F, and the relative mean deviation, P. As such, the test F was calculated by Equation 12.

$$F = \left(\frac{(MR_{calc}{}^{2})_{mean}}{\left(MR_{exp} - MR_{calc}\right)^{2}{}_{mean}} \right) \qquad (12)$$

RESULTS AND DISCUSSION

Extruded fish feed drying curves

Figures 3 and 4 show the solid drying curves at different air temperatures and constant airflow velocity.

The profiles of the feed drying curves shown in Figures 3, 4, and 5 are characteristic of solid materials. It was also observed that the increase of the air temperature resulted in larger sample drying rates, which were less pronounced in the beginning of drying.

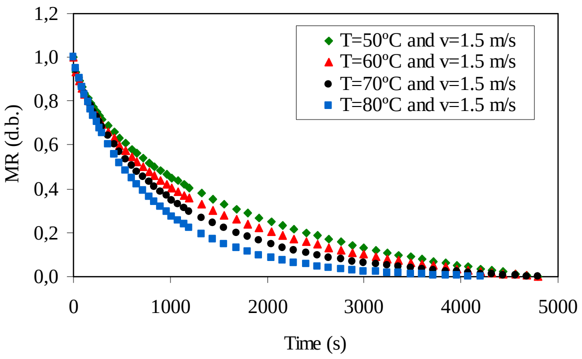

Figure 3 - Influence of air temperature (v=1.5 m/s) on fish feed drying.

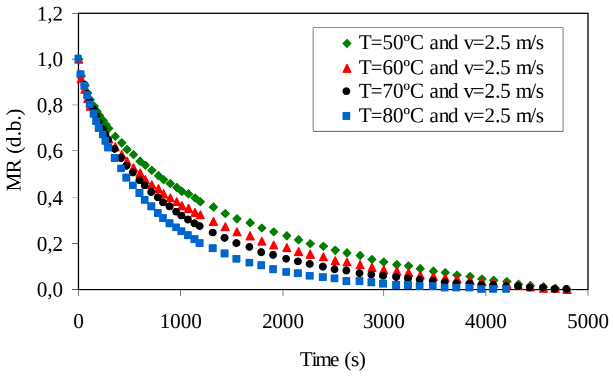

Figure 4 - Influence of air temperature (v=2.5 m/s) on fish feed drying.

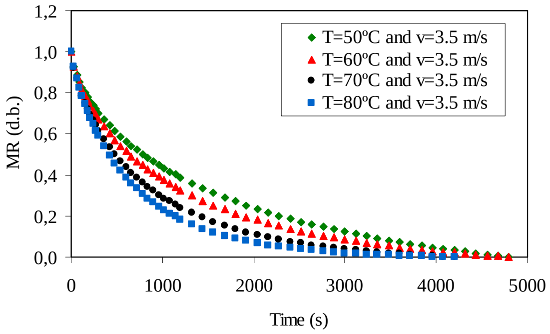

Figure 5 - Influence of air temperature (v=3.5 m/s) on fish feed drying.

Figures 6 and 7 show the solid drying curves at different airflows velocities and constant air temperature.

The feed drying curves shown in Figures 6 and 7 were slightly influenced by the airflow velocity and more pronounced at higher temperatures. At 50°C, the curves practically overlapped, while at 80°C, the influence of the temperature was more pronounced.

The feed drying curves shown in Figures 3 to 7 were as reported by Mrkic et al. (2007) for the drying of broccoli and by Luz et al. (2009) for the drying of soybean meal.

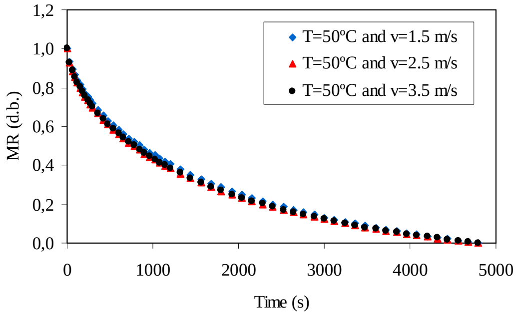

Figure 6 - Influence of airflow velocity (T=50ºC) on fish feed drying.

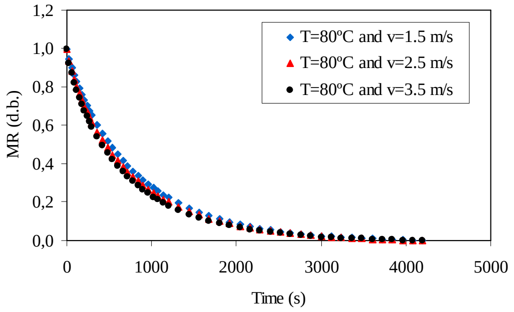

Figure 7 - Influence of airflow velocity (T=80ºC) on fish feed drying.

