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Sorption isotherms of ingredients and diets for poultry

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Aditional Keywords

Storage.
Water activity.
Conservation.
Deterioration.
Moisture.

SUMMARY

This study aimed to determine sorption isotherms of ingredient and poultry diet. The samples were encapsulated in capsules and dehydrated by oven-drying in a desiccator for more than 24 hours. The samples were transferred to desiccator containing water in the base and placed in the oven, with one sample of each material being removed at incremental intervals. The sample was weighed and for determination of water activity and for dry matter. The moisture and water activity data were evaluated by eight mathematical models. The GAB mathematical model fitted the experimental data to constitute the isotherm for each material. Type II sorption isotherms were found, except for BHT: demonstrated values that did not fit the isotherm determination. The hygroscopic behavior of the ingredients were: L- threonine, limestone, BHT, DL- methionine, L-valine, L- tryptophan, phosphate, kaolin, vitamin supplement, salt, mycotoxin deactivator, pelleted rooster diet, mash rooster diet, mash layer diet, pelleted layer diet, corn, bacitracin zinc, vitamin mineral supplement, phytase, rice bran, wheat bran, mineral supplement, soybean meal, coccidiostat, L- Lysine HCl and choline chloride. Ingredients and diets have different hygroscopic behavior: can lead to deterioration and low accuracy in nutritional values of diet, since formulation is based on as-is fed basis.

Isotermas de sorción de ingredientes y dietas para aves de corral

RESUMEN

Este estudio tenía como objetivo determinar las isotermas de absorción de los ingredientes y la dieta de las aves. Las muestras se encapsularon en cápsulas y se deshidrataron por secado en horno en un desecador durante más de 24 horas. Las muestras se transfirieron al desecador que contiene agua en la base y se colocaron en el horno, retirándose una muestra de cada material a intervalos incrementales. La muestra se pesó y para la determinación de la actividad del agua y la materia seca. Los datos de humedad y actividad del agua fueron evaluados por ocho modelos matemáticos. El modelo matemático del GAB ajustó los datos experimentales para constituir la isoterma de cada material. Se encontraron isotermas de sorción de tipo II, excepto para el BHT: valores demostrados que no encajaban en la determinación de la isotermia. El comportamiento higroscópico de los ingredientes fue: L-treonina, piedra caliza, BHT, DL-metionina, L-valina, L-triptófano, fosfato, caolín, suplemento vitamínico, sal, desactivador de micotoxinas, dieta del gallo en pellets, dieta del gallo en pellets, dieta de la capa de puré, dieta de capas peletizadas, maíz, bacitracina-zinc, suplemento vitamínico mineral, fitasa, salvado de arroz, salvado de trigo, suplemento mineral, salvado de soja, coccidiostático, L-lisina HCl y cloruro de colina. Los ingredientes y las dietas tienen diferentes comportamientos higroscópicos: pueden llevar a un deterioro y a una baja precisión en los valores nutricionales de la dieta, ya que la formulación se basa en su estado.

Palabras Clave

Almacenamiento.
Actividad de agua.
Conservación.
Deterioro.
Humedad.

Information

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INTRODUCTION

The storage and use of ingredients and diets for poultry is not always done in conditions that are favorable to their conservation. In order to improve the efficiency of the ingredients it is important to understand how they interact with the environment, especially in terms of water flow (Gabbi, Cypriano & Piccinin 2011, pp. 784-793).

The presence of water in diet occurs in two forms: in bound form, with restricted mobility, and in free form, which is water available for physical, chemical and microbiological reactions and subject to interaction with the environment, which is expressed by the water activity (a_w), where the total moisture is determined by the sum of these two forms of water. This moisture is not static, since the ingredients adsorbs or desorbs moisture from the environment in which it is stored, depending on the temperature and relative air humidity.

Therefore, the water present in ingredients and diets is a risk factor for in-storage quality and conservation, since it can lead to deterioration by fungal development and mycotoxin production. Furthermore, it hinders handling and transportation and dilutes the total nutrients, proportionally reducing nutritional value of the product and consequently the animal performance and productivity (Coradi 2010, p. 191).