Considering the effect of the air temperature and airflow velocity on feed drying, the model parameters of Equations 4 to 6 were estimated and related by statistical parameters (R^2, P, and EE) that were analyzed for the different drying conditions.

As observed in Table 1, the Thompson and Page models presented satisfactory results with the best statistical fitting values. However, the Page model stands out for its greatest R^2 and lowest P and EE values, demonstrating to be the most suitable one to estimate the extruded feed drying curve. The fitting results of this model are shown in Figures 8, 9 and 10.

Table 1 - Estimated parameters of the models of the drying curves of extruded fish feed.

Models	**Drying conditions**		**Model parameters**		**Fitting parameters**		
	T (°C)	**v (m/s)**	***a***	***b***	***R^2***	***P***	***EE***
Wang and Singh	50	1.5	-0.00054	$7.47121.10^{-8}$	0.920	69.72	0.0844
Wang and Singh	50	2.5	-0.00056	$7.94652.10^{-8}$	0.901	84.42	0.0929
Wang and Singh	50	3.5	-0.00056	$7.87756.10^{-8}$	0.905	76.85	0.0913
Wang and Singh	60	1.5	-0.00059	$8.48285.10^{-8}$	0.905	97.48	0.0935
Wang and Singh	60	2.5	-0.00062	$9.24961.10^{-8}$	0.854	137.17	0.1116
Wang and Singh	60	3.5	-0.00061	$9.12356.10^{-8}$	0.879	154.08	0.1039
Wang and Singh	70	1.5	-0.00064	$9.66913.10^{-8}$	0.896	214.48	0.1000
Wang and Singh	70	2.5	-0.00066	$1.02270.10^{-7}$	0.857	292.25	0.1161
Wang and Singh	70	3.5	-0.00075	$1.33356.10^{-7}$	0.848	250.35	0.1105
Wang and Singh	80	1.5	-0.00076	$1.35585.10^{-7}$	0.899	420.71	0.1012
Wang and Singh	80	2.5	-0.00079	$1.42869.10^{-7}$	0.855	473.64	0.1171
Wang and Singh	80	3.5	-0.00081	$1.48412.10^{-7}$	0.821	594.13	0.1280
	T (°C)	**v (m/s)**	***a***	***b***	***R^2***	***P***	***EE***
Thompson	50	1.5	-24.0895	$1.57090.10^{-1}$	0.989	41.25	0.0317
Thompson	50	2.5	-19.2253	$1.50518.10^{-1}$	0.989	45.15	0.0311
Thompson	50	3.5	-20.2168	$1.52220.10^{-1}$	0.989	44.41	0.0310
Thompson	60	1.5	-23.0511	$1.65142.10^{-1}$	0.993	46.07	0.0251
Thompson	60	2.5	-14.7191	$1.51697.10^{-1}$	0.992	54.45	0.0256
Thompson	60	3.5	-18.9435	$1.61734.10^{-1}$	0.994	51.60	0.0236
Thompson	70	1.5	-30.0880	$1.96186.10^{-1}$	0.997	50.32	0.0158
Thompson	70	2.5	-21.9348	$1.82583.10^{-1}$	0.997	58.75	0.0165
Thompson	70	3.5	-18.2384	$1.80992.10^{-1}$	0.996	55.22	0.0182
Thompson	80	1.5	-47.6436	$2.61564.10^{-1}$	0.999	43.58	0.0089
Thompson	80	2.5	-29.5501	$2.24693.10^{-1}$	0.999	56.00	0.0109
Thompson	80	3.5	-24.7011	$2.17210.10^{-1}$	0.999	63.46	0.0111
	T (°C)	**v (m/s)**	***k***	***n***	***R^2***	***P***	***EE***
Page	50	1.5	0.00296	$8.14313.10^{-1}$	0.994	34.97	0.0226
Page	50	2.5	0.00372	$7.90533.10^{-1}$	0.995	36.34	0.0213
Page	50	3.5	0.00353	$7.96408.10^{-1}$	0.995	36.09	0.0213
Page	60	1.5	0.00329	$8.15995.10^{-1}$	0.997	35.95	0.0160
Page	60	2.5	0.00524	$7.63735.10^{-1}$	0.997	38.96	0.0152
Page	60	3.5	0.00413	$7.95080.10^{-1}$	0.998	37.45	0.0138
Page	70	1.5	0.00287	$8.55210.10^{-1}$	0.999	35.55	0.0081
Page	70	2.5	0.00397	$8.19738.10^{-1}$	0.999	37.45	0.0075
Page	70	3.5	0.00491	$8.02480.10^{-1}$	0.999	35.11	0.0087
Page	80	1.5	0.00232	$9.13796.10^{-1}$	0.999	29.84	0.0041
Page	80	2.5	0.00344	$8.67847.10^{-1}$	0.999	32.02	0.0040
Page	80	3.5	0.00412	$8.50077.10^{-1}$	0.999	32.29	0.0035

The Page model has been largely used to represent the drying curves of food and agricultural product, as shown in Figures 8, 9 and 10. These studies include the drying of urucum by Guedes and Faria (2000), green malt by Santosi et al. (2001), popcorn and barley by Corrêa et al. (2001) and beans by Corrêa et al. (2007).