Understanding the interaction of ingredients with the environment and their hygroscopic behavior is of fundamental importance (Gabbi, Cypriano & Piccinin 2011, pp. 784-793). Sorption isotherms represent important mathematical models for predicting product interaction with the environment, allowing one to determine the ideal humidity of the environment where the ingredient is stored in order to avoid the risk of microbiological growth and loss of nutritional value. Moreover, it is necessary to consider its moisture content for the precise dosage of ingredients used in poultry diet formulations (Park, Cornejo & Fabbro 2008, pp. 83-94).

The characteristics of sorption isotherms demonstrate a material's ability to absorb water from or release water into the surrounding environment, when placed in atmospheres with controlled relative humidity at a given temperature (Costa 2010, p. 203).

Therefore, in order to provide information to support the adequate storage of ingredients and reduce risks of errors in diet formulations, this study aimed to determine the sorption isotherms of the ingredients commonly used in layer breeder diets.

MATERIAL AND METHODS

The experiment was conducted at the Physical-Chemical Analysis Laboratory of *Embrapa Swine* and *Poultry*, Concórdia/SC, and the samples were taken from ingredients at the Embrapa's animal feed mill.

In order to obtain the isotherms, the main ingredients that constitute diet for layer breeder were used: corn, soybean meal, wheat bran, rice bran, mycotoxin deactivator, salt, choline chloride, dicalcium phosphate, calcitic lime, L- Lysine, kaolin, BHT, bacitracin zinc, phytase, coccidiostat, DL- methionine, L- valine, L-threonine, L- tryptophan, mineral supplement, vitamin supplement, vitamin mineral supplement, and rooster and layer diet (mash and pelleted forms).

During the experiment period the temperature (T°C) and relative humidity (RH%) of the medications storeroom and bagged ingredients storage area of the diet production facility were monitored using a data logger (Test Equipment – 174 H), programmed to take measurements every 30 minutes. Weekly samples of three ingredients and diets (approximately 100 grams) were taken and stored in plastic containers.

A sample of each ingredient and of the animal diets were analyzed in as-sampled condition to determine the water activity (a_w) and moisture content (M%) on an as-fed basis. Eight samples of each material, weighing approximately five grams, were packaged in purpose-designed plastic capsules for the a_w equipment and subjected to dehydration in a desiccator containing silica, in a vacuum, and at a temperature of 30°C for more than 24 hours in a model 002 CB oven. After that period, the samples were transferred to a desiccator containing water in the base and they were then placed into the oven again, and one sample of each material was removed at incremental intervals of 0, 1, 3, 5, 7, 8: 30; 24 and 26 hours from the start. Once removed from the oven, the sample was weighed on an analytical scale and then placed in the measurement chamber of the equipment for a_w determination. Once the a_w was determined, the samples were subjected to oven drying for 12 hours at 105°C and weighed again in order to ascertain the M%. The value was expressed on an as-fed basis and dry matter basis. This procedure was repeated three times for each ingredient sample. The RH% and T°C inside the desiccators were monitored using a data logger (Test Equipment – 174 H).

The a_w and M% data for the samples subjected to humidification were evaluated using the GAB, BET, Halsey, Kunh, Henderson, Oswin, Mizrahi and Smith mathematical models, supported by the tool SOLVER (available on Microsoft Excel), establishing the isotherm for each of the ingredients. The GAB – Guggenheim-Anderson-de Boer mathematical model (nonlinear regression analysis – equation 01) was the only model to which the experimental data fitted in order to constitute the sorption isotherm. This model was chosen based on the highest determination coefficient value (R^2).

Equation 01: $M\% = m_0.c.k.a_w \ / \ [(1-k.a_w).\ (1+(c-1).k.a_w)]$

Where: M% - moisture content; m_0 - monolayer; a_w - water activity; c and k – constants.

RESULTS

The moisture content and water activity data for the ingredient in sampled condition at the feed mill are presented in **Table I**. It could be verified that the mash and pelleted poultry diets, corn, soybean meal, wheat bran, rice bran and BHT presented heightened moisture levels.

The elevated relative humidity in the feed mill during the winter period might explain the heightened water activity and moisture content values (as-fed basis) of the ingredients and of the diets in sample condition (**Table II**).

In relation to water activity, the highest values found for the ingredients were: 0.766; 0.729; 0.720; 0.713; 0.711; 0.649; 0.646 and 0.641 for pelleted layer diet, salt, pelleted rooster diet, mash layer diet, industrial kaolin, soybean meal, mash rooster diet and wheat bran, respectively.

Table III shows the adjustment constants of the GAB mathematical model (X_m, C and K) for the sorption isotherms.

Due to the fact that the highest peaks in moisture increase in different points, the percentage of moisture absorbed by the vitamin supplement, salt, mycotoxin deactivator, pelleted rooster diet, mash rooster diet, mash layer diet, pelleted layer diet, corn, bacitracin zinc, vitamin mineral supplement, phytase, rice bran,

Table I. **Water activity and moisture content of the ingredient in sampled condition at the feed mill** (Actividad del agua y contenido de humedad del ingrediente en la condición de muestreo en la fábrica de piensos).

Ingredients	Water Activity (a_w)	Moisture Content (as-fed basis)	Moisture Content (dry matter basis)
Pelleted Layer Diet	0,766	12,94	14,87
Salt	0,729	1,46	1,48
Pelleted Rooster Diet	0,720	12,53	14,33
Mash Layer Diet	0,713	11,90	13,51
Industrial Kaolin	0,711	1,95	1,99
Soybean Meal	0,649	11,75	13,31
Mash Rooster Diet	0,646	11,49	12,98
Wheat Bran	0,641	11,41	12,88
Corn	0,639	12,66	14,50
Phosphate	0,639	4,52	4,74
Rice Bran	0,602	10,2	11,35
Limestone	0,575	0,06	0,06
L-Tryptophan	0,566	0,18	0,18
Mycotoxin Deactivator	0,552	6,25	6,67
Vitamin Supplement	0,541	3,83	3,98
Mineral Supplement	0,536	3,09	3,18
Phytase	0,529	8,94	9,82
DL-Methionine	0,526	0,10	0,10
L-Valine	0,507	0,14	0,14
L-Threonine	0,505	0,05	0,05
Vitamin-Mineral Supplement	0,483	4,30	4,50
BHT	0,482	30,5	43,89
L-Lysine HCl	0,444	1,56	1,58
Coccidiostat	0,426	5,75	6,10
Bacitracin Zinc	0,333	5,62	5,96
Choline Chloride	0,150	1,45	1,47

wheat bran, mineral supplement, soybean meal, L-Lysine and choline chloride (as-fed basis) was determined in the a_w range between 0.5 and 0.7, and for the L- threonine, limestone, BHT, DL- methionine, L- valine, L- tryptophan, phosphate, industrial kaolin, and coccidiostat (as-fed basis) between 0.6 and 0.8. The hygroscopic behavior of the ingredients was: L- threonine (0.04%), limestone (0.05%), BHT (0.06%), DL- methionine (0.10%), L-valine (0.14%), L- tryptophan (0.15%), phosphate (0.96%), industrial kaolin (1.13%), vitamin supplement (1.44%), salt (1.64%), mycotoxin deactivator (2.87%), pelleted rooster diet (2.95%), mash rooster diet (3.13%), mash layer diet (3.17%), pelleted layer diet (3.18%), corn (3.21%), bacitracin zinc (3.27%), vitamin mineral supplement (3.34%), phytase (3.49%), rice bran (3.69%), wheat bran (4.12%), mineral supplement (4.15%), soybean meal (4.28%), coccidiostat (5.81%), L- Lysine HCl (7.22%) and choline chloride (25.89%).

According to Krist, Nichols and Ross (1999, pp. 540-547), ingredients with water activity values between 0.600 and 0.850 are considered intermediate, and those with values less than 0.600 are considered as having low water activity, therefore the data found fell into the intermediate water activity range ($0.60 < a_w < 0.85$) and low water activity category ($a_w < 0.60$). Lima and Sant'ana (2011, pp. 125-129), in a study with salted and dried fish, found intermediate water activity levels, varying between 0.74 to 0.75.

Paglarini et al. (2013, pp. 299-305*),* working with isotherms of mango pulp (*Mangifera indica L.*) manteiga variety, and Moreira *et al.* (2013, pp. 1093-1098), in a study with isotherms of mash lyophilized, also observed the best fit for of the experimental data by the GAB mathematical model.