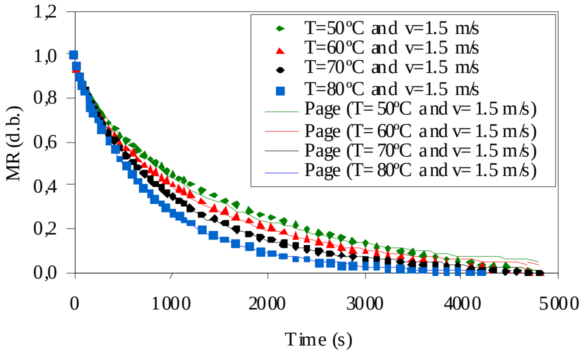

Figure 8 - Experimental and predicted dimensionless moisture of fish feed drying at airflow velocity of 1.5 m/s.

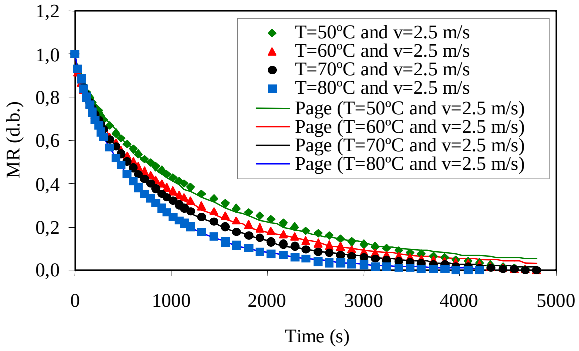

Figure 9 - Experimental and predicted dimensionless moisture of fish feed drying at airflow velocity of 2.5 m/s.

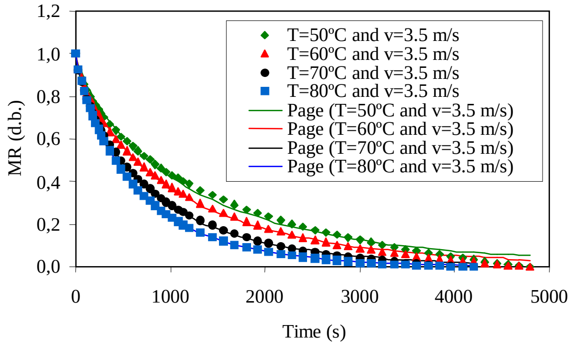

Figure 10 - Experimental and predicted dimensionless moisture of fish feed drying at airflow velocity of 3.5 m/s.

Adsorption equilibrium isotherms of extruded fish feed

The mean experimental results of water activity (a_w) and the respective equilibrium moisture values (X_e) of extruded fish feed at 30, 40, 50, and 70ºC are shown in Table 2 and in Figure 11.

Table 2 and Figure 11 showed that the equilibrium moisture of the extruded fish feed had higher increase for water activity values between 0.597 and 0.834. The air temperature influenced the equilibrium moisture of the feed, leading to an increase in the moisture adsorption. As such, the maximum value obtained was 0.20 (d.b.) at 30°C, 0.695 (d.b.) at 40°C, 1.542 (d.b.) at 50°C, and at 2.3 (d.b.) at 70°C. The great variation of the feed moisture showed that handling care was necessary, mainly in ambient with relative humidity of air higher than 0.6.

Figure 11 showed that the adsorption equilibrium isotherms of the feed had sigmoid form. This behavior is characteristic of food products, such as macaroni, flour, bread, cracker, egg albumin (Geankoplis, 1993), and chitin (Moraes et al., 2005).

Table 2 - Experimental equilibrium moisture (Xe) for the different water activity values (a_w) of feed at 30, 40,50 and 70°C.