Monolayer moisture (X_m) refers to the amount of water strongly adsorbed over the primary sites, considered as the unit where the ingredient is most stable. From the monolayer moisture one can ascertain the lowest water content contained in each ingredient. According to Goula, Karapantsios and Adamopoulos (2008, pp. 73-83), monolayer moisture is that which gives rise to (at a certain temperature) the greatest stability and minimal losses in the quality of the diet product. The X_m parameter is important as it can be related to the start of a series of diet deterioration chemical reactions (Ferreira & Pena 2003, pp. 251-255). The increase in monolayer moisture associated to an increase in temperature is not common to all ingredients, and,

Table II. Maximum, mean and minimum values of the temperature and relative humidity in the bagged material store room and medications room of the animal feed mill (Valores máximos, medios y mínimos de la temperatura y la humedad relativa en el almacén de material embolsado y la sala de medicamentos de la fábrica de alimentos para animales).

Environmental Variable	Temperature (°C)			
	Summer		Winter	
Temperature (°C)	Bagged Material Storeroom	Medications Room	Bagged Material Storeroom	Medications Room
Maximum	33,7 °C	28,8º C	23,0º C	19,2º C
Mean	26,3°C	26,3º C	14,5 º C	15,8º C
Minimum	20,3°C	23,4º C	8,2º C	12º C
Relative Humidity (RH%)				
Relative Humidity (RH%)	Bagged Material Storeroom	Medications Room	Bagged Material Storeroom	Medications Room
Maximum	92,10%	87,7%	81,20%	80,20%
Mean	76,03%	74,70%	67,50%	68,30%
Minimum	49,50%	63,50%	43,20%	56,10%

according to Ferreira and Pena (2003, pp. 251-255), this behavior can be explained by the fact that a temperature increase can make modifications in the physical structure of the product, making a larger quantity of active sites with affinity to water molecules available or increasing the solubility of solutes in the product, thus retaining more water molecules in the monolayer.

The constants C and K from the GAB model are related to the effect of the temperature. According to Catelam, Trindade and Romero (2011, pp. 1196-1203), C is the constant that is related to the sorption heat in the monolayer and K refers to the sorption heat in the multilayer. According to Gabas *et al*. (2007, pp. 246-252), low temperatures favor the force of the adsorbate-adsorbent interaction, causing a certain increase in the values of the constant C. Timmermann (2003, pp. 235-260) states that the constant K of the GAB model increases with the force of the adsorbate-adsorbent interaction and values greater than 1 are physically inadequate, indicating an infinite adsorption.

In relation to determining the sorption isotherms of the ingredients used in formulating mash and pelleted diet for roosters and laying hens, a similar behavior was found in the format of the isotherms for all the materials. In accordance with the IUPAC classification (1985, pp. 603-619), the isotherms can present five different formats. As a rule, the experimental results of the sorption isotherms showed curves with sigmoidal behavior, implying type II isotherms, characteristic of soluble, protein and chalky products, and also common to food products (IUPAC 1985, pp. 603-619).

According to Brunauer, Emmett and Teller (1938, pp. 309-319), these curves indicate the type of forces that intervene in the binding between the water and the hygroscopic material surface and enable evaluations of the superficial structure, stability during storage, and provide information for the design of adequate packaging to guarantee better product conservation.

Therefore, as the formulation of poultry diet takes into account the ingredient in its as-fed basis, special care should be taken with the moisture content of the ingredients, since there may be differences between the formulate and what is consumed by the animal, due to the water adsorption.

According to Rahman (2008) microorganisms begin to grow from a water activity level of 0.600 onwards. Hence, the ingredients and diets with water activity values of less than 0.600 are guaranteed against the growth of fungi, mold and bacteria.

The sorption isotherms (**Figure 1**) demonstrate that the corn, wheat bran, soybean meal, rice bran, mash rooster diet, pelleted rooster diet, mash layer diet and pelleted layer diet will be microbiologically stable ($a_w < 0.6$) when they have a moisture content of less than 11.66, 10.98, 10.19, 9.64, 10.67, 10.54, 9.73 and 9.63 %, respectively.

It is found that at low water activity levels the ingredients adsorb small amounts of water, and at high levels of water activity there is an accentuated increase in adsorbed water, which result was also found by Cardoso (2012, p. 60), working with mixed flours of rice and barley husk.

Furthermore, the addition of water in the extrusion process, for example, results in increased moisture and water activity of diets, affecting the nutritional value of the dietary protein (Murakami 2010, p. 40).

Moreover, it is important to highlight that in their final portion, corresponding to the highest water activities, the isotherms demonstrated a more hygroscopic behavior, characterizing a rise in the curve. That same behavior was also verified by Canuto, Afonso and Costa (2014, pp. 179-185), when studying water adsorption of freeze-dried papaya pulp powder.

The sorption isotherms of the aminoacids (**Figure 1**) show that L- valine, L- threonine, L- tryptophan, L- lysine HCI, and DL- methionine will be microbiologically stable ($a_w < 0.6$) when they present a moisture content of less than 0.14, 0.01, 0.16, 3.43, and 0.12 %, respectively.

Slight decreases in the balance moisture are found with increase in temperature. Ferreira and Pena (2003,

Table III. Estimated adjustment parameters of the GAB mathematical model (X_m, C and K) for the sorption isotherms (Parámetros de ajuste estimados del modelo matemático GAB (Xm, C y K) para las isotermas de sorción).

Material	X_m [(1)]	C[(2)]	K[(3)]	R^2
Mycotoxin Deactivator[(4)]	3,131	19,28	0,901	0,99
Mycotoxin Deactivator[(5)]	3,21	18,539	0,931	0,99
Choline Chloride[(4)]	57,44	0,826	0,573	0,97
Choline Chloride[(5)]	52,91	0,66	0,78	0,98
Kaolin[(4)]	0,533	2,936	0,969	0,57
Kaolin[(5)]	0,54	2,749	0,974	0,57
Bacitracin Zinc[(4)]	4,37	12590	0,856	0,99
Bacitracin Zinc[(5)]	4,57	2786,5	0,896	0,99
Vitamin Supplement[(4)]	1,773	95044	0,877	0,99
Vitamin Supplement[(5)]	1,797	44430	0,894	0,99
Layer Mineral Supplement[(4)]	2,814	8,019	0,961	0,97
Layer Mineral Supplement[(5)]	3,471	2,944	0,962	0,96
Mineral Supplement[(4)]	0,741	17460	1,256	0,82
Mineral Supplement[(5)]	0,756	3290,9	1,264	0,83
Mash Layer Diet[(4)]	5,002	559,2	0,812	0,99
Mash Layer Diet[(5)]	5,282	424,9	0,853	0,99
Pelleted Layer Diet[(4)]	4,902	10276	0,819	0,99
Pelleted Layer Diet[(5)]	5,147	1593,2	0,862	0,99
Pelleted Rooster Diet[(4)]	5,839	372,39	0,747	0,99
Pelleted Rooster Diet[(5)]	6,183	603,91	0,794	0,99
Mash Rooster Diet[(4)]	5,739	4806,9	0,771	0,98
Mash Rooster Diet[(5)]	6,102	1349,2	0,817	0,98
Corn[(4)]	7,055	28,439	0,704	0,97
Corn[(5)]	7,599	23,719	0,756	0,97
Soybean Meal[(4)]	4,625	1323,4	0,911	0,99
Soybean Meal[(5)]	4,843	748,9	0,955	0,99
Wheat Bran[(4)]	5,365	87,817	0,861	0,97
Wheat Bran[(5)]	5,656	80,4	0,911	0,96
Rice Bran[(4)]	4,627	146,03	0,872	0,99
Rice Bran[(5)]	4,841	136,66	0,915	0,98
Limestone[(4)]	0,024	199920	0,957	0,81
Limestone[(5)]	0,024	34622	0,957	0,81
Phosphate[(4)]	2,346	258942	0,628	0,31
Phosphate[(5)]	2,325	110208	0,663	0,31
Phytase[(4)]	4,754	71,622	0,848	0,99
Phytase[(5)]	4,971	67,362	0,892	0,99
Coccidiostat[(4)]	3,437	125,93	0,935	0,99
Coccidiostat[(5)]	3,587	96,241	0,966	0,99
Salt[(4)]	10,988	0,002	1,27	0,89
Salt[(5)]	11,365	0,002	1,272	0,90
L-Lysine HCl[(4)]	25,46	0,063	0,869	0,94
L-Lysine HCl[(5)]	29,32	0,057	0,887	0,94
DL-Methionine[(4)]	0,052	158,12	0,957	0,76
DL-Methionine[(5)]	0,052	135,46	0,957	0,76
L-Threonine[(4)]	0,39	0,006	1,008	0,94
L-Threonine[(5)]	0,018	1104,9	1,034	0,97
L-Valine[(4)]	0,056	4659	1,008	0,91
L-Valine[(5)]	0,056	6714,1	1,008	0,91
L-Tryptophan[(4)]	0,067	2550,5	0,982	0,94
L-Tryptophan[(5)]	0,067	5078,7	0,983	0,94