Saline solutions	**a_w 30°C**	**X_e 30°C**	**a_w 40°C**	**X_e 40°C**	**a_w 50°C**	**X_e 50°C**	**a_w 70°C**	**X_e 70°C**
LiCl	0.113	0.000	0.112	0.000	0.111	0.009	0.109	0.000
CH_3CO_2K	0.216	0.000	0.204	0.004	0.192	0.026	0.168	0.080
$MgCl_2.6H_2O$	0.324	0.036	0.318	0.077	0.312	0.116	0.300	0.140
$NaNO_2$	0.635	0.039	0.616	0.079	0.597	0.122	0.559	0.250
NaCl	0.750	0.101	0.748	0.393	0.746	1.114	0.742	1.800
KCl	0.834	0.201	0.818	0.695	0.802	1.542	0.770	2.300

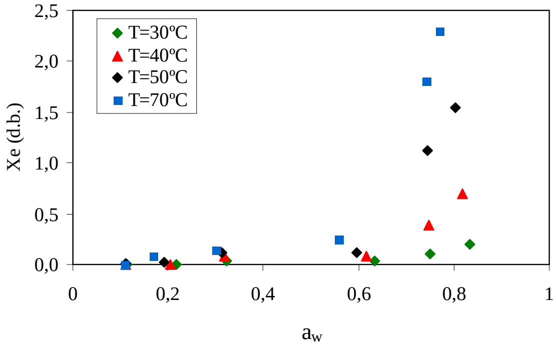

Figure 11 - Experimental adsorption isotherms of extruded fish feed at different temperatures.

Table 3 showed the estimated parameters for the models of Equations 9 to 11 and their respective statistical parameters (R^2, P, and EE) that were analyzed for the different drying conditions.

As observed in Table 3, the Peleg and Henderson-Thompson models showed satisfactory results according to statistical fitting parameters. However, the Peleg model stands out for its greatest R^2 and F and lowest P values, being the most appropriate one to estimate the equilibrium moisture of extruded fish feed. The fitting results of this model are presented in Figure 12.

Table 3 - Estimated parameters of the models of the equilibrium isotherms of extruded fish feed.

Models	**Drying Conditions**	**Model Parameters**				**Fitting Parameters**		
	T (°C)	***a***	***b***	***c***	***d***	***R²***	***F***	***P***
Peleg	30	0.920	9.883	0.059	1.068	0.981	62.55	12.62
Peleg	40	3.180	7.903	0.060	0.636	0.988	73.49	92.98
Peleg	50	3.092	6.400	3.452	6.400	0.981	43.85	67.69
Peleg	70	13.819	6.789	-0.226	6.789	0.995	328.75	33.77
Henderson-Thompson	30	-0.096	0.387	-337.904		0.964	70.56	17.94
Henderson-Thompson	40	0.350	0.326	-307.689		0.981	65.08	35.67
Henderson-Thompson	50	0.100	0.333	-309.307		0.973	34.30	68.32
Henderson-Thompson	70	0.100	0.288	-331.625		0.995	178.34	35.23
Keey	30	90161.470	0.100			0.920	15.13	21.20
Keey	40	366606.900	0.100			0.880	11.48	340.70
Keey	50	1009593.00	0.100			0.860	9.26	419.07
Keey	70	2075025.00	0.100			0.864	2.44	376.00

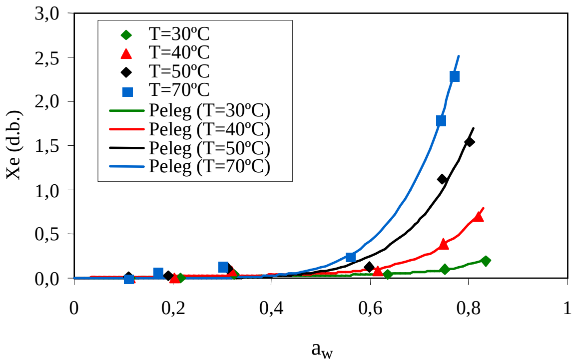

Figure 12 - Experimental and predicted adsorption isotherms of fish feed drying at different temperatures.

As shown in Figure 12, fitting equilibrium isotherms to the Peleg model also presented satisfactory results for Bartlett pear (Park et al., 2001), texturized soybean protein (Cassini, 2004) and dehydrated leaves of coriander (Lima et al., 2007).

CONCLUSIONS

The drying air temperature significantly affects the drying curves of extruded fish feed, while the effect of airflow velocity was limited.

The statistical analyses showed that the variation of the moisture content of extruded fish feed could be represented by the Page model which has been very efficient in the study of drying phenomena of several food products in the literature.

The equilibrium isotherms of extruded fish feed had sigmoid form, as has been frequently reported for food products in the literature.

The feed equilibrium isotherms were little influenced by air temperature variations for water activity values between 0 and 0.6, but for water activity over 0.6 the equilibrium isotherms could reach 2.3 (d.b.) in the evaluated conditions.The fitting of the equilibrium isotherms of the feed to the Peleg model presented the best statistical results and proved to be the most suitable one to predict the equilibrium moisture of extruded fish feed.

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