[(1)] Monolayer moisture content; [(2), (3)]constants; [(4)]moisture content on an as-fed basis; [(5)]moisture content in dry matter basis

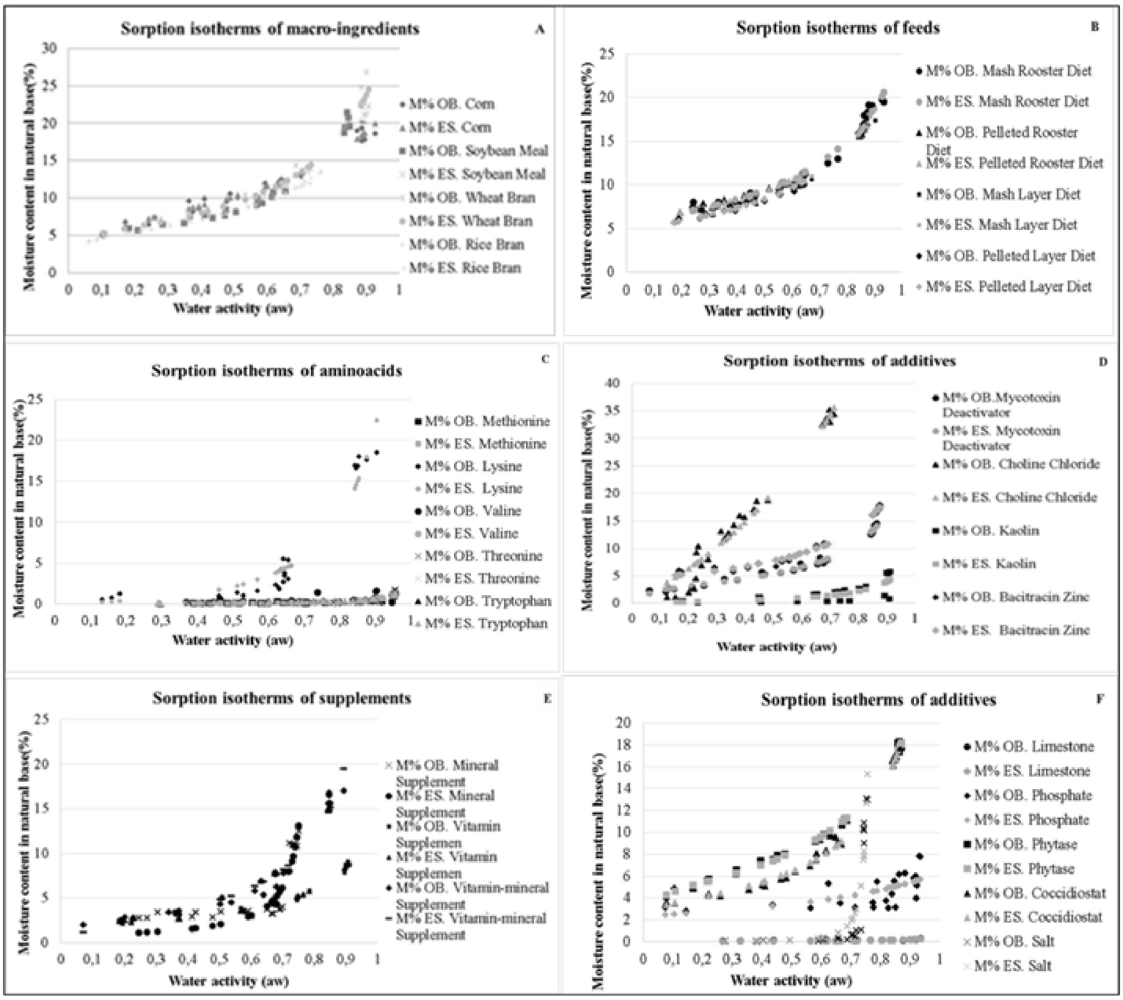

Figure 1. A: Sorption isotherms of macro-ingredients (corn, soybean meal, wheat bran and rice bran) on an as-fed basis, where: M% OB: observed moisture content; M% ES: estimated moisture content, B: Sorption isotherms of feeds (mash rooster diet, pelleted rooster diet, mash layer diet and pelleted layer diet), C: Sorption isotherms of aminoacids (methionine, lysine, valine, threonine and tryptophan). D: Sorption isotherms of additives (mycotoxin deactivator, choline chloride, kaolin and bacitracin zinc). E: Sorption isotherms of supplements (mineral supplement, vitamin supplement and vitamin-mineral supplement). F: Sorption isotherms of additives (limestone, phosphate, phytase, coccidiostat and salt) (A: Isotermas de sorción de macroingredientes (millo, fariña de soia, farelo de trigo y salvado de arroz) en base a alimentación, onde: M% OB: contido de humidade observado; M% ES: contido de humidade estimado, B: Isotermas de sorción dos pensos (dieta de puré de galo, dieta de galo granulado, dieta de capa de puré e dieta de capa granulada), C: Isotermas de sorción de aminoácidos (metionina, lisina, valina, treonina y triptófano). D: Isotermas de sorción de aditivos (desactivador de micotoxinas, cloruro de colina, caolín y bacitracina cinc). E: Isotermas de sorción dos suplementos (complemento mineral, suplemento vitamínico e suplemento vitamínico-mineral). F: Isotermas de sorción de aditivos (piedra caliza, fosfato, fitasa, coccidiostático y sal).

pp. 251-255) justified this behavior based on the increased vapor pressure of the water in the air and on the product surface, bearing in mind that this increase is greater on the product surface, as it contains more water molecules than in the air. Therefore, the greater vapor pressure results in a greater water loss so that balance is attained.

The sorption isotherms of the supplements are presented in **Figure 1**. In relation to the microbiological stability ($a_w < 0.6$) of the supplements it is observed that the vitamin supplement will be stable when it presents a moisture content of less than 3.74%, the vitamin mineral supplement, 6.09% and the mineral supplement when the moisture is less than 3.00%.

For a given temperature, it can be verified that the moisture content of the product increases with water activity. According to Costa (2010, p. 203), this occurs because the vapor pressure of the water in the product accompanies the increased vapor pressure of the environment in which it is inserted. The sorption isotherms of the additives (**Figure 1**) used in the formulation of the diets demonstrate that the mycotoxin deactivator, choline chloride, kaolin, bacitracin zinc, limestone, phosphate, phytase, coccidiostat, salt and BHT will be microbiologically stable ($a_w < 0.6$), when they present a moisture content of less than 6.52, 26.64, 1,02, 8.98, 0.06, 3.76, 9.55, 7.77, 0.33, and 26.51%, respectively.

For the BHT, the data obtained did not fit for the determination of the sorption isotherm, in other words, it presented values for which it was not possible to observe any moisture adsorption by the ingredient.

In relation to the choline chloride, the ingredient with the highest hygroscopicity, care must be taken, since the increase in free water content in the mixture can lead to loss of hydrosoluble vitamins, as well as operational problems in the production of animal diets.

CONCLUSIONS

The format of the sorption isotherms of the ingredients and diets assessed are type II, or sigmoidal, common among feed, protein and chalky products, except for BHT, which presented values that do not fit for isotherm determination.

Ingredients and diets have different hygroscopic behavior, which can lead to deterioration and low accuracy in nutritional values of diet, since formulation is based on as-is fed basis.

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